

## Synthesis and Estimation of Flower like MoS<sub>2</sub> Nanostructures through Hydrothermal Process

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**Abstract:** MoS<sub>2</sub> nanoflowers were successfully synthesized by a simple hydrothermal process with the help of a surfactant. The products were characterized by X-ray powder diffraction (XRD), energy dispersive spectroscopy (EDS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). X-ray diffraction results showed that the as-prepared product was the hexagonal phase of MoS<sub>2</sub> without any impurity. TEM and SEM images showed that the MoS<sub>2</sub> nanoflowers had uniform sizes with diameter of about 1–2 μm and were constructed with many irregular nanosheets as a petal-like structure with thickness of several nanometers. A possible formation mechanism of the MoS<sub>2</sub> nanoflowers was preliminarily proposed on the basis of observations of a time-dependent morphology evolution process.

### 1) Introduction:

Over the last many times, important attention has been paid to the conflation and study of transition essence dichalcogenide nanosized structures because of its layered structure, a group of anisotropic accoutrements with strong cling within the layers and weak interlayer relations. MoS<sub>2</sub>, a typical concentrated transition essence sulfide, due to its unique parcels can be extensively applied in hydrogen storehouse, and as a catalyst, solid superlubricants, solid-state secondary lithium battery cathodes and so on (1 – 5). It's well known that parcels of accoutrements are concerned with their size, shape and specific face area. Compared with bulk accoutrements, nanomaterials always show unique size and shape dependent parcels (6). Nanoscale MoS<sub>2</sub> with specific morphologies and unique parcels is of great interest to experimenters exploring the design of MoS<sub>2</sub> accoutrements. thus colorful synthetic styles were explored to prepare MoS<sub>2</sub> insubstantial's, including gas-phase responses or solid – gas (7), ray ablation (8), thermal corruption (9), magnetron sputtering (10), sonochemical conflation (11) and electron ray irradiation activation (12). Still, all the below styles either involve a high temperature procedure or a complicated manipulation; in addition, the morphology and size of the products are hard to control also, the environmental regulations must also be taken into consideration in the developed styles. Up to now, it's still a great challenge to develop a simple and new system at low temperature to synthesize MoS<sub>2</sub> insubstantial in sufficient quantities.

Compared with the other styles, hydrothermal conflation is considered as an effective way to prepare inorganic nanomaterials due to similar graces as mild synthetic conditions, simple manipulation and good crystallization of the products (13). Lately Huang et al.

(14) described the conflation of hierarchical flower- suchlike MoS<sub>2</sub> spheres via a hydrothermal system using WO<sub>3</sub> or H<sub>2</sub>WO<sub>4</sub> as an cumulative Essence. (15) employed an ionic liquid-supported hydrothermal system to synthesize flower- suchlike MoS<sub>2</sub> microspheres with mean

periphery of about 2 mm. Herein, the main ideal of this exploration was to develop a simple and effective system for the conflation of MoS<sub>2</sub> nanoflowers via a hydrothermal process at low temperature. Latterly, the asset MoS<sub>2</sub> nanoflowers were characterized exercising XRD, EDS, SEM and TEM. Likewise, a possible conformation medium was banded to reveal the base of the effect of response time.

### **3) Experimental:**

#### *Synthesis of MoS<sub>2</sub> nanoflowers:*

All chemical reagents were of logical chastity and used directly without farther sanctification. The experimental procedure was designed as follows. 0.88 g of Na<sub>2</sub>MoO<sub>4</sub>, 0.725 g NH<sub>2</sub>OH • HCl and 1.40 g of CH<sub>4</sub>N<sub>2</sub>S were dissolved in 50 ml deionized water; also 0.18 g of CTAB was added into the result under constant shifting; pH value of the admixture was acclimated to 6 by the addition of 2 spook/ L HCl. The admixture was also transferred into a 100 ml Teflon- lined pristine sword autoclave and sealed, and the autoclave was placed in a pre-heated roaster at 180 °C for 24 h and naturally cooled down to room temperature. Black effects were collected by centrifugation and washed with distilled water and absolute ethanol several times, and eventually dried in vacuum at 60 °C for 10h.

#### *Estimation of MoS<sub>2</sub> samples:*

The X-ray diffraction patterns were recorded using a D8 advance (Bruker-AXS) diffractometer with Cu  $\alpha$  radiation ( $\lambda=0.1546$  nm). The morphologies and structures of the samples were characterized by surveying electron microscopy (SEM, JEOL JXA- 840A) and transmission electron microscopy (TEM) with a Japan JEM- 100CX II transmission electron microscope.

