HYDROTHERMAL SYNTHESIS OF 2D NANOMATERIALS

A PROJECT REPORT

SUBMITTED IN PARTIAL FULFILMENT

OF THE REQUIREMENTS FOR THE AWARD OF THE

OF

DEGREE OF MASTER OF SCIENCE

IN

APPLIED PHYSICS

Submitted by:

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MAY, 2022

DECLARATION

We hereby certify that the work which is presented in the Major Project-Il/Research Work entitled in fulfilment of the requirement for the award of the Degree of Masters of Applied Physics in and submitted to the Department of Applied Physics Delhi Technological University, Delhi is an authentic record of my/our own, carried out during a period from me August 2021 to May 2022 under the supervision of Prof Nitin K Puri.

The matter presented in this report/thesis has not been submitted by us/me for the award of any other degree of this or any other Institute/University.

Purvi Khanna
2K20/MSCPHY/24

SUPERVISOR CERTIFICATE

To the best of my knowledge, the report comprises original work. It has not been submitted in part or whole for any other Course/Degree to this University or elsewhere as per candidate's declaration.

Place:

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Prof. Nitin K Puri

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ABSTRACT

Two-dimensional (2D) materials have received a considerable recognition due to their interesting properties and promising uses. A typical layered semiconductor material, $MoSe_2$ is a narrow bandgap semiconductor material and belongs to the family of Transition Metal Dichalcogenides (TMDs). This task uses a cheap, scalable, and simple hydrothermal synthesis method to synthesize $MoSe_2$ with and without surfactant. The prepared samples are analysed by techniques such as X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) Spectroscopy and Scanning Electron Microscopy (SEM), Energy dispersive X-ray Spectroscopy (EDS). The results obtained from the characterization techniques suggest the successful synthesis of $MoSe_2$ nanosheets which have potential applications in gas sensors.

INTRODUCTION

Transition Metal Dichalcogenides (TMDs) have been fondly studied over the past decade among many researchers because of their tuneable bandgap and excellent chemical and structural properties. The rich family of 2D nanomaterials include transition metal dichalcogenides (TMDs) with the chemical formula TZ_2 in which T is a transition metal atom (Mo, W) and Z is a chalcogen atom (S, Se, or Te). Single layered TMDs have attracted the attention due to the optical and electronic properties are unique. High surface to volume ratio 2D nanomaterials is a captivating feature of these materials, which make them suitable candidate for sensing applications. While MoS2 has been explored much, few studies are there with the synthesis of $MoSe_2$ with and without surfactant.

Amongst various synthesis methods employed for synthesis of $MoSe_2$ with and without surfactant hydrothermal method is considered as the best method as it involves low temperatures compared to other physical and chemical techniques.

To date, several surfactants including hexadecyl trimethylammonium bromide (CTAB), polyvinyl pyrrolidone (PVP), the copolymer Triblock (P123), etc. have been explored to vary the morphology of 2D nanomaterials. These surfactants help to achieve the fine tuning of morphology and thus better performance for varied applications. To the best of our knowledge, no reports are there studying the role of dual surfactants (SDS and acetic acid) in varying the morphology of $MoSe_2$ [6,9,10].

EXPERIMENTAL SECTION

Firstly 0.5mmol ammonium molybdate $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ was dissolved in suitable solvent with constant stirring and Selenium solution was prepared and added to the above solution continuing the stirring. After mixing the solutions well, it is transferred into autoclave for twenty-four hours at 220 ℃. A black precipitate was collected which was then further washed with DI water and ethanol thrice. The final products are dried in vacuum oven for overnight.

The same samples were also prepared by adding appropriate amount of single and dual surfactants.

