

Appendix

Appendix

Table AT1. Measurements and mean thickness of exfoliated WS₂ nanosheets

Measurements	Thickness of WS ₂ nanosheet (nm)	Mean (nm)
1	112.136	105.855
2	99.988	
3	110.114	
4	120.226	
5	86.812	

Table AT2. Raman peaks of exfoliated WS₂ nanosheets with different laser powers and the corresponding phonon modes

Laser power (mW)	Distinct Raman Peaks (cm ⁻¹)	Corresponding phonon modes
0.025	353.815	E ¹ _{2g}
	420.667	A _{1g}
0.05	348.651	2LA(M)
	352.094	E ¹ _{2g}
0.25	420.667	A _{1g}
	321.062	
0.5	348.651	E ¹ _{2g}
	420.667	A _{1g}
2.5	348.651	2LA(M)
	352.094	E ¹ _{2g}
5	418.959	A _{1g}
	173.051	LA(M)
5	295.12	Mixed mode
	350.373	E ¹ _{2g}
	417.251	A _{1g}
	579.749	Mixed mode
	699.676	Mixed mode
	127.285	Discussed in the main text
	185.333	
	255.195	
	322.789	
	346.929	E ¹ _{2g}
413.833	A _{1g}	
698.022	Discussed in the main text	
801.637		

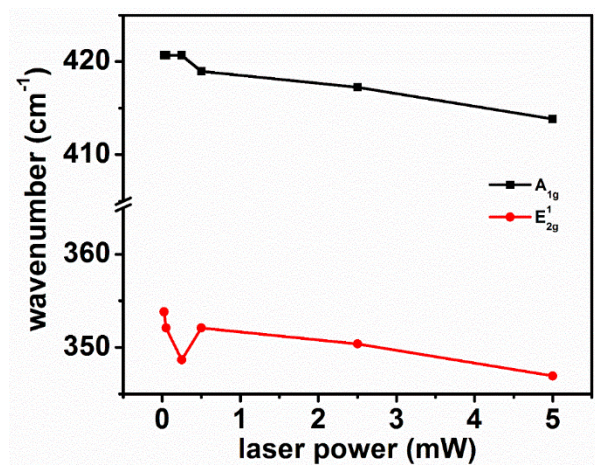


Fig. A1. Shifting of E_{2g}^1 and A_{1g} mode with laser power.

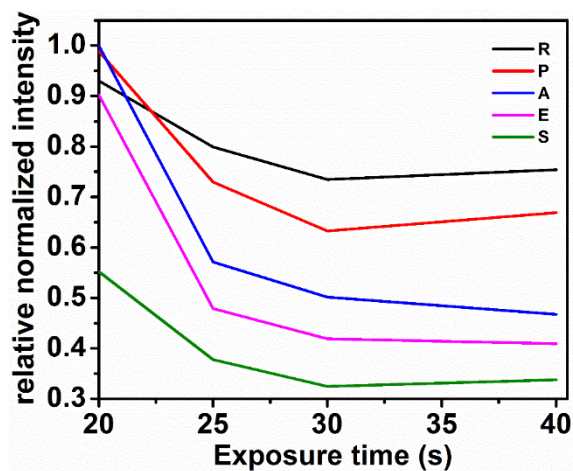


Fig. A2. Relative intensities of the Raman peaks R, P, A, E and S with respect to exposure time.

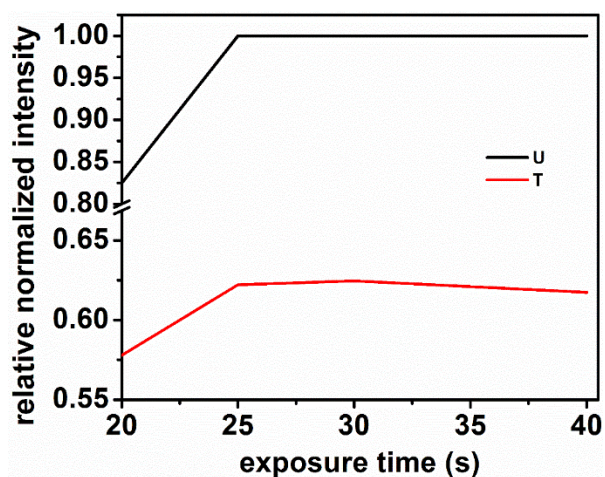


Fig. A3. Relative intensities of the Raman peaks U and T with respect to exposure time.