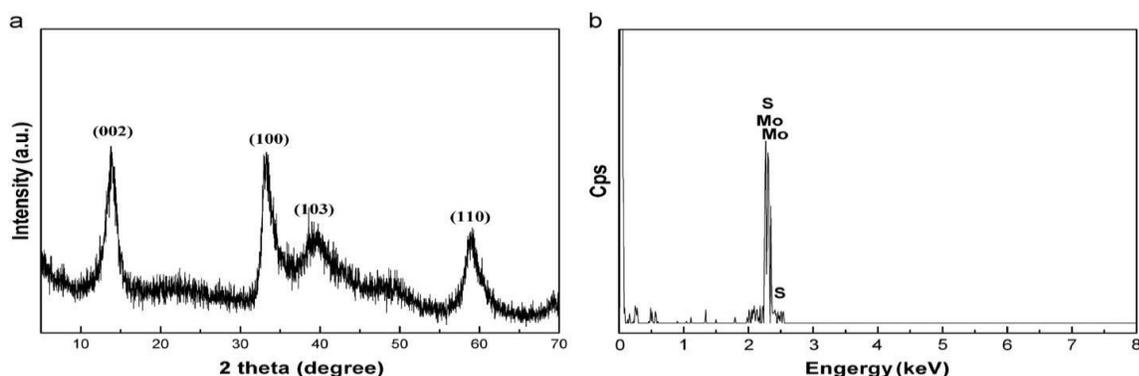
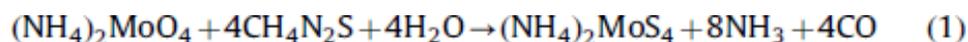
### **3) Result and Discussion:**

The crystalline structure and section purity of MoS<sub>2</sub> nano- flowers have been confirmed with the aid of XRD. As shown in Fig. 1a, all determined diffraction peaks may be systematically indexed to those of the hexagonal segment of MoS<sub>2</sub>, which might be in top agreement with the values of popular card (JCPDS No. 37-1492). No peaks from other impurities are detected within the XRD pattern, indicating that the pattern was relatively crystalline. Power-dispersive X-ray spectrometer (EDS) outcomes are proven in Fig. 1b, which reveals that the flowers consist of element Mo and S; no other element become observed. Furthermore, the Equantification of the peaks suggests that the atom ratio of Mo to S is about 1.98:1, which may be very close to the stoichiometry of MoS<sub>2</sub>.

The scale and morphology of MoS<sub>2</sub> samples were identified by way of SEM, TEM and HRTEM. Fig. 2a and b indicates the SEM images of the as-prepared MoS<sub>2</sub> sample, which screen that the standard product has a flower-like structure of about 1 to two mm length. It may be definitely seen that many irregular nanosheets mixture collectively and gather into the nanoflowers with the help of CTAB. The morphology and structure of the as synthesized MoS<sub>2</sub> nano-flowers have been in addition characterized using TEM and HRTEM

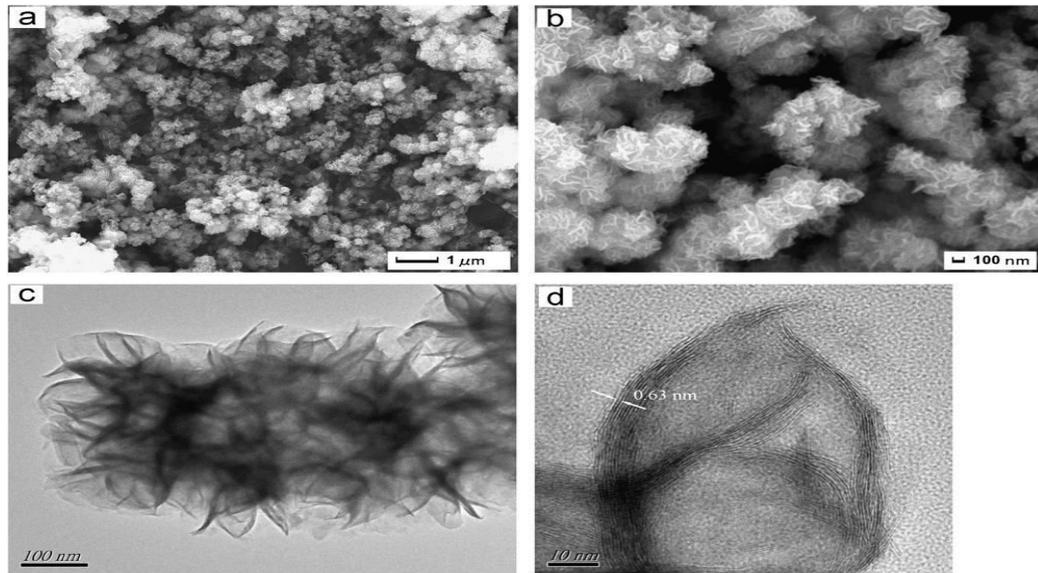
Fig. 2c suggests a normal TEM photo of MoS<sub>2</sub> nanoflowers; the effects are regular with the above SEM outcomes. Extra details for MoS<sub>2</sub> shape are illustrated with the aid of HRTEM as shown in Fig. 2d; it indicates that the layer structures of the products overlap every other. The mean value of distance among the 2 lattice fringes is zero.63 nm, which is in desirable settlement with the theoretical spacing for (002) planes of the hexagonal MoS<sub>2</sub> structure.