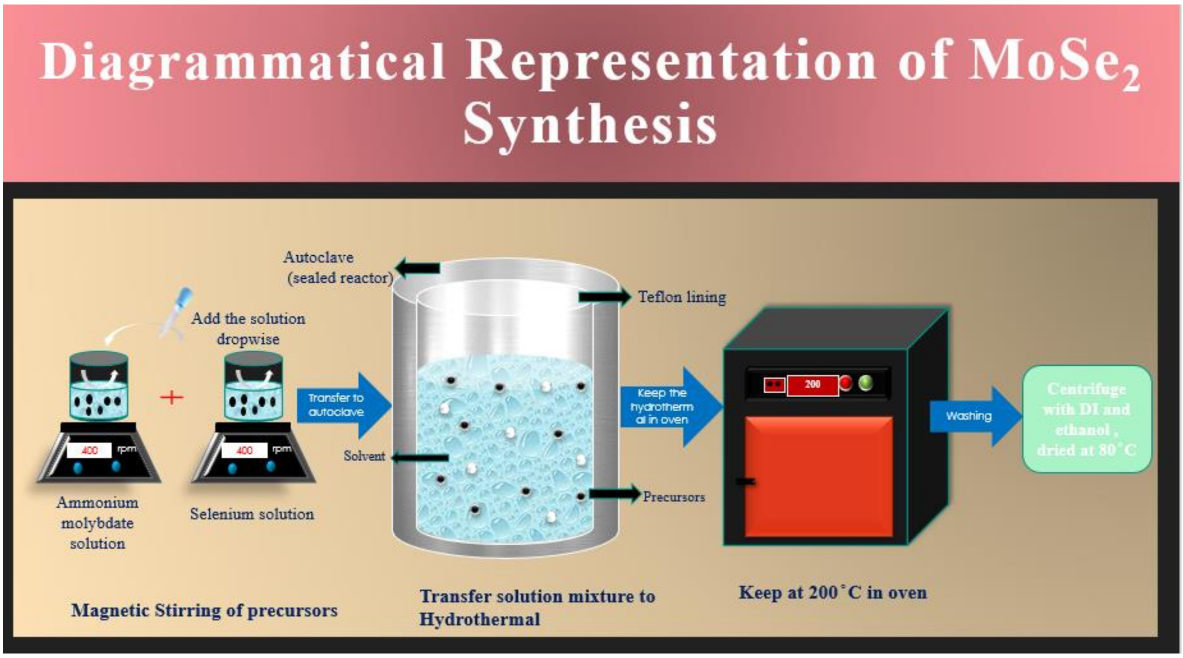

Figure 1 Experimental process of synthesis

RESULTS AND DISCUSSIONS

1. X-Ray Diffraction (XRD)

X-ray diffraction analyses the structure of the material. Basically, the atomic planes within a crystalline solid act as the diffraction grating for the X-rays, enabling the measure of their respective distances using Bragg's Law. XRD was done for the prepared samples.

All the peaks are consistent with the hexagonal $MoSe_2$ structure (JCPDS No. 17-0887). While the XRD peaks of $MoSe_2$ with single surfactant shift to lower angle direction. The reason could be be the oxygen incorporation between the layers of $MoSe_2$ which decreased the angle.