Calculation of fringe width of untreated WS₂ nanosheets

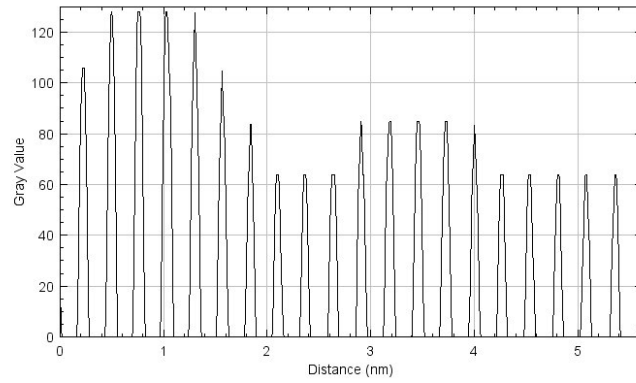


Fig. A4. Plot details of the untreated WS₂ nanosheets shown in **Fig. 2.9(d)**.

From A4,

Width of 21 fringes = 5.62 nm

Width of 1 fringe ~0.27 nm

Therefore, fringe width of untreated nanosheets ~0.27 nm

Calculation of fringe width of treated WS₂ nanosheets (Taking the top middle portion of the Fig. 2.10(d))

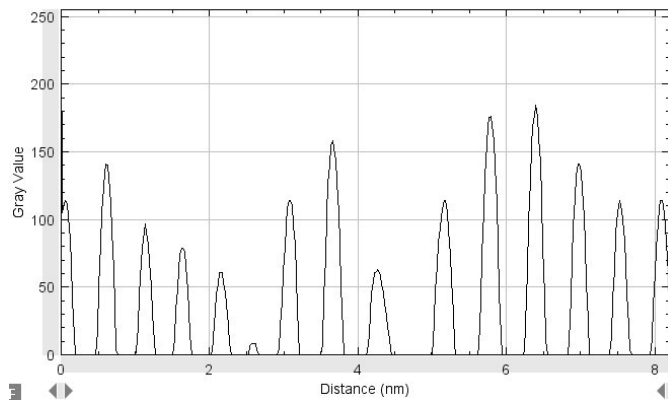
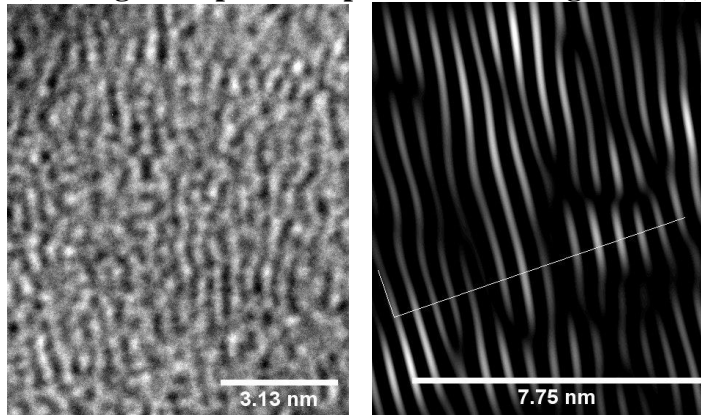


Fig. A5. Images and plot details of the top middle portion of treated WS₂ nanosheets shown in **Fig. 2.10(d)**.

