

In-situ generation of Silver Nano-particles on Langmuir-Blodgett Film Template of MoS₂ : Fabrication and Its Application in SERS

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Abstract

This paper is concentrated on the fabrication of next generation SERS active scaffold through *in situ* generation of silver nanoparticles on MoS₂ matrix in Langmuir-Blodgett (LB) film. The plasmonic “hot spots” formed over the as prepared substrate works as effective agents for localization of electromagnetic field owing to huge enhancement in Raman Signals. The as prepared Ag- MoS₂ substrate was meticulously characterized and the efficiency of the as prepared substrate as a SERS scaffold was investigated using Raman probe Rhodamine-6G molecules down to ultrasensitive concentrations. The as prepared SERS active platform was engaged further for detection of biologically significant epinephrine at ultralow concentrations down to 10⁻¹¹ M. In the current study the as fabricated sensor divulges its precision and effectivity efficiently. Thus, in future this scaffold can be used as a next-generation sensor for detections of chemical and bio-chemicals even down to negligible quantities.

INTRODUCTION

The uniqueness of Two-dimensional (2D) materials in physical, chemical properties enticed in engaging a lot of research of late. Trans metal di chalcogenides (TMDCs) like MoS₂, VS₂, MoSe₂, ReS₂, WS₂, WSe₂ like variants are broadly engaged in developing new-generation wearable electronic and optoelectronic sensors and smart gadgets [1–51] Molybdenum disulphide (MoS₂), a significant member of 2D-TMDCs family, possess multilayered sandwich structure of Molybdenum and sulphur interconnected with interlayer weak van der Waal force. Recent explorations indicated these materials alone or modified with plasmonic nano-particles can act as excellent agents as SERS active platforms which in term can be utilised as excellent tool for trace detection of different chemicals, bio-chemicals.

Keeping these things in cognizance, here we present a proforma in fabrication of cost-effective SERS active scaffold through *in situ* growth of silver nanoparticles (AgNPs) on the Langmuir-Blodgett (LB) film template of MoS₂ nanoparticles. The surface morphologies and bonding configurations of Ag-MoS₂ substrates have been explored from FESEM and XPS Studies. The SERS responsiveness of the Ag-MoS₂ substrate has been substantiated by using Raman probe Rhodamine-6G (R6G) molecule even down to trace levels. The reason behind the enhancement of Raman signals has also been addressed in the present report. The substrate has been further utilized for sensing neuro-transmitter molecule epinephrine (EP) at ultrasensitive concentrations. To the best of our knowledge this work may be considered as one of the first reports where *in situ* grown AgNPs on the LB film scaffold of MoS₂ have been successfully employed as an effective SERS active sensor.

1. EXPERIMENTAL SECTION

1.1. Synthesis of MoS₂ Nanoflowers and Fabrication of LB Films :

The synthesis methodology of MoS₂ nanostructures using a one-step hydrothermal reaction were described in earlier study reported elsewhere[52, 53]. In brief, aqueous solution of ammonium molybdate tetrahydrate (NH₄)₆Mo₇O₂₄·4H₂O (~ 75 mg/ml) and thiourea [CH₄N₂S] (~ 37.5 mg/ml) were assorted to make the final volume ~ 70 ml. The blend was then transferred into 100 ml Teflon lined autoclave for hydrothermal treatment at 180 °C for 24 h. After the completion of the hydrothermal reaction the black coloured residue was collected and washed numerous times with Milli-Q water and ethanol simultaneously and dried in tube furnace in inert gas atmosphere to avoid any oxidation. The powder was then kept in vacuum enclosure for future investigations.

For LB deposition of the as synthesised MoS₂ a computer coupled LB system was used to deposit the particulates in Y-type deposition. Firstly, polytetrafluoroethylene trough has been cleaned repeatedly with ethanol, chloroform and Milli-Q water. The air-water interface was cleaned with vacuum aspirator to remove any unwanted particles from the surface numerous times until the surface pressure variation reaches 0.01 mN/m under compression. For the film deposition, chloroform was used for dispersion of the MoS₂ at Low concentration (~ 0.18 mg/ml). The dispersion was gently spread with a quartz syringe as if the drop gets attenuated on the upper surface of the air-water interface. A 150µl MoS₂ dispersion was gently spread onto the surface and left for 2.5 hours. The interface was then compressed with movement of the barriers at a speed of 5 mm/min in order to form a dense LB layer in the interface and a single stroke has been used to transfer MoS₂ on a vertically placed glass substrate at a target pressure of ~ 17mN/m. These as prepared LB films have been carefully preserved in vacuum and dried for future use.

1.2. Decoration of Silver Nano-particles on the as prepared LB Film of MoS₂:

For the decoration of AgNPs on the LB film template, three sets of as fabricated LB films of MoS₂ (MoS₂-LB) were dipped in the aqueous solution of AgNO₃ (~ 10⁻³ M) and were exposed to UV radiation for various time scales ranging from 1 to 4 hours. Three different sets of LB films were removed from AgNO₃ solution after 1, 2 and 4 hrs of exposure times (ETs) and kept in vacuum desiccator for further characterisation. The films so accomplished after 1, 2 and 4 hrs of ETs are henceforth designated as substrate S1, S2 and S3 respectively. The change of the colour of the AgNO₃ solution during UV exposure from silvery grey to yellowish and subsequently to brownish yellow with time confirms formation of Ag-Nano particles primarily.

2. RESULTS AND DISCUSSION

2.1. Characterisation of the as prepared MoS₂ Nanoflowers (NFs):

Figure. 1(a) and (b-f) shows FESEM and HRTEM images of the as synthesized MoS₂ in the form of powder was captured at different scales of magnifications. The FESEM image, as shown in Fig. 1 (a) exhibits flower like arrangements with diameter ~ 800 nm having MoS₂ flakes as the petals. The corresponding HRTEM image [Fig. 1 (b)] shows noticeable variation in contrast revealing the layered nature. [52, 54]. The HRTEM further shows slightly curved edges signifying thin layered architecture of the as prepared sample.. HRTEM images elaborate numerous curled traces, ripplocations and parallel fringes along the edge sites indicate the weak Van Der wall forces between layers [52, 55–57]. The interlayer spacing has been estimated to ~ 0.68 nm along (002) plane marked in Figure. 1(d) in accordance with the existing literatures reported elsewhere [56, 58, 59]. From the images it is found that the material is ~ 5–9 layers in thickness. HRTEM images further show defect rich arrangements along the edges and discontinuity on the basal and edge planes of the MoS₂ NFs from the Fig. 1(c-f) (highlighted in square boxes).