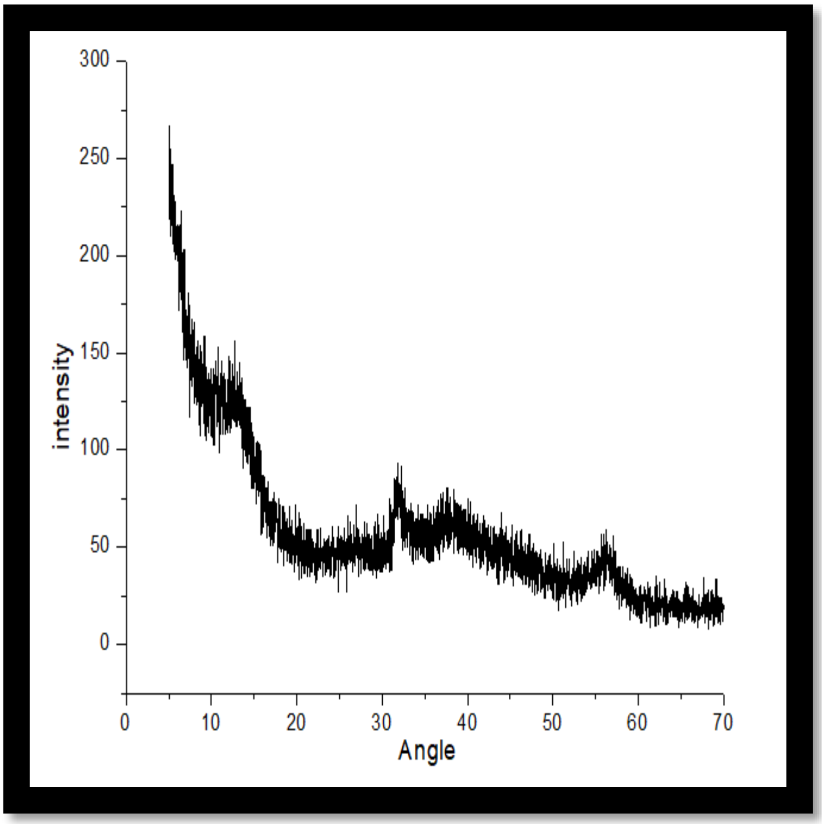

Figure 2 XRD plot for pristine $MoSe_2$

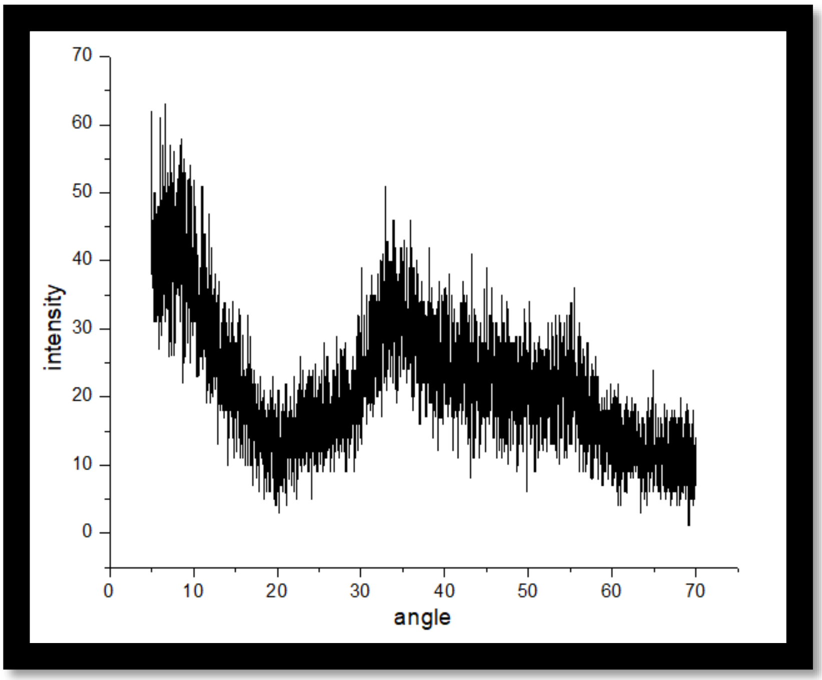

Figure 3 XRD plot for single surfactant sample of $MoSe_2$

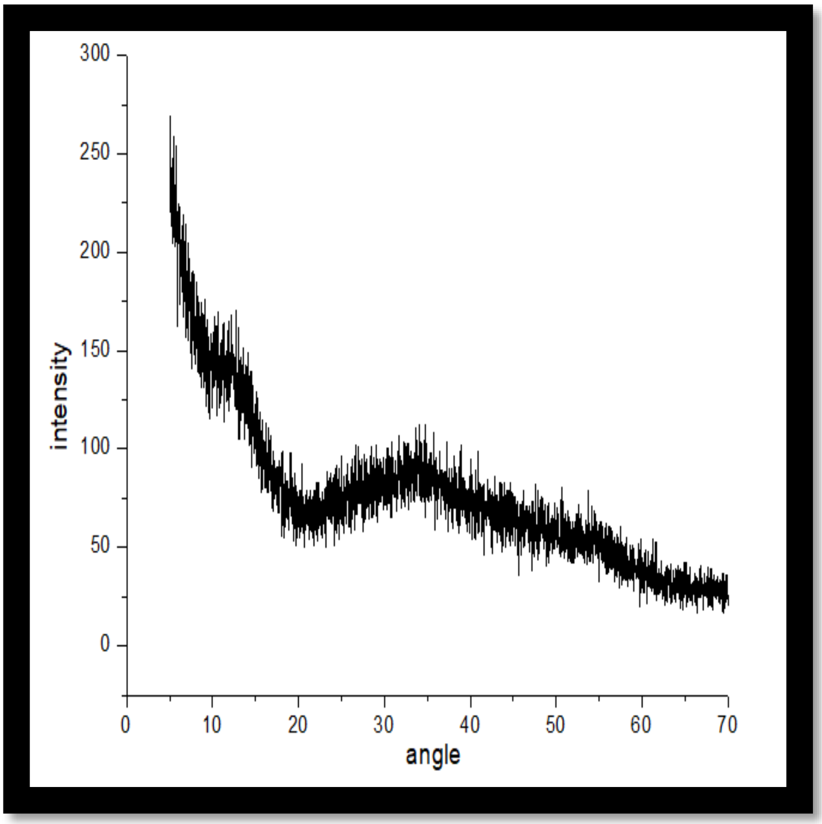

Figure 4 XRD plot of dual surfactant sample of $MoSe_2$

2. Fourier Transform Infrared Spectroscopy (FTIR).

FTIR spectroscopy is done to find the presence of functional groups present in MoSe2 sample. And the peaks corresponding to the FTIR spectrum are shown in Table T1.

Table T1:

Wavenumber (cm^{-1}) in pristine sample	Wavenumber (cm^{-1}) in sample with surfactant	Band assignment	Reference
936	1067	C-O stretching vibration	3
1242	1378	C-N vibration	1,2
1596	1610	C=O stretching vibration	4
2349	2353	C=O asymmetric stretching vibration	3
2677	2988	C-H asymmetric stretch vibration	4
	3685	O-H stretching vibration	4

