International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 06 Issue: 06 | June 2019 www.irjet.net p-ISSN: 2395-0072

Hydrothermal Synthesis of Molybdenum Disulfide

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Abstract – *In this study we report the effect of preparation conditions of MoS_2 nanoparticles by hydrothermal method by varying the Sulphur precursors (thiourea, thioacetamide, sulphur) on the morphologies of MoS_2 where investigated. The as-synthesized materials were characterized by x-ray diffraction (XRD), field emission scanning microscopy (FESEM).*

Results showed that the three kinds of morphologies of MoS_2 were obtained by varying sulphur precursors. Morphologies varied from tube spheres shape to cluster of threads to clusters of spongy cotton.

***Key Words*: Hydrothermal, MoS_2, Controllable Morphologies.**

1. INTRODUCTION

Molybdenum disulfide (MoS_2) is a typical layered transition metal dichalcogenide formed by the stacking of weakly interacting 2D S-Mo-S layer like graphite, which shows many potential applications [1], such as solid state lubricants [2], supercapacitor [3, 4], rechargeable batteries [5], hydrogen generation [6] , hydrogen storage [7] and as a photocatalyst to degrade pollutants or produce hydrogen [8]. The performance of nano-MoS_2 were varying or depending upon its size, shape and structure. Therefore, the preparation of MoS_2 with specific morphology is of great interest. During these preparation processes, the additive [9, 10] were usually used to control their morphologies. It is well-known that the starting materials, molar ratios, hydrothermal temperature and time during the hydrothermal processes will play important role in the morphologies control, crystal phase and performance. Zheng [11] group used MoO_3 and $Na_2S{\cdot}9H_2O$ as precursors to prepare MoS_2 materials which morphologies varied from amorphous nano-spheres to crystalline nano-flowers when hydrothermal temperatures enhanced from 200 to 320 ^oC. Chen[12] group used $Na_2MoO_4{\cdot}9H_2O$, $SC(NH_2)_2$ and graphene oxide as starting reactants to anchor nano-flakes MoS_2 onto graphene. The results showed that the nano-flakes structure is unchanged and crystal phase of MoS_2 changed from 1T in the low-temperature region (below 180 ^oC) to 2H in the high-temperature region (above 210 ^oC). The present work focuses on investigating effects of S/Mo molar ratios, hydrothermal temperature and time on crystal phase structure and pore structures and morphologies of MoS_2.

2. Materials and Methods

All the chemicals used were of analytical grade. Sodium Molybdate (Na_2MoO_4), Sulphur (S_8), Thiourea ($(NH_2)_2CS$), Thioacetamide (CH_3CSNH_2), surfactants namely, Sodium Dodecyl Sulphate ($CH_3(CH_2)_{11}SO_4\,Na$), N,N,N,N-Cetyl Trimethy Ammonium Bromide ($C_{19}H_{42}BrN$) and solvents like Tetrahydrofuran (THF), N,N Dimethylformamide (DMF), Acetone and Toluene were purchased from Merck India Ltd.

2.1Experimentation

In a typical hydrothermal procedure, 0.6048 g of Na_2MoO_4 and 0.16 g of S_8) were weighed and dissolved in 30mL of deionized water. 0.1g of SDS was added to the reactant solution under vigorous stirring for well dispersion of reactants and transferred into a Teflon lined autoclave. The autoclave was sealed, maintained at 180°C for 24h, and then cooled to room temperature naturally. A black precipitate was first retrieved from the solution; finally the as-formed precipitate was centrifuged, washed sequentially with deionized water and acetone and dried at 60°C for 2h. The same procedure was followed for second set of experiment (0.6048 g of Na_2MoO_4 and 0.38 g ($(NH_2)_2CS$)) and third set of experiment (0.6048 g of Na_2MoO_4 and 0.375 g Thioacetamide (CH_3CSNH_2)).

2.2 Characterization

The synthesized nanoparticles were characterized on a PANalytical powder diffractometer with CuKα radiation (λ=1.5418 Å). The morphologies, sizes and purity of the nanoparticles were analysed by SEM (Tescan-VEGA3 LMU) analysis.