Characterization of MoS₂ LB Film:

The FESEM image of the as prepared MoS₂-LB film is shown in Fig. 2. The electron microscopy image and its elemental mapping analyses reveal the presence of molybdenum (Mo) and sulphur (S) indicative of MoS₂ with lateral dimensions in the range spanning from ~ 300 to 700 nm. The image shows flaky nature of the as deposited MoS₂ and also shows curl edges exhibiting its ultrathin nature.

2.2. Morphological Characterisation of the Ag-MoS₂ Substrates:

The morphologies of Ag-MoS₂ substrates have been observed by FESEM which are shown in Fig. 3 (A-I, A-II); (B-I, B-II) and (C-I, C-II) depicting the substrates S1, S2 and S3 respectively. In the case of S1 substrate clear flakes of MoS₂ can be observed with very few adsorbed AgNPs into the petals. The corresponding EDX spectrum has been shown in Fig. S1 (a). Upon intermediate exposure of UV (~ 2 hrs) in case of substrate S2 the morphology of the substrate is remarkably different from S1 and is marked by the appearance of aggregated domains of plasmonic AgNPs [Fig. 3(B-I, B-II)]. This result thus signifies reduction of Ag⁺ ions upon UV exposure on AgNO₃ soaked over MoS₂-LB film substrate. The corresponding EDX [Fig. S1 (b)] shows the elemental analysis of the S2 substrate. Prolonged exposure of UV (~ 4 hrs.) results in dense agglomeration of AgNPs, as being recorded for the S3 substrate in the FESEM images [Fig. 3(C-I, C-II)]. These in situ grown AgNPs rich domains formed aggregated domains over the LB film template which can act as probable “hot spots” in the enhancement of Raman signals of probe molecules. The EDX spectrum shown in Fig.S1(c) marks the presence of the constituent elements in S3 substrate.

As S3 substrate is seen to harbour maximum agglomerated domains of AgNPs, hence the elemental surface mapping of the S3 substrate has been further investigated. The results are shown in Fig. 4. Akin FESEM images [Fig. 3(C-I, C-II)], the surface mapping image along with its elemental mapping also

shows dense aggregated domains of AgNPs together with the presence of its constituent elements Mo, S, Ag distributed evenly throughout the entire substrate.

The reason behind the aggregation of the AgNPs on the MoS₂ flakes may probably be rationalized as follows. From our earlier studies we have shown that the as prepared MoS₂-LB film is sulphur rich and hence shows p-type character[52]. Due to exposure of UV radiation, the Ag⁺-ions in AgNO₃ may get attracted to the sulphur rich domains which act as nucleation centres wherein reduction occurs leading to formation of Ag⁰ on the substrate.[60–63]

In order to verify the exclusive presence of Ag⁰ and the identification of the chemical composition and the interaction of AgNPs with MoS₂ in MoS₂-Ag S3 substrate, the X-ray photoelectron spectroscopy (XPS) studies have been performed. The narrow scan spectra in the regions of interest are shown in shown in Fig. 5. The High resolution narrow scan spectrum in the BE window of ~ 220–240 eV marks prominent peaks centered ~ 226 and 231 eV owing to the doublet Mo 3d_{5/2} and Mo 3d_{3/2} respectively for the oxidation state Mo⁴⁺. The peaks are red shifted to higher binding energy state than the MoS₂-LB films reported in our previous work due to adsorption of AgNPs[52]. Interestingly these bands upon deconvolution marks the presence of both 1T and 2H phases with slightly up shifted binding energy positions signifying n-type doping. Moreover, the percentage contribution of the phases 1T and 2H upon Ag interaction in contrast to the LB films reported elsewhere is found to be ~ 60 %, 0 % fr 1T, 2H phases respectively. Thus, 1T phase dominance in the substrate is evident vis-à-vis 2H dominance in LB film template. Presence of both the phases and the upshift of the BE in the Ag-S3 substrate is further confirmed by the presence of XPS peaks in the BE window of ~ 158–166 eV. High resolution XPS spectrum of Ag 3d indicate two distinct peaks centered at ~ 367.7 and 373.84 eV. These peaks are ascribed to Ag 3d_{5/2} and Ag 3d_{3/2} respectively.[64–69] Moreover, transfer of electrons from the sulphur rich MoS₂ LB film to the AgNPs result in the reduction of unnaturalized Ag⁺ ions to Ag⁰ atoms thereby favouring the overall population of the naturalized species on the Ag-MoS₂ S3 substrate.

Interestingly, closer inspections of the FESEM images [Fig. 3A-(I, II), B-(I, II) and C-(I, II)] of S1, S2 and S3 reveal fractal like behaviour in their respective morphologies. To verify this observation, the fractal dimensions of all the substrates (S1,S2 and S3) have been estimated following the box counting method. In this evaluation method, square boxes with a specific size of S (in pixel) are used to fill in the investigated area and F_q be the number of boxes to fill such area. F_q obeys the following relation

$$F_q = S^D$$

where D is the fractal dimension. The fractal dimensions for all the substrates have been estimated from the linear fit of the ln (F_q) versus ln (S) plot. From Fig. 6 it is seen that the estimated fractality for the substrates S1, S2 and S3 are ~ 1.23, 1.27 and 1.40 respectively which indeed indicates the fractal nature of the substrates. From the calculations it is clearly seen that S3 substrate shows maximum fractal dimension ~ 1.40, whose FESEM image shows aggregated plasmonic domains of AgNPs.