From A5,
Width of 15 fringes = 8.2 nm
Width of 1 fringe ~0.55 nm

Therefore, fringe width of top middle portion of treated nanosheets ~0.55 nm

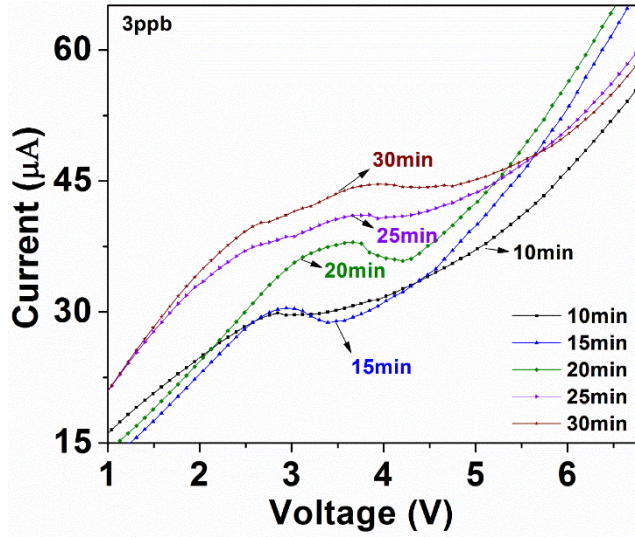


Fig. A6. *I-V* characteristics of sensing unit treated with 3 ppb Zn²⁺ ions.

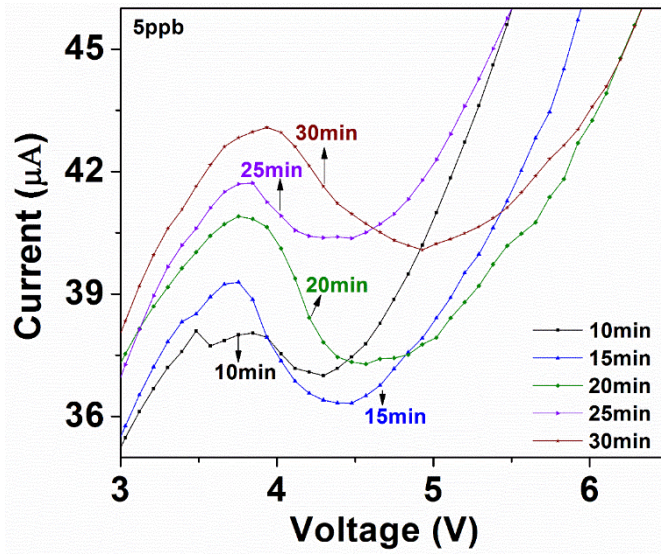


Fig. A7. *I-V* characteristics of sensing unit treated with 5 ppb Zn²⁺ ions.

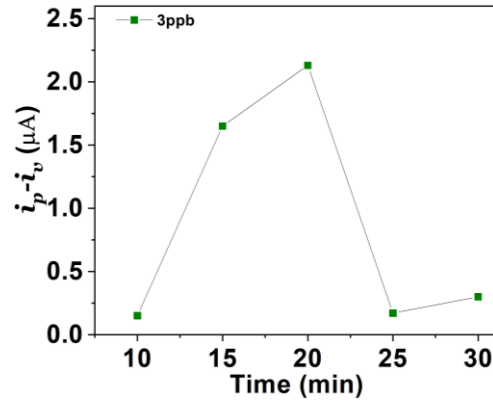


Fig. A8. Difference between current corresponding to peak and valley point ($i_p - i_v$) at different interaction time for the sensing unit treated with 3 ppb Zn^{2+} ions.

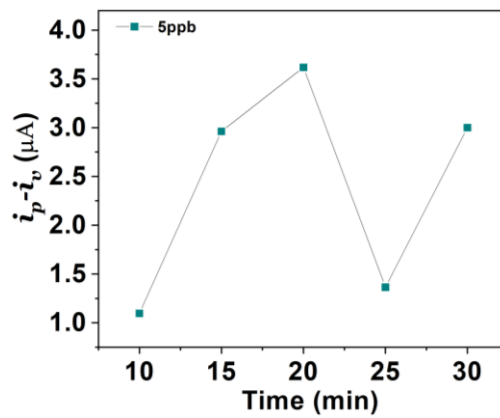


Fig. A9. Difference between current corresponding to peak and valley point ($i_p - i_v$) at different interaction time for the sensing unit treated with 5 ppb Zn^{2+} ions.

