Synthesis of MoS$_2$ Ultrafine Particles: Influence of Reaction Condition on the Shape and Size of Particles

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Abstract

Nano MoS$_2$ (5-10) nm have been successfully synthesized using wet chemical synthesis in an aqueous solution employing ammonium molybdate, citric acid, and ammonium sulfide as the reactants. The high-speed homogenizer played a vital role to control size in the narrow range of (5-10) nm. XRD pattern reveals the broadening in peak intensity with the enhanced crystallinity of (002) peak at 10.4°, having a crystallite size 2 nm. TEM and EDX studies elucidate the presence of average particle size in the range of (5-10) nm with elemental molybdenum and sulfide ion. Our work has provided easy and convenient technique to synthesize nano MoS$_2$ for development of nano lubricant with reduced wear and coefficient of friction.

Keywords

Molybdenum disulfide, High-speed homogenizer, Aqueous solution, Ultrafine nanoparticles

Introduction

In past few years, there has been considerable interest among researchers in the study of transition metal dichalcogenide layered compounds, a group of anisotropic materials with strong bonding within the layers and weak interlayer interactions [1]. Apart from WS$_2$ (Tungsten disulfide), MoS$_2$ (Molybdenum disulfides) has drawn attention among researchers due to its extensive applications as catalysts and lubricants [2]. Molybdenum disulfide (MoS$_2$) naturally occurs as molybdenite has layered hexagonal packed structure consisting of -S-Mo-S- sheets stacked one after another by weak Vander Waals interactions. The layered structure shows smooth sliding contact resulting in low wear and coefficient of friction property for its use as a lubricant [3]. It has got appreciable electron band-gap (-1.7 eV), has developed an upsurge interest in MoS$_2$ based solar cell and solar hydrogen production materials [4]. The applicability of MoS$_2$ can further be exploited by reducing the size of MoS$_2$ crystals, which will also improve the lubrication properties in bearing and other heavy-wear applications and will promote the further development of nano lubricants [5]. The two synthetic approaches for the synthesis of MoS$_2$ nanoparticles: Physical and chemical methods are primarily employed for the...
synthesis of Molybdenum based nanoparticles [6]. Chemical synthesis involves wet chemical methods, sonochemical and chemical vapor deposition, sol-gel processing, spray pyrolysis, co-precipitation, metathesis reaction. Physical process involves high energy sources, which is more complicated than chemical method. Moreover, the chemical method gives us the uniformity in shape and size of a particle in contrast to the physical approach [7]. Hydrothermal technique, a chemical route for the synthesis of this particle utilizes water as a reaction solvent is drawing attention among researchers for an environmental reason, avoids the complexity and handling of dangerous organic solvents and good crystallization of the products are achievable [8]. Studied a successful technique was employed using an ultrasonic cracking approach for the synthesis of MoS2 nanorods and microparticles [9]. A successful technique was employed using an ultrasonic cracking approach for the synthesis of MoS2 nanorods using a soft chemical synthetic method at 90 °C in the presence of ammonium molybdate tetrahydrate, and sodium sulfide as precursors of Mo had been reported [10]. High dispersion factor in MoS2 by using (NH4)2MoS4 and N2H4·H2O via aqueous medium precipitation method was carried out [11]. The ball-like MoS2 Nanoparticles on calcination of amorphous molybdenum sulfide was achieved through precipitation from the solution of Na2MoO4 and thioacetamide in water and alcohol [12]. In recent years, many studies have been carried out on the application of nanoparticles in the field of lubrication. The reduction of friction and wear are dependent on the characteristics of nanoparticles such as size, shape and concentration. Many researchers report that the concentration of nanoparticles in the base-oils is an important parameter while formulating the nano-lubricants [13]. It has already been established that addition of solid lubricant particles, such as, MoS2, h-BN, WS2, and IF-MoS2 to mineral oil improves the tribological properties of lubricants [14]. COSMA additives show anti-wear behavior and in combination with ZDDP, exhibit a good synergistic effect, reducing the wear-scar diameter by 60% and increasing the initial seizure load from 50 kg to 85-95 kg [15]. However, its applicability for a positive outcome is dependent on the purity concerning the stoichiometry and phase. Therefore, there is need to study their synthetic methods which can be employed for the better yield with high purity. The synthesis of MoS2 nanoparticles smaller than 10 nm has remained a challenge but using dispersion unit we have achieved MoS2 nanoparticles less than 10 nm. The earlier reported work uses (Ammonium Molybdate, Ammonium sulfide, and Citric acid) as reactants for obtaining MoS2. This reagent applies a simple and scalable process and involves low-cost reagents, instead of other more complex reaction methods [1-5]. In this work of metal dichalcogenides, we report a simple low-temperature wet chemical synthesis method for preparation of MoS2 nanoparticles with control of the concentration of reactants, stirring speed, duration, and temperature as the important parameters. Another aspect of the present investigation is to highlight the use of nano MoS2 for lubricant chemists in understanding better the place and role of nanochemistry in the creation of reduced wear and coefficient of friction property than base oil and solid lubricants commercially available.