2.3. Study of SERS Efficiency of MoS₂-Ag substrate:

The efficacies of the as prepared substrates as SERS sensing platforms have been tested with the Raman probe R6G molecule. R6G at concentrations in the range of 10^{-8} M to 10^{-14} M concentrations have been adsorbed on the substrates S1, S2 and S3 respectively. The enhanced resolved SERS spectra have been depicted in the Fig. 7 (a-c). The SERS spectra of R6G adsorbed on the substrates show enhanced vibrational Raman bands at $\sim 611, 770, 1186, 1362, 1504, 1571,$ and 1650 cm^{-1} attributing to the characteristic vibrational signatures of R6G can be clearly seen even down to 10^{-14} M [70–74]. This may be owing to the dense AgNPs aggregation formed on the MoS₂ LB scaffold clearly visible from the FESEM images (vide ante, cf. Figure 3).

The plasmonic nanoparticle in synergy with charge transfer in MoS₂ may source such huge enhancement in Ag-MoS₂- S3 substrate. The enhancement factors (EFs) of the R6G molecules so absorbed on the substrates S1, S2 and S3 were estimated. The results are presented in Table S1. The outcomes clearly indicate the S3 substrate shows the best enhancement among the three substrates S1, S2 and S3. Extending the calculation of EF for all the concentrations of R6G molecules in case of S3 maximum EF found $\sim 1.46 \times 10^{10}$. Some EFs of the existing reports along with the as prepared substrate has been tabulated in the Table S2. The results clearly show the as prepared substrate is very much efficient as a SERS sensing scaffold.

To understand the reason behind the enhancement of SERS signals, probable charge transfer (CT) pathways with the energy dispositions are shown in the Fig. 8. Using an excitation laser source with excitation wavelength $\sim 532\text{ nm}$ ($E_{\text{ex}} \sim 2.33\text{ eV}$), the electrons in the LUMO of the probe R6G molecules undergoes molecular resonance and electrons can move to its HOMO orbitals. Considering the Ag-MoS₂ system the probable charge transfer (CT) may occur in a two-step process. Primarily the electrons from the HOMO level of R6G ($\sim -6\text{ eV}$) may be excited to the Fermi energy level of the Ag ($\sim -4.84\text{ eV}$) and subsequently the hot electrons from Ag may transit to the CB state ($\sim -4.4\text{ eV}$) of the MoS₂ through the Schottky barrier. [75–79] The CT interaction results in change of the polarizability in the system which in turn may contribute to the enhancement of Raman signals as shown in Fig. 7. Thus, the overall huge enhancement in the SERS spectra of probe R6G molecules adsorbed on Ag-MoS₂ substrate may be owing to the plasmonic LSPR effects of AgNPs [70, 73, 74, 80] in synergy with CT mechanism.

2.4. Epinephrine Detection at ultrasensitive concentrations:

Epinephrine is a naturally occurring catecholamine neurotransmitter (NT) playing a great role in human immunity responses in body. Epinephrine is one of the most abandoned catecholamine neurotransmitter along with dopamine and nor epinephrine. Metabolism of neurotransmitters is most important in human body which involves transmission of impulses in human central nervous system. SERS spectra of epinephrine adsorbed on the Ag-MoS₂ S3 substrate at ultrasensitive concentrations depicted in the Fig. 9. The epinephrine doesn't have functional groups having strong affinity of pairing among them

moreover, they simply get attached over the silver surface reducing analyte and SERS enhancer separation. Concentration dependent SERS spectra have been taken for concentration of aqueous solution of epinephrine ranging from 10^{-6} M to 10^{-13} M. Peaks have been found at $\sim 603, 640, 741, 780, 960, 1014, 1078, 1170, 1286, 1346, 1532, 1578, 1618 \text{ cm}^{-1}$. These Raman bands are similar that of previous reports [81–83]. Raman bands at $\sim 640, 741 \text{ cm}^{-1}$ are due to in plane and out plane ring deformation of CH_2 twisting. In plane ring deformation of catechol and aliphatic $-\text{OH}$ give rise to the bands at $\sim 1014, 1078, 1178 \text{ cm}^{-1}$. The Raman peak at $\sim 603 \text{ cm}^{-1}$ arises from asymmetric ring deformation of aliphatic $-\text{CH}_2$ twisting. H-O-C-H bending results in band at 1286 cm^{-1} . Other Raman bands ~ 1346 and 1532 cm^{-1} arise from CH-CH_2 bending and C-NH bending respectively. SERS signals from substrate are clearly resolved up to trace concentration even at 10^{-13} M.[81–83].

Wide range of methods for separation and determination of NT like HPLC, fluorescence, tandem mass spectrometry, capillary electrophoresis, chemiluminescence, amperometry, electrometry ion selective method, flow injection method is complex in process and NT sometime eludes at time of separation and processing moreover reproducibility of these techniques are less. SERS in contrast shows a better response due to simple, one click, non-destructible methodology having better sensitivity and reproducibility.

3. CONCLUSIONS

The prime focus of the paper is fabrication of novel SERS active hetero-structure using Langmuir-Blodgett (LB) films of MoS_2 flakes with surface modulation using in situ grown silver nano particles (AgNPs). The plasmonic arrangements of the AgNPs over the substrate facilitates gap plasmons. These interstitial active sites act as hot-sites owing to the overall enhancements of SERS signals. The SERS sensing efficacy has been thoroughly explored using Raman reporter molecule and the substrate exhibits remarkable spectral reproducibility and sensitivity. Epinephrine is used at trace concentrations to check the response of the substrate in practical applications. Thus, the as prepared SERS active sensor can be cast off as new generation SERS sensing scaffolds to detect different chemical and bio chemicals which will be the interest of our future explores.

Declarations

Conflict of interest:

There is no conflict of interest

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There are no such funding details to mention.

Author Contribution

Chayan Kumar Mitra: Writing-original draft, Visualisation, Methodology, Investigation, Formal analysis, Data curation, Conceptualisation. Surojit Pande: Investigation, Writing-review editing. Joydeep Chowdhury: Writing-original draft, Writing-review editing, Supervision, Visualisation, Formal analysis, Funding acquiring. All the authors were involved in preparing the final manuscript.

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Data Availability

Data sets generated during the current study are available from the corresponding author on prior reasonable request and not be shared publicly.