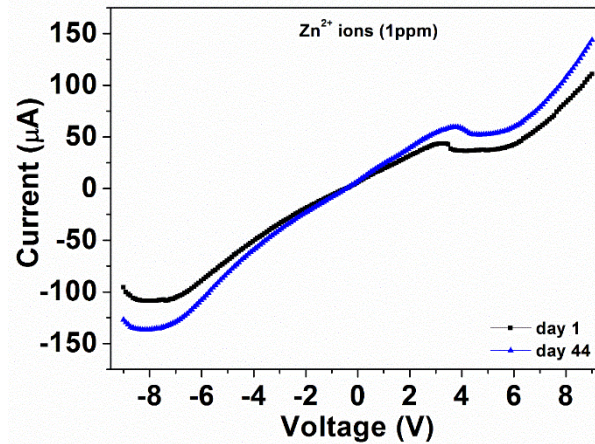


Fig. A10. I - V characteristics of the sensing unit treated with 1 ppm Zn^{2+} ions on day 1 and day 44.

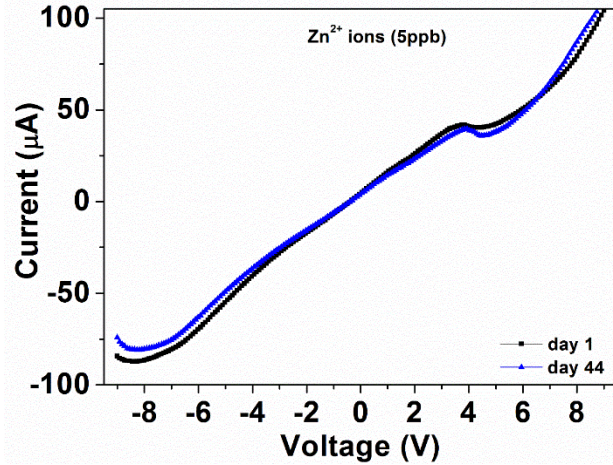


Fig. A11. I - V characteristics of the sensing unit treated with 5 ppb Zn^{2+} ions on day 1 and day 44.

When the device was used more than once, then the current in the device gradually decreases. However, after using 3 to 4 times, no significant change in the (i_p - i_v) (Current corresponding to peak and valley of negative resistance region) has been observed after heavy metal ion treatment. Therefore, the same sensor unit can be used for 3-4 times. However, the sharpness of (i_p - i_v) decreases if we use the unit over and over again.

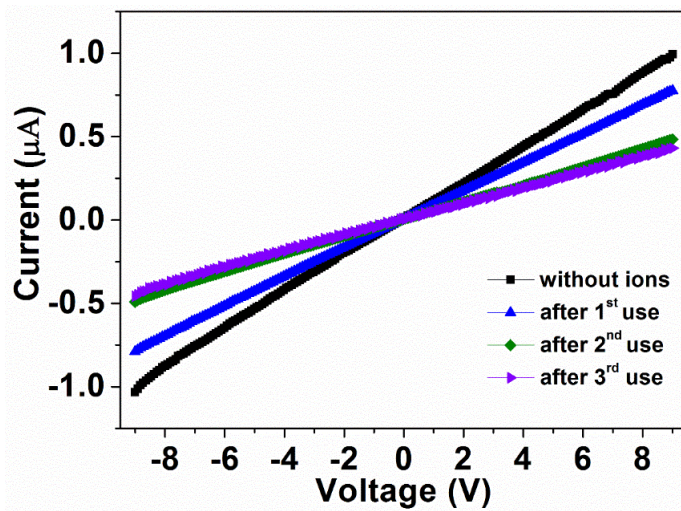


Fig. A12. I - V characteristics of the sensing unit after repeated use.