To better apprehend the underlying reason of the formation mechanism of that novel MoS<sub>2</sub> flowers, their boom manner has been investigated inspecting the goods harvested at distinctive periods of getting older time. As shown in Fig. 3a, many nanoparticles with the diameter of five–20 nm had been acquired for getting older period 6 h. Further, some sheet-like systems were additionally acquired, coexisting with the nanoparticles. With the growth of response time to 12 h, the nanosheets start to gather by means of themselves and the flowerlike systems begin to appear as shown in Fig. 3b. Upon gradual assembly of MoS<sub>2</sub> nanosheets, well- defined nanoflowers had been finally produced while response time reached 24 h. Maximum of the received merchandise are nanoflowers and nearly no MoS<sub>2</sub> nanoparticles may be discovered (Fig. 2). Especially, growing the reaction time to forty eight h, perfect crystalline flowerlike MoS<sub>2</sub> structures have been obtained (Fig. 3c). Therefore, primarily based on the above experimental outcomes, the formation mechanism of flowerlike MoS<sub>2</sub> systems is proposed to continue thru a three-step manner [16]. As all of us recognize, the crystal boom mechanisms in answer are so complex that the actual crystallization mechanism remains an open question [14]. Preliminary nucleation, oriented aggregation, and Ostwald ripening, and so on. Had been followed to account for the process of crystal boom. The formation process of MoS<sub>2</sub> flowers is schematically illustrated in Fig. Four. Within the first level, Na<sub>2</sub>MoO<sub>4</sub> and CH<sub>4</sub>N<sub>2</sub>S react with every other to shape Na<sub>2</sub>MoS<sub>4</sub> [12]. Then Na<sub>2</sub>MoS<sub>4</sub> may be easily reduced to MoS<sub>2</sub> nanoparticles with the assist of NH<sub>2</sub>OH • HCl in the hydrothermal method. Based on above evaluation, the above reactions may be expressed as follows:



Sooner or later, the nanoparticles commenced to bring together collectively and spontaneously combination into MoS<sub>2</sub> nanosheet structures as a way to lessen the high floor power via the process known as oriented aggregation [15]. Eventually, properly-defined MoS<sub>2</sub>

Fig. 1. (a) XRD pattern and (b) EDS of the as-prepared MoS<sub>2</sub>. Nanoflowers.



Fig(2), (a,b)-SEM, (C)- TEM and (D)-HRTEM images of MoS<sub>2</sub> nanoflowers.