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Figures

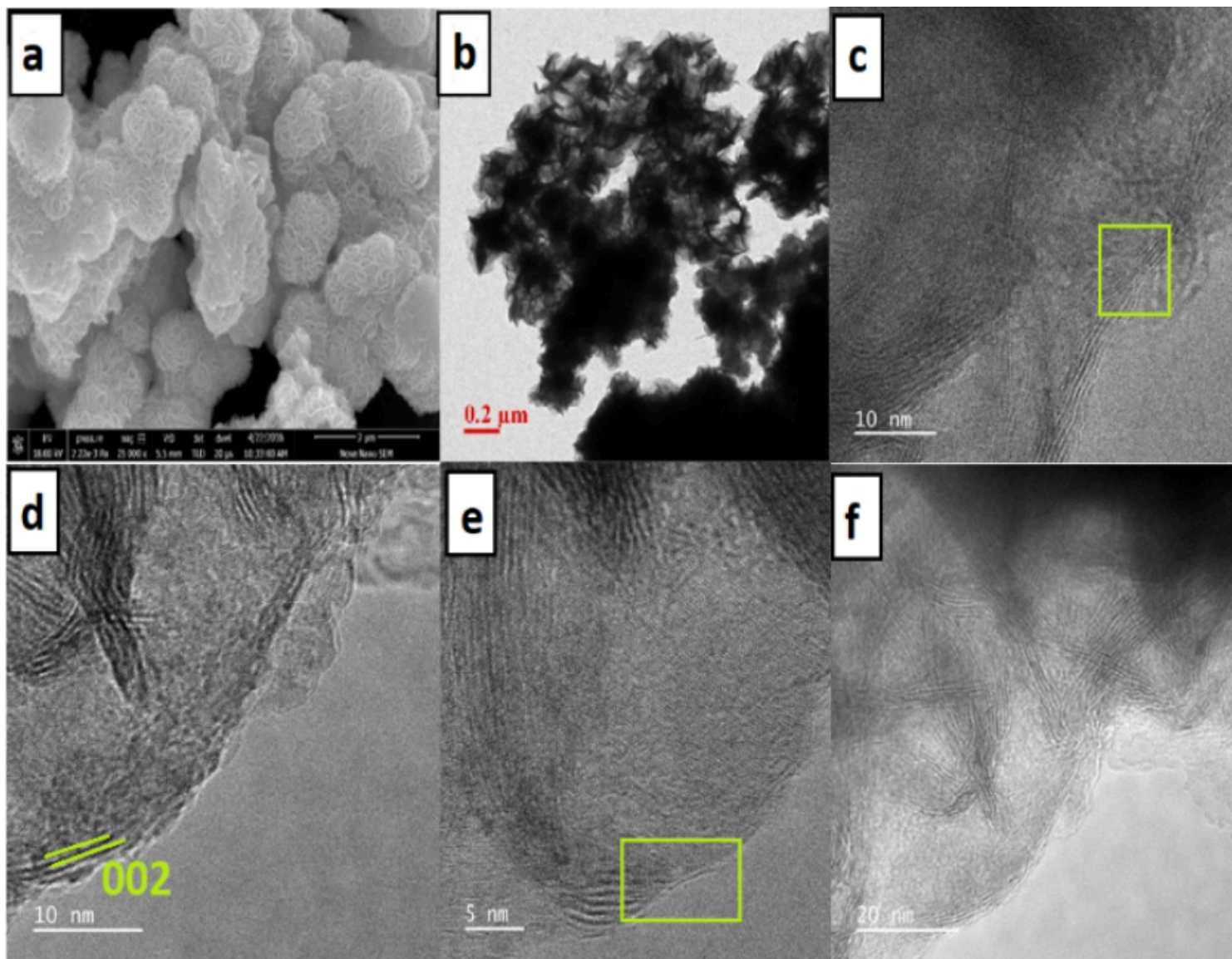


Figure 1

(a) FESEM, (b) TEM and (c-f) HRTEM of the as prepared MoS₂ NFs.