Expt. No.	Precursor	Surfactant	Temp(^{0}C)	Phase	Morphology
1	0.6048 g Na_2MoO_4 + 0.16 g S_8	SDS	180	MoS_2	Tube Spheres
2	0.6048 g Na_2MoO_4 + 0.38 g ($(NH_2)_2CS$)	-	180	MoS_2	Clusters of Threads
3	0.6048 g Na_2MoO_4 + 0.375 g (CH_3CSNH_2))	CTAB	180	MoS_2	Clusters of spongy cotton

Table -1: Characteristics of experiment performed

3. Results and discussion

MoS_2 powders were synthesized by hydrothermal method using 0.6048 g Na_2MoO_4 + 0.16 g S_8+ 0.5g SDS at 180 ^{0}C for 24 hours. Table-1 shows the characteristics of experiments performed.

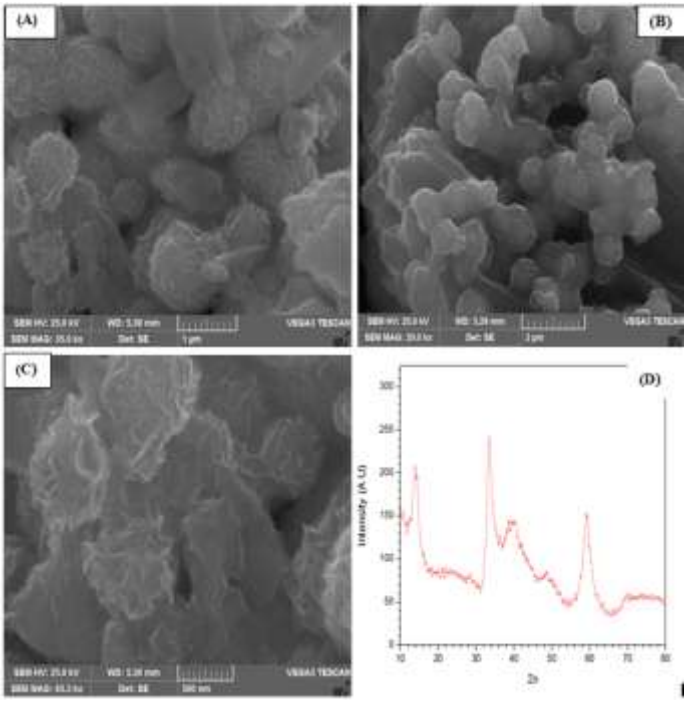

Fig -1: SEM images of MoS_2 nanoparticles. SEM image at a magnification of (A) 1µm (B) 2µm (C) 500 nm with dimensions (D) XRD of MoS_2 nanoparticles

Fig -1(D) shows the XRD pattern of synthesized powder. It can be seen that the diffraction peaks are indexed as MoS_2. Fig -1 (A)-(C) shows the SEM images. It can be seen that many tube like spheres like nano architectures with aggregation are present. Further, these images show the uniform nanoparticle clusters. The aggregation can be attributed to the surfactant SDS which aids in self-assembly of nanoparticles. The end to end growth of nanoparticles helped in the formation of clusters of MoS_2 architectures.