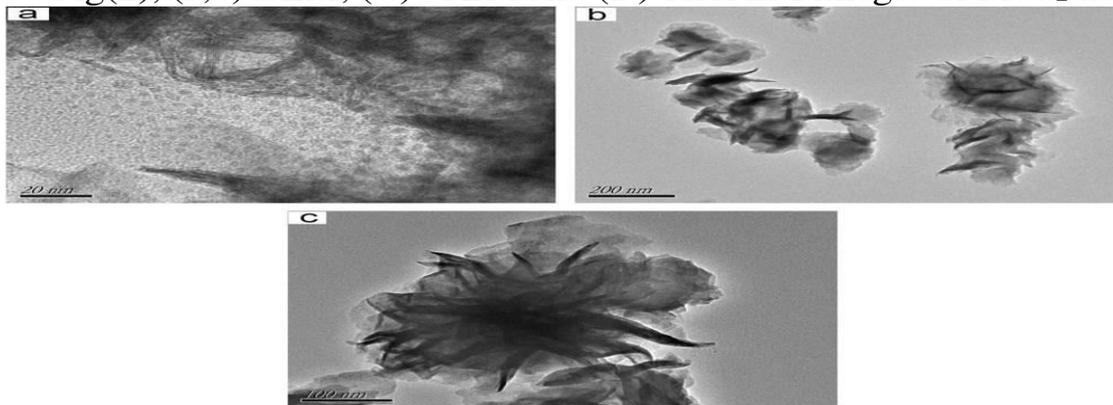
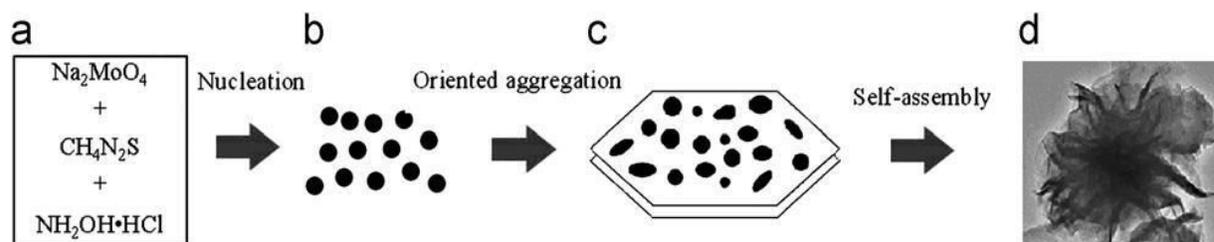


Fig. 3. TEM images of MoS<sub>2</sub> samples obtained under different reaction times

Nanoflowers are shaped from numerous MoS<sub>2</sub> nanosheets through a self-assembly system with the help of the surfactant. The absorption of the surfactant on the ones surfaces will cast off a part of the dangling bonds and bring about the discount in their floor strength, and therefore suppress the increase alongside those surfaces and cause them to seem in the final morphology. With a similarly boom inside the reaction time, the scabbled MoS<sub>2</sub> flower- like structures built by way of several nanocrystals may not be properly crystallized, and thus Ostwald ripening dictates the boom and recrystallization with enough ripening time [16]. The scabbled flower like systems of MoS<sub>2</sub> transform into well-defined MoS<sub>2</sub> nanoflowers thru the Ostwald ripening technique.



#### 4) Conclusion:

In summary, MoS<sub>2</sub> nanoflowers with mean diameter of approximately 1 to 2 μm were successfully synthesized with the aid of a CTAB assisted hydrothermal procedure under moderate conditions at the temperature one hundred eighty °C for 24 h. The experimental consequences recommend that the flower-like MoS<sub>2</sub> product is formed through a nucleation-oriented aggregation self meeting increase method. It is our hope that this efficient and simple artificial route can be applied as a preferred technique for the instruction of other transition metal sulfide nano or micro material

#### References:

- 1) Remskar M. *Adv Mater* 2004; 6:1497–504.
- 2) Tenne R. *Nat Nanotechnol* 2006; 1:103–11.
- 3) Kertesy M, Hoffman R. *J. Am Chem Soc* 1984;106:3453–60.
- 4) Tang GG, Tang H, Li CS, Li WJ, Ji XR. *Mater Lett* 2011;65:3457–60.
- 5) Tang H, Li CS, Song HJ, Yan XH, Yang XF. *Cryst Eng Commun* 2011.
- 6) Chen J, Li SL, Xu Q, Tanaka K. *Chem Commun* 2002; 16:1722–3.
- 7) Feldman Y, Wasserman E, Srolovitz DJ, Tenne R. *Science* 1995; 267:222–5.
- 8) Hu JJ, Zabinski JS, Sanders JH, Bultman JE, Voevodin AA. *J Phys Chem B* 2006;
- 9) Zelenski CM, Dorhout PK. *J Am Chem Soc* 1998; 120:734–42.
- 10) Ellmer K, Mientus R, Seeger S, Weib V. *Phys Status Solid A* 2004;201:
- 11) Dhas NA, Suslick KS. *J Am Chem Soc* 2005; 127:2368–9.
- 12) Chhowalla M, Amaratunga GAJ. *Nature* 2000; 407:164–7.
- 13) Yu QJ, Liu SK, Li MH, Tian YM. *Mater Chem Phys* 2008; 108:188–91.
- 14) Huang WZ, Xu ZD, Liu R, Zheng YF. *Mater Res Bull* 2008; 43:2799–805.
- 15) Ma L, Chen WX, Li H, Xu ZD. *Mater Chem Phys* 2009; 116:400–5.
- 16) Dai XJ, Luo YS, Fu SY, Chen WQ, Lu Y. *Solid State Sci* 2010; 12:637–42.