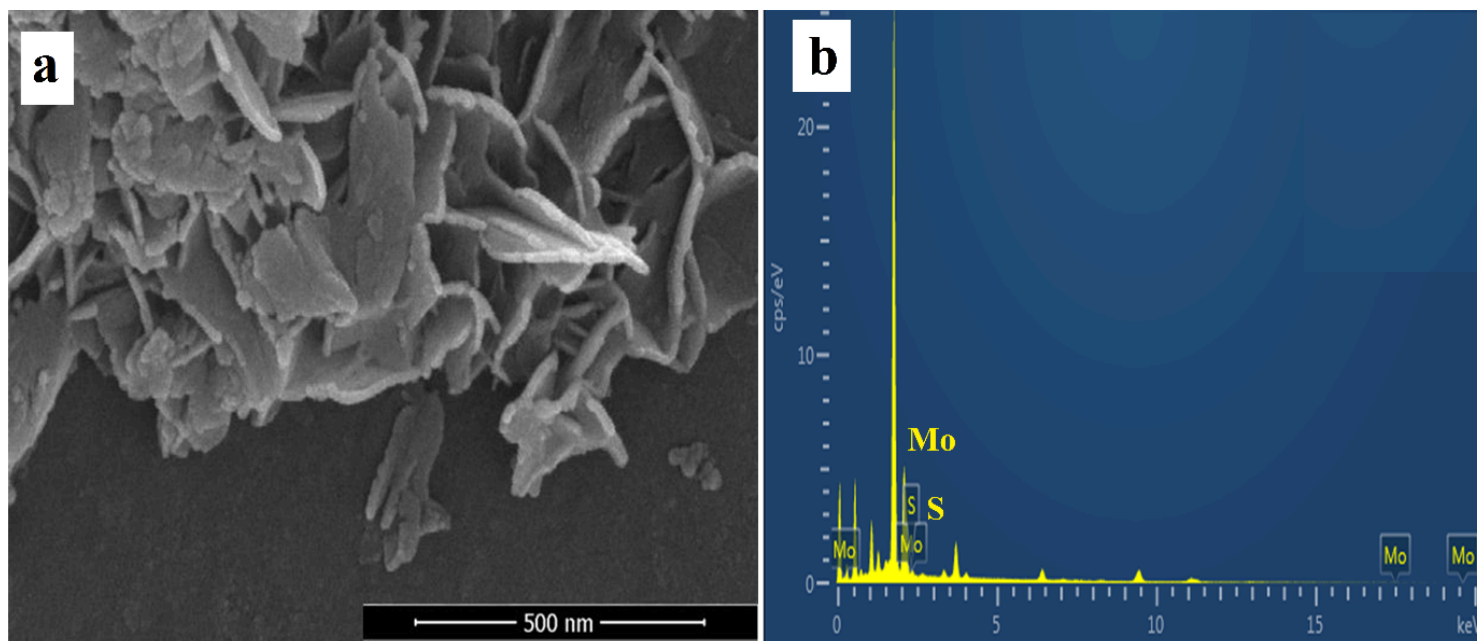


Figure 2

(a) FESEM image and (b) EDX spectra of as synthesized MoS₂ LB films

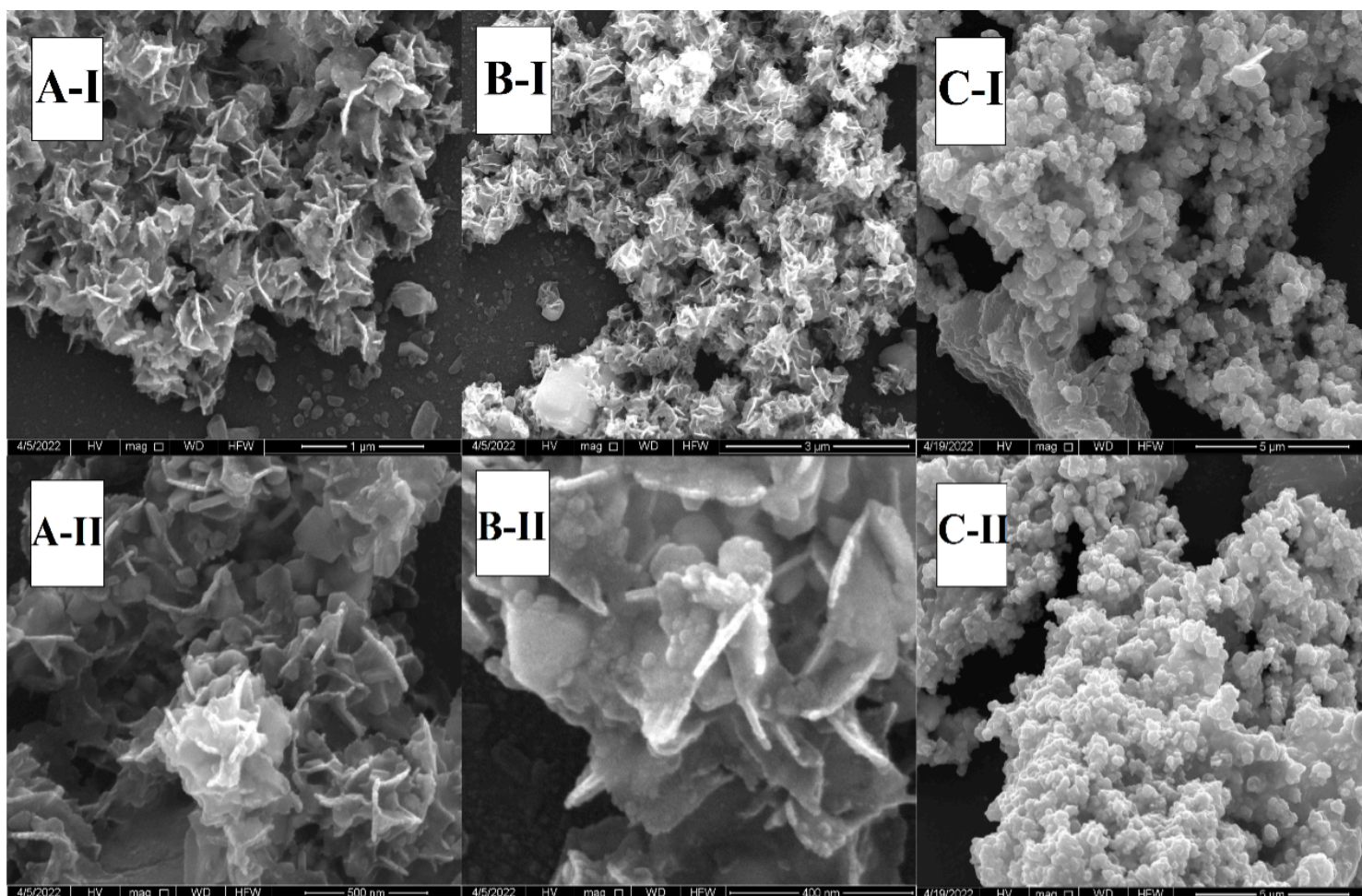


Figure 3

FESEM images A-(I,II), B-(I,II) and C-(I,II) of the as synthesised MoS₂-Ag substrates S1, S2 and S3 respectively.