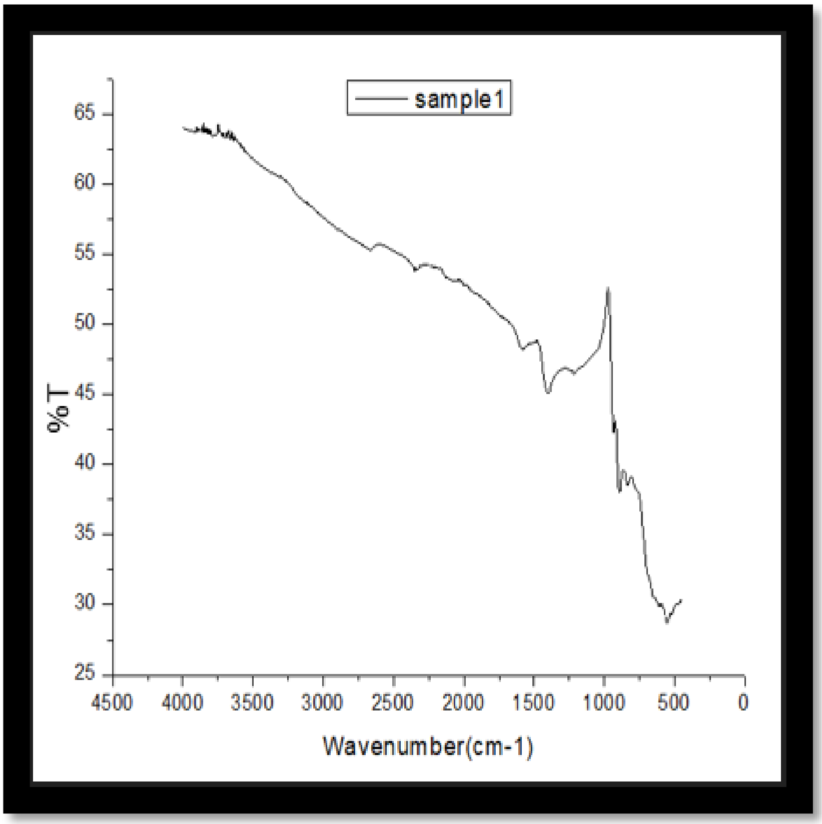

Figure 5 FTIR plot of pristine $MoSe_2$ sample

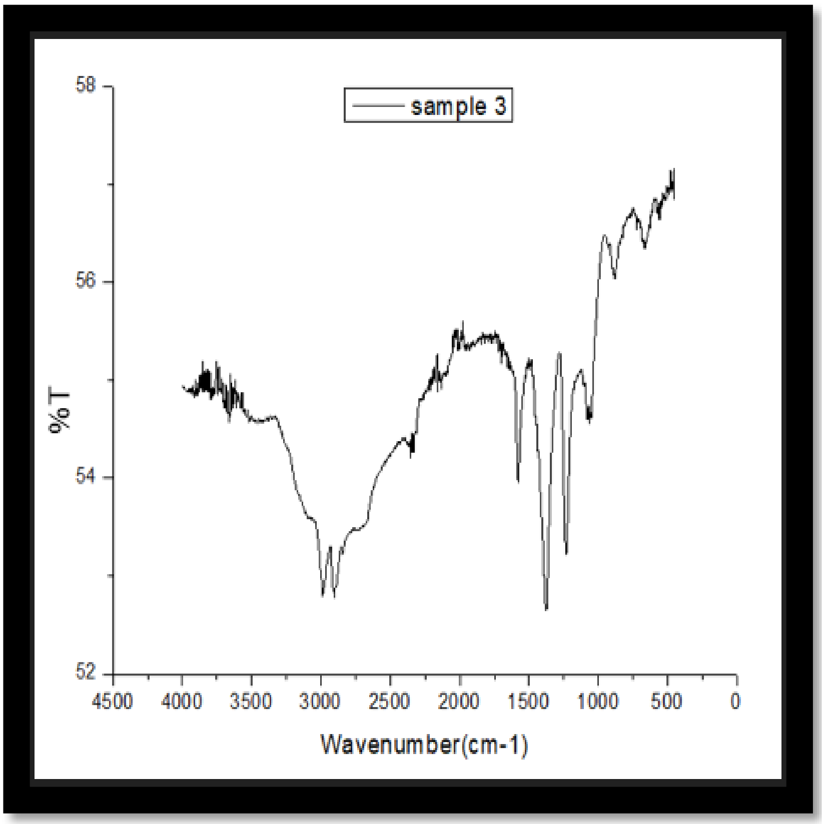

Figure 6 FTIR plot of single surfactant sample

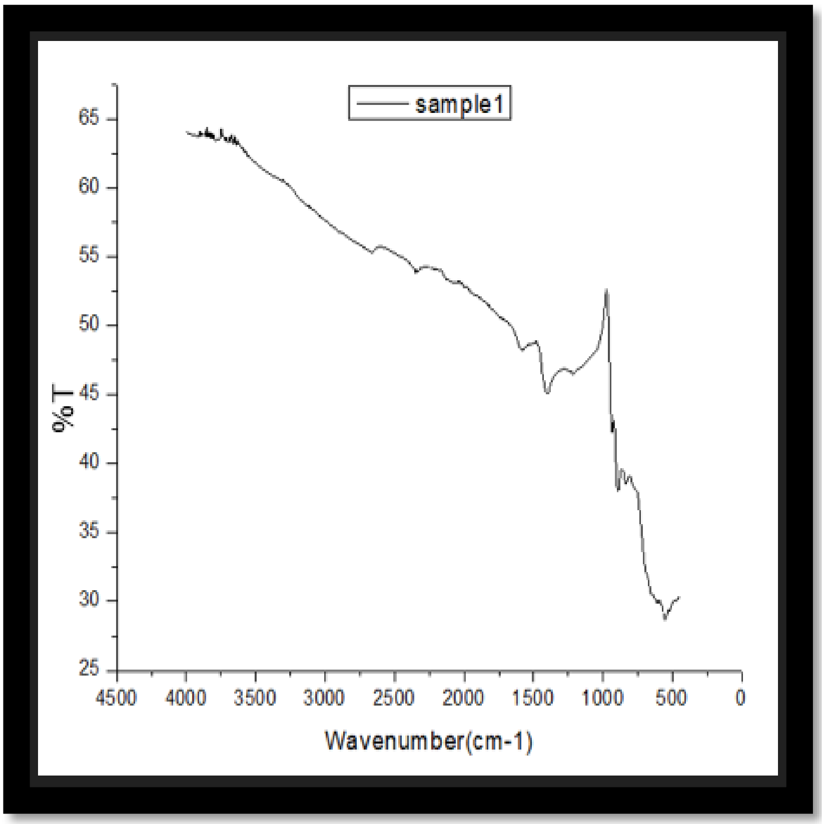

Figure 7 FTIR plot of dual surfactant sample

3. Scanning Electron Microscopy (SEM) and Energy dispersive X-ray Spectroscopy (EDX)

The SEM images of $MoSe_2$ is shown in figure 6-8. The images clearly depict the formation of nanosheets for pristine MoSe2 sample, while in sample with single surfactant agglomeration can be seen (figure7). Figure 8 shows the sample with dual surfactant. It can be seen that in this sample some nanosheets along with agglomerated clusters are formed. Hence Sample with single surfactant and dual surfactant need more optimisation.

