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Microwave-assisted exfoliation of MoS_2 with mixed solvents: structure and morphological studies

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INTRODUCTION & AIM

Two-dimensional nanomaterials, especially transition metal dichalcogenides (TMDCs), exhibit distinctive electronic and photonic properties [1]. Among them, MoS_2 is notable for its bulk-to-monolayer transition, shifting from an indirect to a direct bandgap and gaining a highly active surface with NIR-photothermal and antioxidant functions, enabling uses in flexible electronics, catalysis, sensing, and biomedical systems [2]. We isolate MoS_2 nanosheets via a top-down, microwave-assisted liquid-phase exfoliation with low energy demand and no complex precursors. Microwave irradiation couples to solvent dipoles and Mo–S bonds, producing ultrafast volumetric heating that transiently increases interlayer pressure and drives rapid delamination. Solvent choice is guided by Hansen Solubility Parameters to minimize solubility distance to MoS_2 [3]. The selected systems yield exfoliated nanosheets confirmed by structural characterization.

METHOD

The optimal solvents can be selected using HSP [3]. To compare the solubility of MoS_2 in the selected solvents, the distance between the HSP of MoS_2 and those of each solvent (Ra) is calculated. A smaller Ra distance indicates greater solubility of MoS_2 in the corresponding solvent [2]. The optimal solvents obtained were 80% acetone / 20% water, 50% acetone / 50% butanediol, and 80% acetone / 20% butanediol and are shown in Table 1.

Table 1. Hansen Solubility Parameters (HSP) and Ra.

SOLVENTS	δD ($MPa^{1/2}$)	δP ($MPa^{1/2}$)	δH ($MPa^{1/2}$)	Ra ($MPa^{1/2}$)
MoS_2	17,8	9	7,5	-
80%Acetone/20% Water	15,5	11,52	14,06	8,399
80%Acetone/20% 1,4- Butanodiol	15,7	10,3	9,88	4,999
50%Acetone/50% 1,4- Butanodiol	16	10,15	14,2	7,692

MoS_2 solutions (0.4 g in 200 mL of solvent) were soaked for 24 hours, treated in a 600 W domestic microwave for 10 minutes, cooled to room temperature, centrifuged at 3000 rpm for 30 minutes, and filtered through PVDF membranes (0.45 µm pore size). MoS_2 nanosheet membranes were obtained after drying at 48°C for 24 hours.

The crystalline structure was characterized using a Bruker AXS model D4 Endeavor X-ray diffractometer with Cu Kα radiation, Raman spectroscopy in Horiba LabRAM HR Evolution equipement, 514 nm excitation, Olympus 100× and SEM analyzes were carried out using a ZEIZZ GeminiSEM 360-8217010165 model microscope at 5 kV.

RESULTS & DISCUSSION

The XRD analysis (Figure 1) shows a decrease in peak intensity, especially the 002 peak, indicating reduced crystal size and coherence in the stacking direction, suggesting a transition from bulk to a sheet-like or nanomaterial morphology.

The Raman spectrum confirms successful exfoliation: bulk MoS_2 shows the characteristic peaks of the 2H phase, specifically an E_{2g} mode at 381 cm^{-1} and an A_{1g} mode at 407 cm^{-1}. For the exfoliated nanosheets, the A_{1g} mode appears at 404–406 cm^{-1} and the E_{2g} mode at 381 cm^{-1}, indicating few-layer nanosheets. The decrease in the separation between the E_{2g} and A_{1g} peaks indicates a progressive reduction in the number of layers in the exfoliated materials.

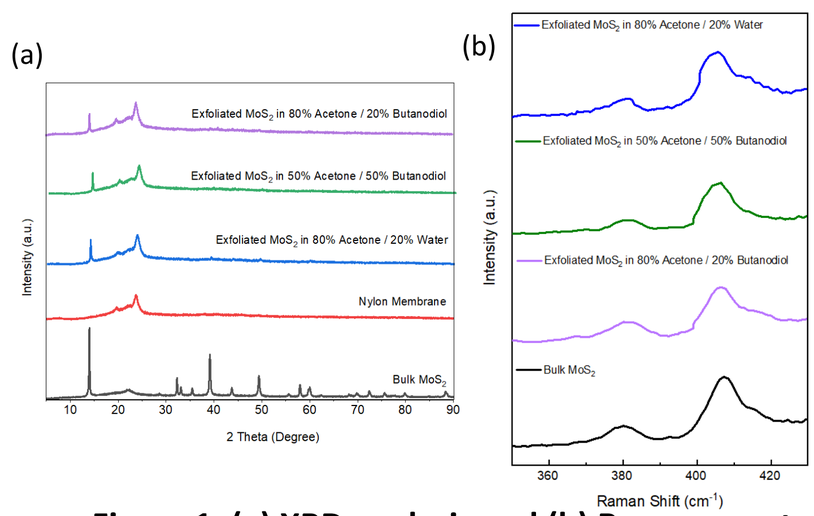

Figure 1. (a) XRD analysis and (b) Raman spectroscopy

This is further supported by SEM images, which reveal thin and abundant exfoliated MoS_2 sheets, confirming successful fragmentation and exfoliation of the bulk particles.

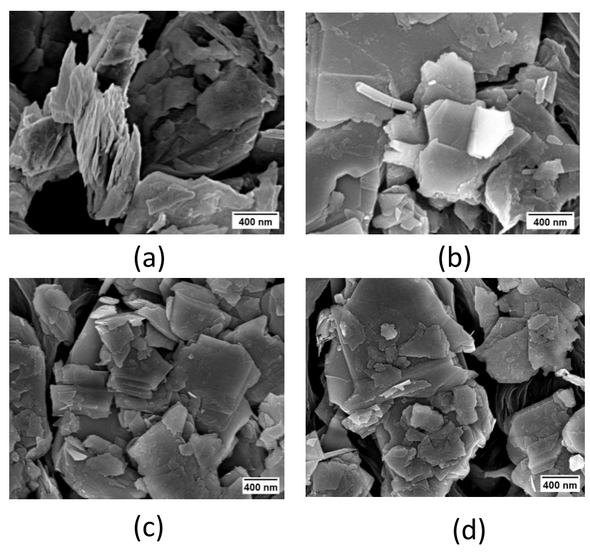

Figure 2. SEM images of (a) bulk MoS_2 and (b–d) exfoliated nanosheets obtained in 50% acetone / 50% 1,4-butanediol, 80% acetone / 20% water, and 80% acetone / 20% 1,4-butanediol, respectively.

CONCLUSION

In conclusion, according to the Raman spectrum, the sample treated with the acetone/water mixture exhibited the smallest E_{2g}–A_{1g} separation, indicating the highest degree of exfoliation. XRD and SEM analyses further suggest a reduction in crystallite size and show that the exfoliated MoS_2 sheets are thin and abundant, consistent with fragmentation and successful exfoliation.

FUTURE WORK / REFERENCES

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