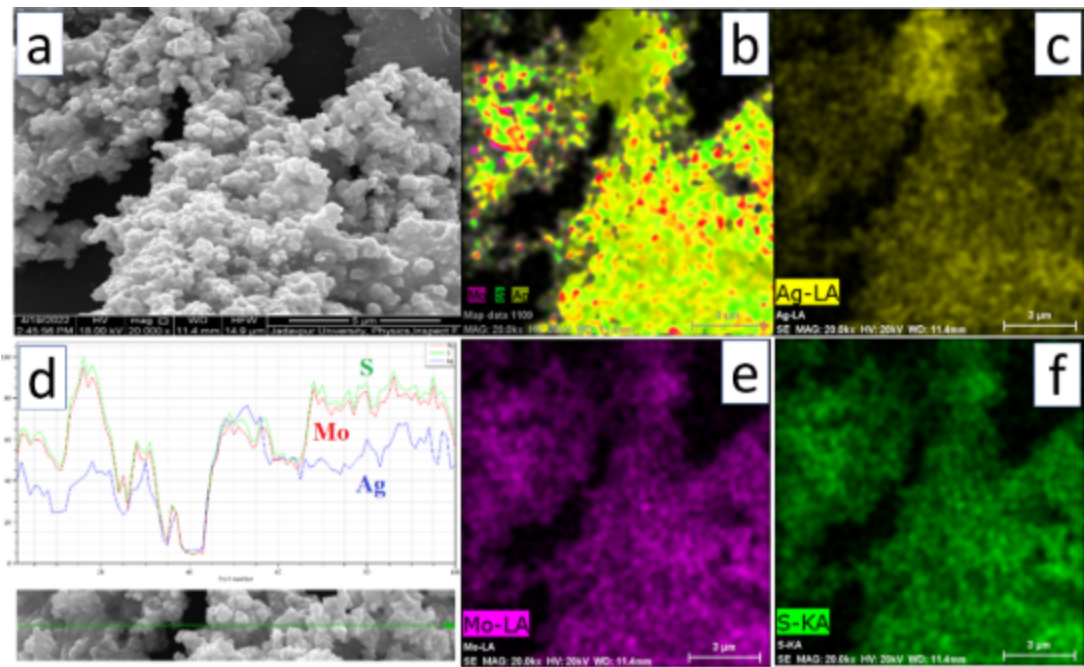


Figure 4

(a) FESEM image, (b) EDX mapping, (c) elemental mapping of Ag, (e) elemental mapping Mo, (f) elemental mapping S the as synthesised S3 substrate and (d) the line profile of the substrate with % presence of different elements along the selected trace.

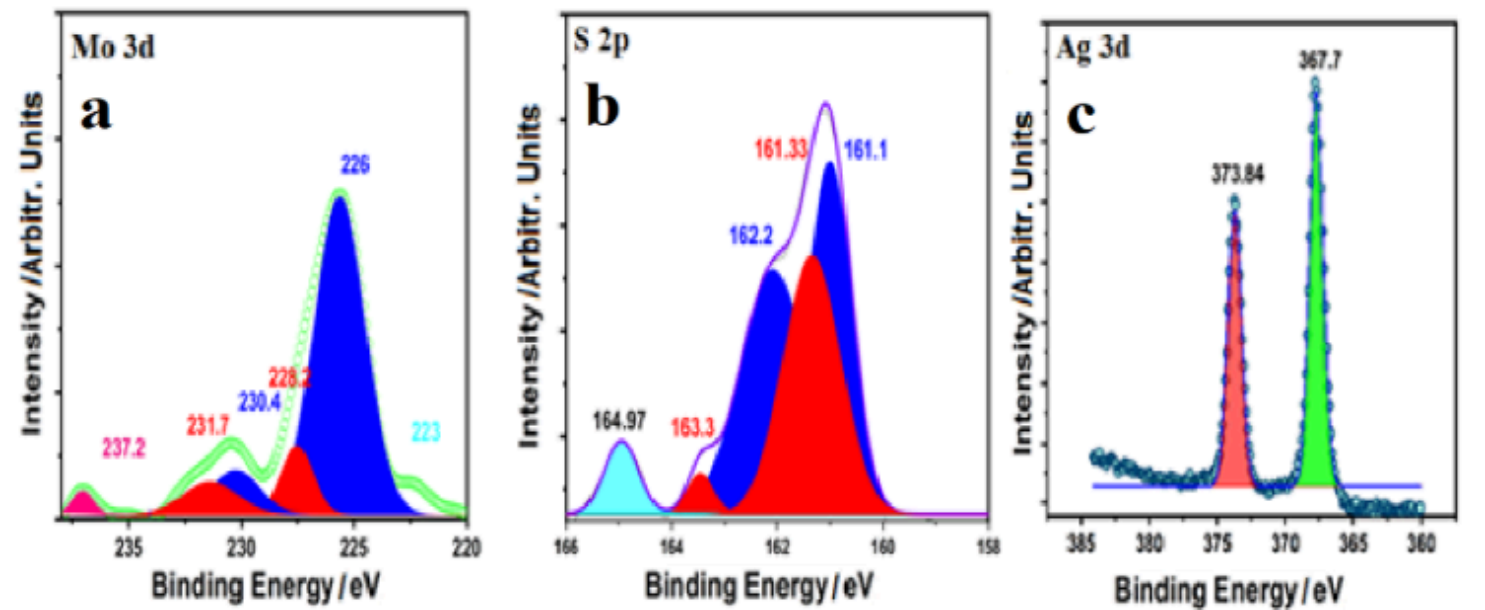


Figure 5

XPS scan spectrum of the Narrow scan of (a) Molybdenum (Mo) and (b) Sulphur (S) and (c) Silver (Ag).