Analysis of XRD spectra of MoS₂ and WS₂ nanosheets

Crystallographic information related to the MoS₂ and WS₂ nanosheets exfoliated in the process described in *method 1, Chapter V*.

Table AT3 Calculation of *d*-spacing for MoS₂ nanosheets

2 θ	FWHM	<i>d</i>-spacing (in nm)
14.55817	0.13735	0.607723
29.20407	0.2381	0.30543
32.87867	0.16076	0.272085
33.6952	0.16392	0.265676
40.81548	0.20578	0.220821
39.7348	0.23798	0.226574
44.16611	0.18212	0.204814
49.80842	0.2747	0.182853
56.01794	0.31399	0.163966
58.33801	0.20417	0.157985
60.33647	0.75305	0.153221
62.81533	0.31635	0.147758
68.51011	0.22057	0.136797
70.23377	0.52387	0.133856
72.80813	0.32854	0.129744

Table AT4 Calculation of *d*-spacing For WS₂ nanosheets

2 θ	<i>d</i>-spacing (in nm)
13.82942	0.639578903
14.18114	0.623794171
22.80843	0.389420991
28.63777	0.311339562
28.29726	0.315008168
32.44733	0.27560267
33.18591	0.269635665
33.74472	0.265297151
39.2079	0.2294969
43.66333	0.20705553
49.39115	0.184300072
49.181	0.185038316
58.09987	0.158575965
58.32341	0.15802129
59.59279	0.154954719
60.12902	0.153700361