**Experimental**

**Materials**

The ammonium molybdate tetrahydrate (NH4)6Mo7O24·4H2O, ammonium sulfide (NH4)2S of Analytical Grade (AR) were purchased from Alpha-Aesar, and citric acid (C6H8O7) is purchased from Merck India Ltd. and was used without further purification. Deionized water of pH (≤ 7) and conductivity of ≤ 5 µS/cm was used as a solvent.

**Synthesis**

The synthesis of MoS2 was carried out in three steps as described below,

**Step 1: Complex formation**

In a typical procedure, Ammonium Molybdate tetrahydrate [(NH4)6Mo7O24·4H2O] (0.809 mM) was dissolved in three-necked Round Bottom flask (RB) containing deionized water (100 ml) which was subjected for continuous stirring at room temperature. Subsequently, citric acid monohydrate (2.42 mM) was added to the above mixture and stirring at 1500 rpm was continued for One hour at 110 °C. Then, the mixture was allowed to attain room temperature, and pH of reaction mixture was maintained to 3 with addition of ammonium hydroxide (NH4OH), which followed the change in primary transparent color to greenish white. The reaction mixture was used for further step without isolation of complex.

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Step 2: Reduction of complex

The use of high-speed homogenizer at 17,000 rpm was used for the effective reaction. The complex mixture prepared in step 1 was reduced by drop wise addition of 20-22 wt% aqueous solution of ammonium sulfide (NH₄)₂S. The careful observation revealed the formation of the precipitate by a change in color of reaction mixture from greenish white to black via red.

Step 3: Purification

The precipitate formed in step 2 was subsequently filtered by using Millipore filtration unit and with complete washing by deionized water. The precipitate was completely dried at 90 °C for 2 hours in vacuum oven. The final product was flushed with nitrogen and stored in the airtight bottle to ensure stability.

Characterization

UV-Vis absorption of MoS₂ nanoparticles was carried out using Agilent Cary 5000 UV visible NIR spectrometer over a scanning range of 200-800 nm. The Fourier transform infrared spectrum (FT-IR) in the range of 4000-400 cm⁻¹ at a resolution of 0.5 cm⁻¹ was collected using Perkin Elmer FT-IR instrument (model spectrum 2), in transmission mode using solid tablet forms of samples. Transmission electron microscope (TEM) using Tecnai G² 20 equipped with EDS was used to measure the elements contained in the samples. The powder structure and phase transformation of as-prepared composites were acquired on a Rigaku RU 200B X-ray Diffractometer (40 kV, 100 mw, Cu Kα radiation) ranging from 10 °C to 90 °C with a rotating anode. The structural property of nanomaterials was studied by Raman spectroscopy using Renishaw instrument. DLS technique was utilized for determination of average size distribution of the nanoparticles using a Malvern Zeta Sizer Nano ZS by (Malvern Instruments).

The thermal stability associated with phase transformation was determined by Thermogravimetric Analysis using TGA SDTQ- 600 TA instrument at a heating rate of 10 °C/min at room temperature to 1100 °C under inert atmosphere. The co-efficient of friction and wear property was determined using TR-30L-PNU-IAS four ball tester instrument of make DUCOM™ by material characterization system. The study was carried out according to ASTMD 4172 under 40 kg load for duration of 3600 second, temperature 75 ± 2 °C and speed of 1200 ± 60 r/min. The 3D surface topography was done by Zeta instrument of make Zeta 500 optical profilometer from 300 mm wafers.

Results and Discussion

Chemistry behind the formation of MoS₂ particles

The formation of MoS₂ nanoparticles is entirely dependent on the initial configuration of the complex with citric acid which further acts as an in-situ formed precursor of the Mo metal. Furthermore, after addition of sulfur source initially built complex reacts immediately and leads to the formation of
MoS$_2$ nanoparticles. The size of the MoS$_2$ NPs was well controlled by the presence of citric acid molecules which acts as the capping agent after dissociation of the complex. The reducing nature of the ammonium sulfide converts compound easily to the MoS$_2$ NPs. The formation and possible mechanism of MoS$_2$ NPs via reduction of initially formed Mo-complex is presented in Scheme 1 and Scheme 2.