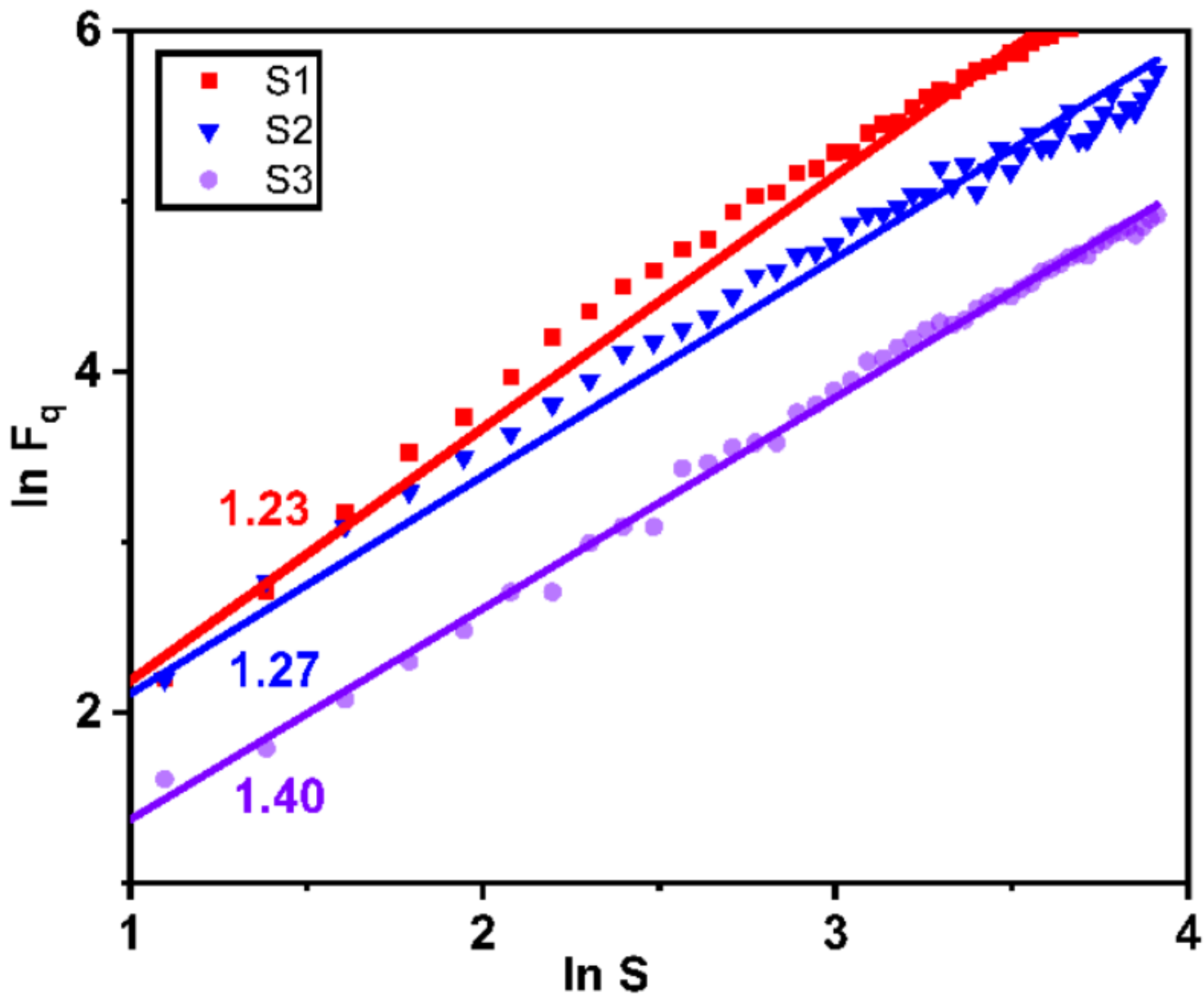


Figure 6

$\ln F_q$ versus $\ln S$ plot obtained from the FESEM substrate S1, S2 and S3

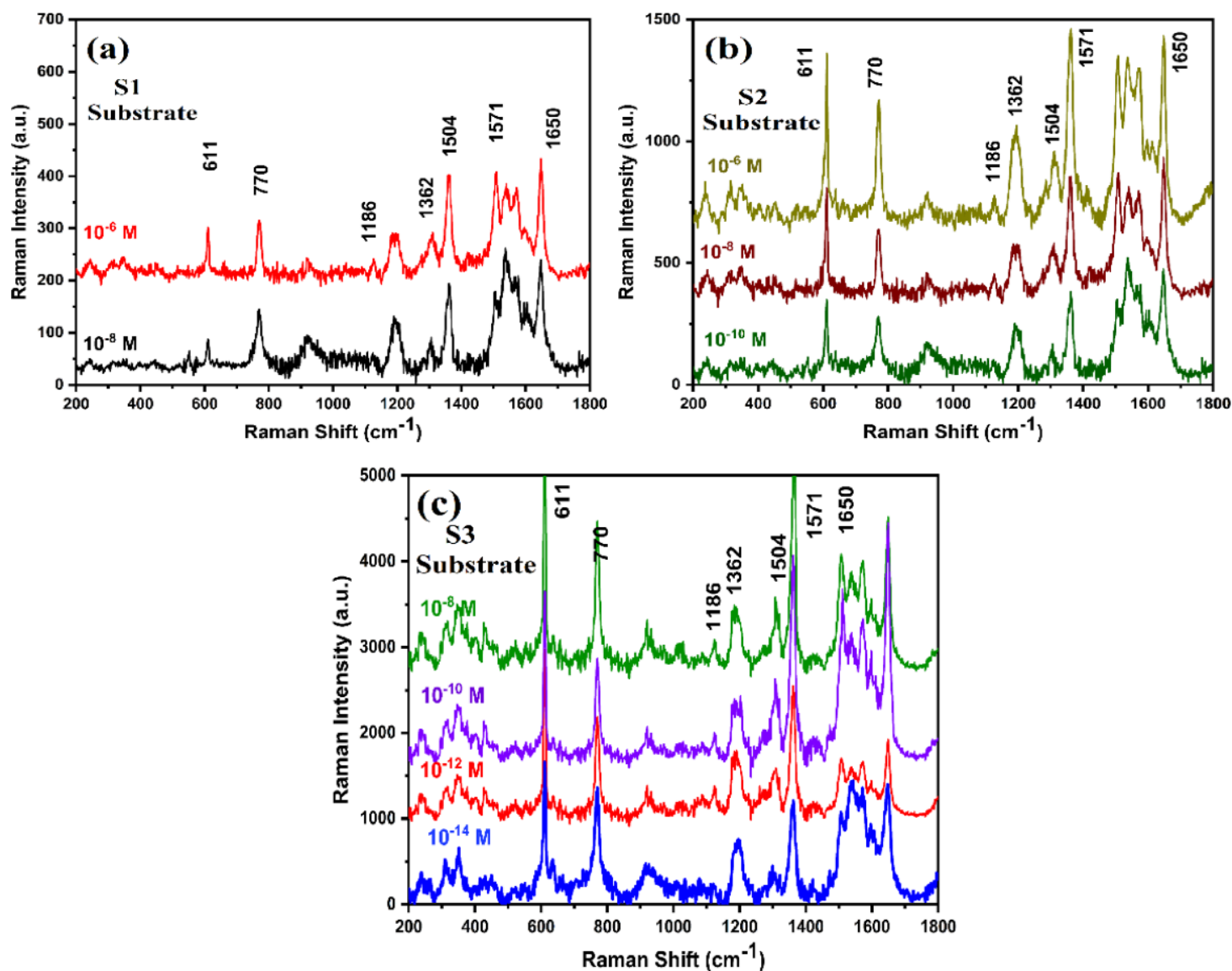


Figure 7

Concentration dependent Surface enhanced Raman spectroscopy (SERS) spectra of R6G molecule at pH ~7 adsorbed on the Ag-MoS₂ (a) S1 substrate (b) S2 substrate and (c) S3 substrate.