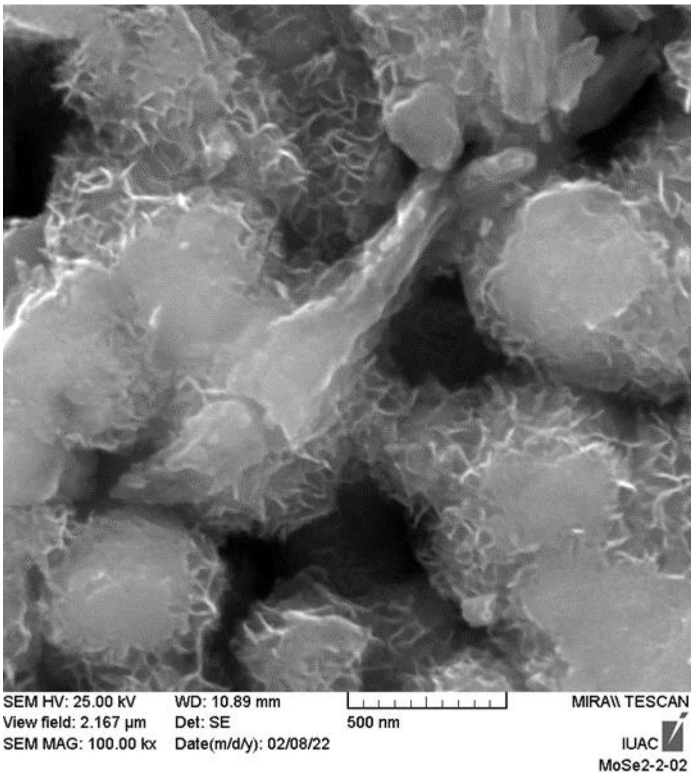

Figure 8 SEM image of pristine $MoSe_2$ sample

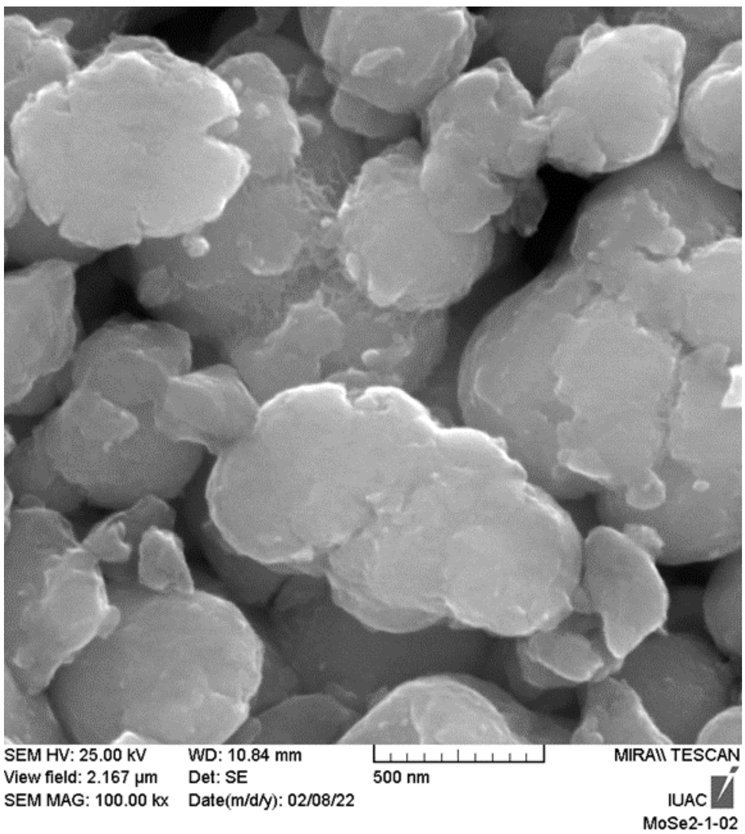

Figure 9 SEM image of single surfactant sample

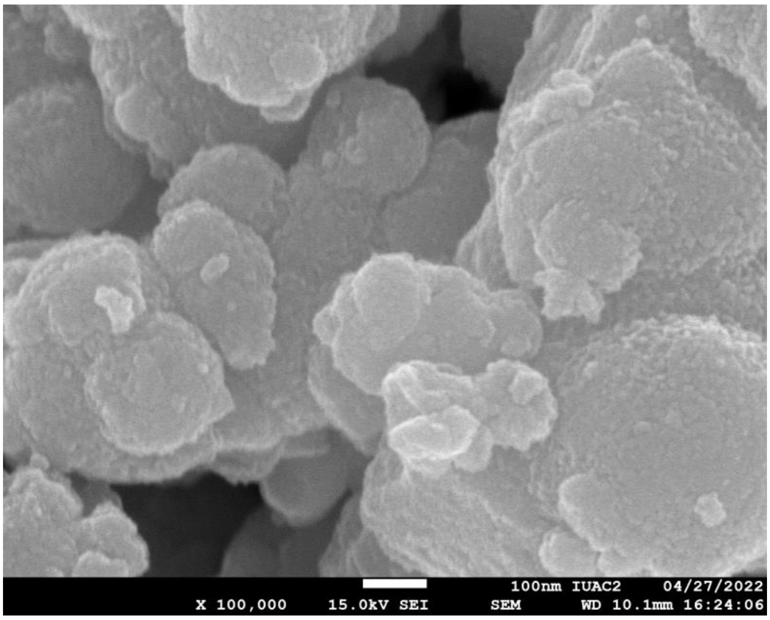

Figure 10 SEM image of dual surfactant sample

The elemental composition of the MoSe2 analysed by EDX spectrum. The spectrum clearly shows the presence of molybdenum and selenium.