Optimization of reaction parameters like molar concentration, stirring speed at complex formation step and reduction step played the significant role. The time duration of reaction and the minute color change observation have been the essential parameters for obtaining small size particles shown in Table 1. Reduction of the complex is most crucial because at this step there is a high chance of nucleation among particles. The variation in the size was achieved using high-speed homogenizer as shown in Figure 1. It has been further observed that at optimized concentration of 0.08 M and speed of 17000 rpm we have achieved smaller particle size with even surface morphology.

**UV-Visible spectroscopy**

The reaction progress was studied by utilizing UV-visible spectrum of pure reactants Ammonium molybdate, citric acid, intermediate (complex) and final products. The absorption peaks for the reactants are observed at 210 nm, while the sharp absorption peaks for intermediate and product are found at 270, 315, 400 and 480 nm as shown in Figure 2. The Bathochromic shift in the peak values to 315 nm and 400 nm with diminishing intensity in reactant peak (210, 200, and 270) indicates formation of MoS$_2$ Nanoparticles which is in agreement with the reported study of [16,17]. Further, the transparent color of reaction solution changes to light green color possibly due to complex formation and finally to black after addition of ammonium sulfide.

**FTIR**

Figure 3 shows the spectrum of ammonium mo-

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**Table 1:** Optimization of molar concentration, stirring speed and obtained inference.

| Sr.no | Optimized Concentration (M) | Stirring speed of complex formation (rpm) | Filtrate color | Inference               |
|-------|----------------------------|----------------------------------------|---------------|------------------------|
| 1.    | 0.08                       | 800                                    | Colorless     | No Complex formation   |
| 2.    | 0.08                       | 1200                                   | Colorless     | Slow Complex formation |
| 3.    | 0.08                       | 1500                                   | Greenish white| Instant color change   |

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Raman spectroscopy has been extensively used for the characterization of carbonaceous materials with different microstructures including graphene, citric acid, and molybdenum disulfide of as prepared samples respectively diluted with Potassium Bromide (kBr) and pressed to form palettes. The band at 460 cm\(^{-1}\) of ammonium molybdate changes into 480 cm\(^{-1}\) corresponds to Mo-S peak proves its consistency with previous reported work \cite{18}. The band at 600 cm\(^{-1}\) and above corresponds to Mo-O vibrations as mentioned by \cite{19}. The signature sharp peaks at about 890 and 798 cm\(^{-1}\) are attributed to the out-of-plane OH bending mode \cite{20}.

**Raman spectra**

Raman spectroscopy has been extensively used for the characterization of carbonaceous materials with different microstructures including graphene.
The $E_{1g}$ mode involves the in-layer displacement of Mo and S atoms, whereas the $A_{1g}$ mode involves the out-of-layer symmetric displacements of S atoms \[22\].

XRD

The XRD patterns of synthesized materials were analyzed for crystallinity and phase structure are taken at 80 °C and annealed temperature of 300
°C shown in Figure 5. The peak broadening at 10 degree and suppression of other relative peaks indicates the synthesized material is more of a single phase than multiphase and is amorphous in nature [24]. Most of the integrated intensity of the X-rays spectrum is confined to a broad peak confirming amorphous materials, and the (002) peak of the crystalline MoS$_2$ is found to be in agreement [25]. The calculation of crystallite size by scherrer formulae eq-1, elucidate the change in size from 2 to 32 nm with gauss curve (Normal distribution) to calculate the full width half maximum (FWHM) value. The peak value shift to a higher angle of 2θ = 32.2 degree when nanoparticle are annealed at higher temperature. The increase in crystalline nature at higher temperature is justified with a decrease in d-spacing value calculated by Bragg’s eq-2, from 0.44 to 0.28 nm Figure 4. The sphere shape of MoS$_2$ particles could be thin MoS$_2$ layers curled up by the high temperature annealing [26]. The removal of oxygen atoms from the lamellar MoO$_3$ is responsible for the decreased crystallinity.