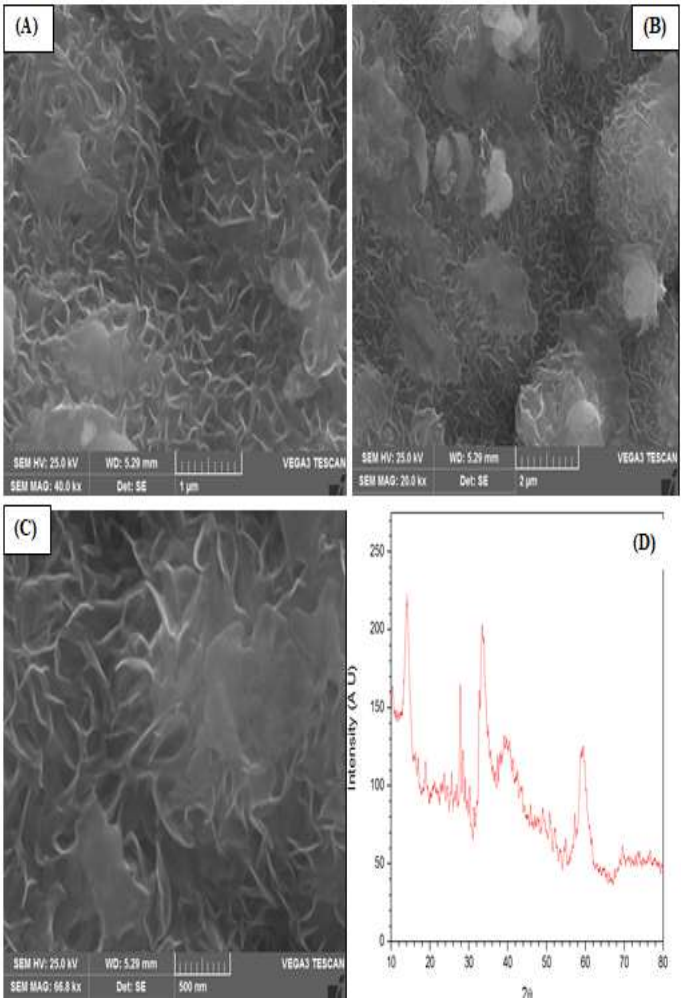

Fig -2: SEM images of MoS_2 nanoparticles. SEM image at a magnification of (A) 1µm (B) 2µm (C) 500 nm with dimensions (D) XRD of MoS_2 nanoparticles

Fig -2(D) shows the XRD pattern of synthesized powder using 0.6048 g Na_2MoO_4 + 0.38 g ($(NH_2)_2CS$))) without surfactant at 180 ^{0}C for 24 hours. It can be seen that the diffraction peaks are indexed as MoS_2. Fig -2 (A)-(C) shows the SEM images. It can be seen that spheres of spongy cotton like nano architectures agglomerated elongated nanoparticles.

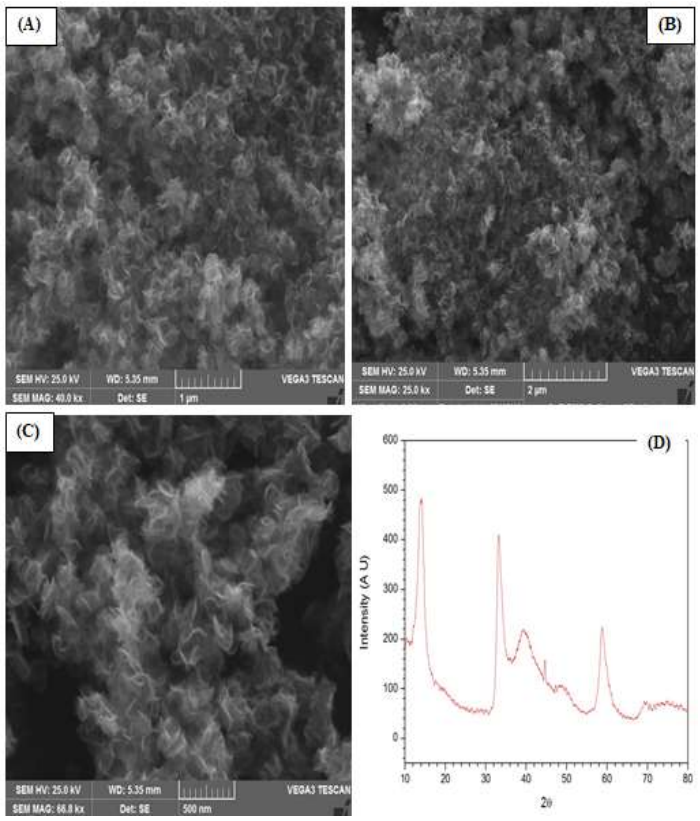

Fig -3: SEM images of MoS_2 nanoparticles. SEM image at a magnification of (A) 1µm (B) 2µm (C) 500 nm with dimensions (D) XRD of MoS_2 nanoparticles

Fig -3(D) shows the XRD pattern of synthesized powder using 0.6048 g Na_2MoO_4 + 0.375 g (CH_3CSNH_2)) + 0.05 g CTAB at 180 ^{0}C for 24 hours. It can be seen that the diffraction peaks are indexed as MoS_2. It can be seen that clusters of spongy cotton like nano architectures agglomerated elongated nanoparticles. It can be inferred that without surfactants there would be formation of clusters. Also, these clusters of nanoparticles are uniform. However, the aggregation is not much pronounced as compared to the addition of SDS. Hence it can be concluded that the SDS results in the agglomeration of nanoparticles giving rise to nanoclusters of large sizes. CTAB, being cationic surfactant caps MoS_2, inhibits the lateral growth of MoS_2 clusters and helps reduce agglomeration of nanoparticles.

3. CONCLUSIONS

In summary, the effect factors on morphologies of MoS_2were investigated. The Results showed that the morphologies of MoS_2 were obtained by hydrothermal method at 180 ^{0}C for 24 hours by varying sulphur precursors. Morphologies varied from tube spheres shape to cluster of threads to clusters of spongy cotton, which is meaningful to obtain three kinds of morphologies to meet the different applications demand in other field.

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