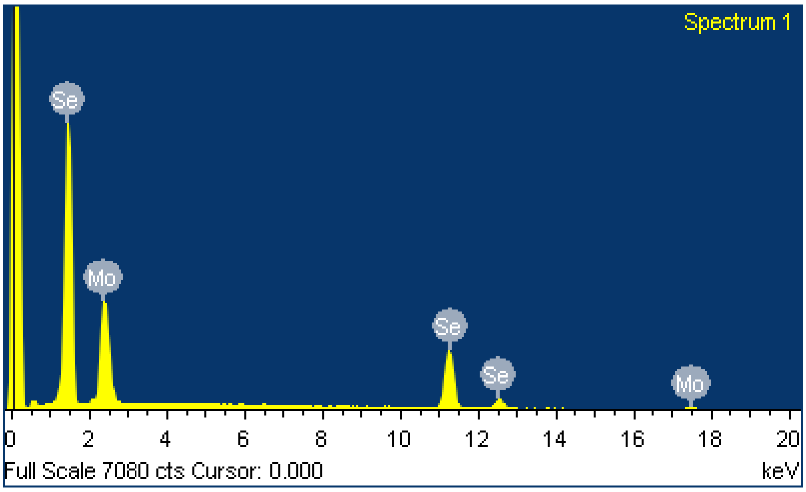

Figure 11 EDS Spectrum of the sample

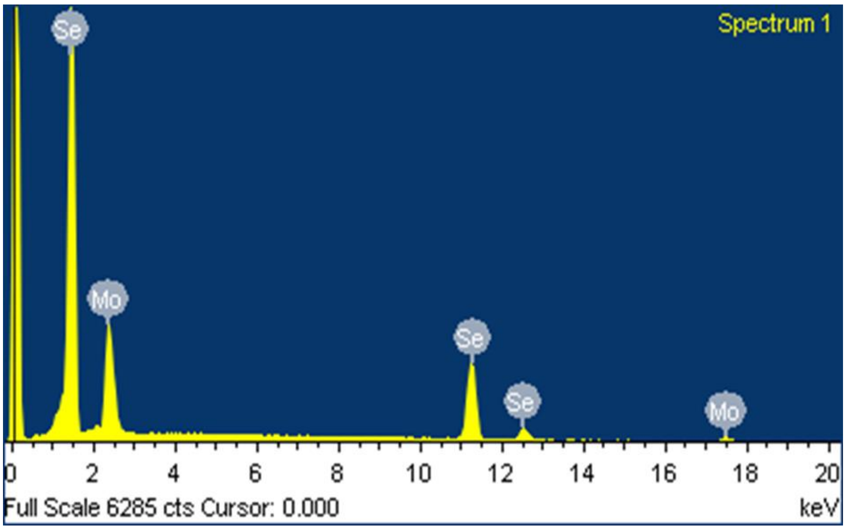

Figure 12 EDX spectrum of single surfactant sample

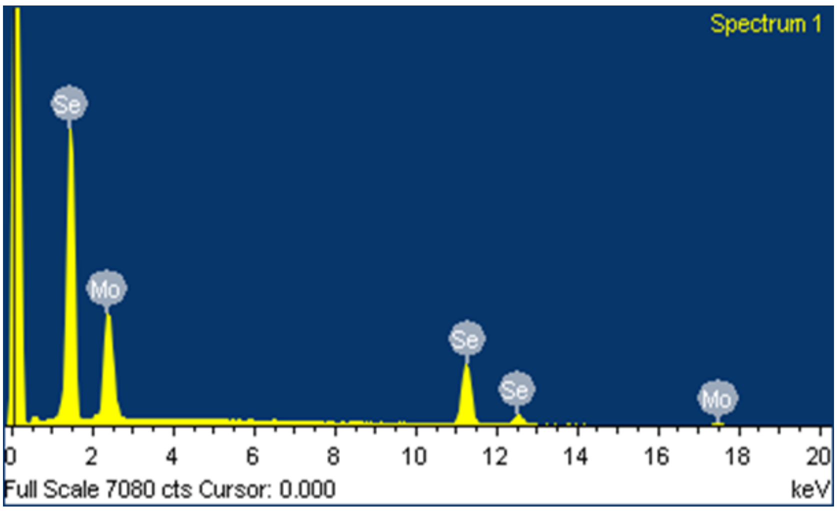

Figure 13 EDX spectrum of dual surfactant sample

CONCLUSION

We have successfully synthesised pristine $MoSe_2$ via facile a low-cost, scalable and hydrothermal/ solvothermal method. The results obtained from the characterization techniques suggest that $MoSe_2$ with and without surfactant needs further optimization. The prepared samples can be further explored for gas sensors.

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