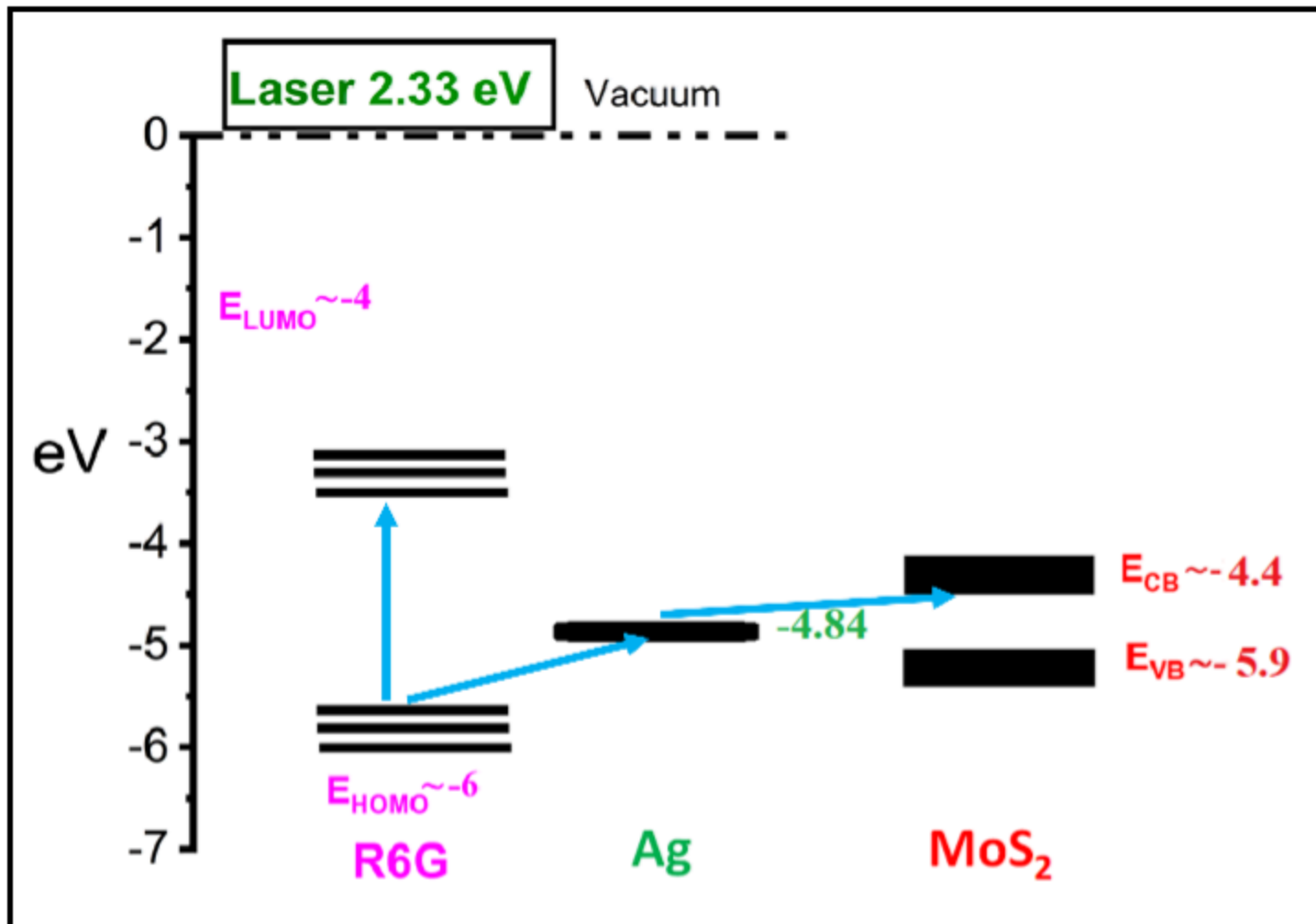


Figure 8

Probable charge transfer pathway of the Ag-MoS₂ with the probe molecule R6G

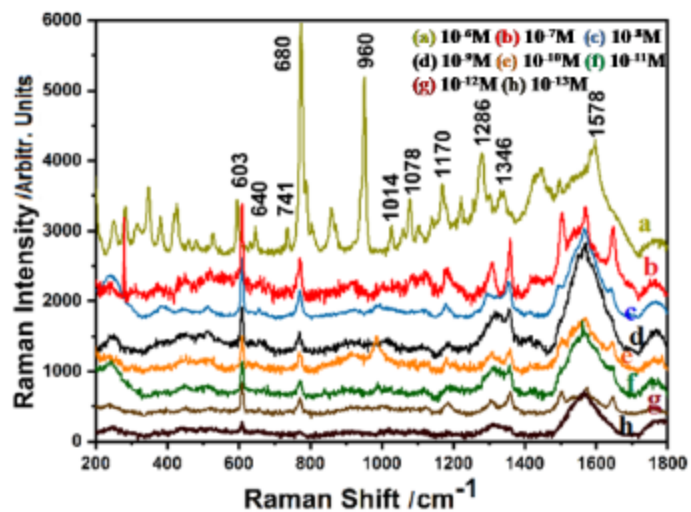


Figure 9

Surface enhanced Raman spectroscopy (SERS) spectra of Epinephrine adsorbed on the Ag-MoS₂ substrate at different concentrations ranging from 10⁻⁶ M to 10⁻¹³M (trace (a) to (h))

Supplementary Files

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