**Bragg’s equation**

\[ n\lambda = 2d\sin\theta \] ........ eq. 2

Where,
- \( n \) is a positive integer
- \( \lambda \) is the wavelength of the incident wave
- \( \theta \) is the scattering angle
- \( d \) is the spacing of the crystal layers (path difference)

**TEM**

Morphology of the MoS$_2$ NPs, were investigated by TEM analysis. The TEM images of as-synthesized nanoparticles are shown in Figure 6. Special specimen techniques for grid preparation was carried out on the gold coated grid of 200 mesh size and followed by the drop-casting, of particles dispersed in methanol using ultrasonicator. Before carrying out the characterization, the sample was completely dried under table lamp for 24 hr. The TEM micrographs of MoS$_2$ display spherical shape with the particle size of about (5-10) nm and homogenous distribution of nanoparticles over a large area in Figure 6a and Figure 6b. The inset in Figure 6a shows the selected area of electron diffraction (SAED) pattern for the sample taken with an aperture size (5 nm), and suggesting the amorphous and circular arrangement of MoS$_2$ nanoparticles [27,28]. It thereby also elaborates the effect of using homogenizer at reduction step to achieve quantitatively the average particle size of 5 nm as shown in Figure 6a and Figure 6b. It can

**Scherer equation**

\[ L = D \frac{\lambda}{\beta \cos \Theta} \] ........ eq. 1

Where,
- \( L \) = crystal size
- \( D \) = shape factor which is approximately unity
- \( \lambda \) = the wavelength of X-ray (= 1.54 cm$^{-1}$, 056 Å)
- \( \Theta \) = the Braggs angle in radians
- \( \beta \) = full width at half maximum of the peak in radians.

**Figure 5:** XRD pattern of MoS$_2$ Nanoparticle, a) Dried at 80 °C, b) Heat treated at 300 °C for one hr.
be concluded that in Figure 6c and Figure 6d the increase in molar concentration simultaneously enlarge size by 20 nm, with the larger size upto 50 nm is shown in Figure 6e and Figure 6f.

**EDX**

Energy dispersive X-ray (EDX) spectrum measurement was used to analyze the entire chemical composition of synthesized particle Figure 7. EDX spectrum of the as-synthesized MoS₂ NPs showed the only presence of two elements viz. Mo and S which confirms the purity of the samples. Other elements such as O, Cl absence indicates sample is free from MoO₃ and MoO₂. The suppressed peak is assigned for copper constituent TEM grid. Furthermore, the quantification of the peaks gives S/Mo atomic ratio of 1:2.06, which is very close to the stoichiometric of MoS₂ and proves the synthesized product is purely stoichiometric and free from any other impurities [29,30].

Figure 6: TEM images of synthesized MoS₂ nanoparticles of 0.08 M a,b) Size < 5-10 nm; c,d) Size < 20 nm; e,f) size 50 nm.
High-speed Homogenizer at reduction step of the complex formation highlights the importance for change in size of MoS$_2$ nanoparticles from 32 to 3 nm confirmed by Dynamic Light Scattering (DLS) study.

**TGA**

Thermal stability of as-synthesized MoS$_2$ NPs was evaluated by TGA analysis. TGA spectrum is

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shown in Figure 9. At high-temperature MoS$_2$ exhibits significant property like lubricants. Thermal behavior of the obtained nano lamellar MoS$_2$ was studied in an inert atmosphere using thermal analyzer at a heating rate of 10 °C/min. The Nano lamellar MoS$_2$ is found to be stable up to 500 °C and further heating up to 700 °C causes a small weight loss of (5%) due to exothermic effect [32]. TGA thermograph depicts three step decomposition of MoS$_2$ NPs, first two-step decomposition is ascribed to the presence of organic matter in the sample while the third decomposition could be due to the MoS$_2$ Ns.

**Conclusion**

In above experimental analysis, MoS$_2$ nanoparticles were obtained by an ambient temperature chemical solution route using inorganic precursors. The tailoring and miniature of reaction parameters like concentration, stirring speed and reaction medium were optimized for attaining desired particle size (5-10) nm. The observation further explores the simple method for obtaining ultrafine particles in comparison to reaction condition reported in the literature and also, promotes large scale production of nano MoS$_2$ particles. From experiments, it has been observed that homogenizer having high speed has played the significant role in achieving small size particles at the time of nucleation. Consequently the 0.08 molar concentrations and 17000 rpm, highlight the reaction parameter to attain an average size of (5-10) nm was consequently considered to be crucial aspects. In this present investigation the characterization of as prepared particle show its crystallinity, thermal stability and high purity with least oxidizing characteristics.

**Acknowledgement**

The authors gratefully acknowledge DNMAT, CCF division of DMSRDE for carrying out DLS and TGA characterization and IIT-Kanpur to carry out TEM. The author wishes to acknowledge DRDO headquarters for their financial support.

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