Analysis of TEM micrographs

1. Measurement of interlayer spacing (d -spacing) in MoS₂ nanosheets, *method 1*, Chapter V.

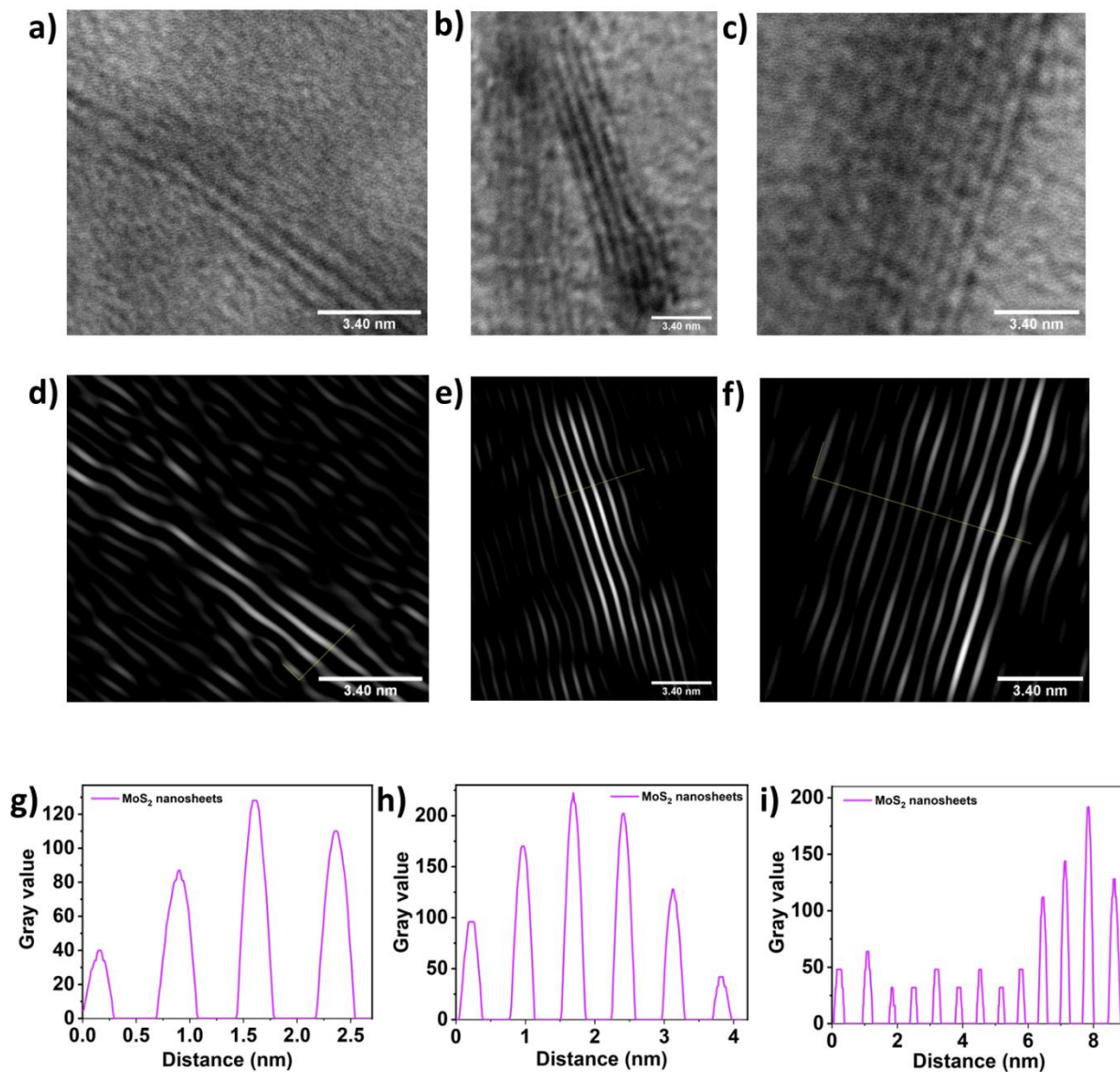


Fig A13. (a), (b), (c) micrographs of MoS₂ nanosheets taken at different positions; (d), (e), (f) the inverse first fourier transformation (IFFT) of (a), (b) and (c) respectively; (d), (e), (f) gray value vs distance graphs of the straight lines drawn perpendicular to the fringe lines drawn on (d), (e) and (f) respectively.

Table AT5. Calculation of d -spacing from the data obtained from plot details in **Fig A13 (g), (h) and (i)**.

Figure	Starting point (X1)	Ending point (X2)	Number of peaks (N)	Interlayer distance (X2-X1)/N (nm)
g	0	2.54	4	0.63
h	0.04	3.97	6	0.65
i	0.05	8.85	13	0.67
Average d -spacing				0.65

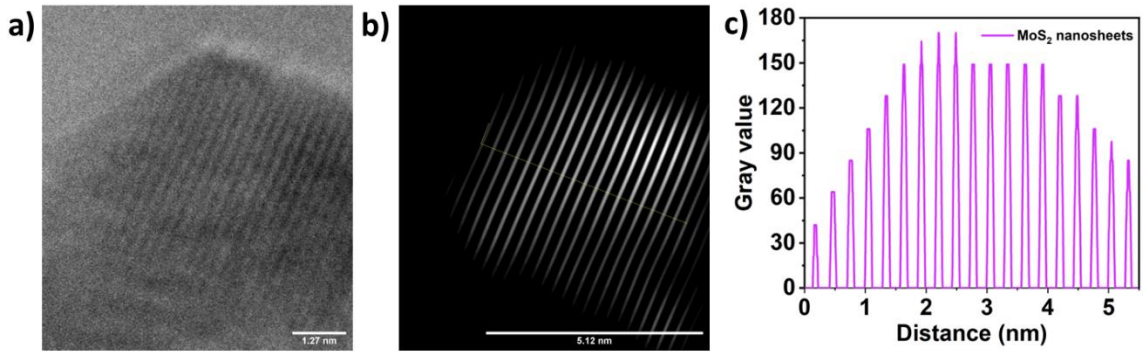


Fig A14. (a) micrographs of MoS₂ nanosheets; (b) IFFT of (a); (c) gray value vs distance graphs of the straight line drawn perpendicular to the fringe lines drawn on (b).

Table AT6. Calculation of d -spacing from the data obtained from plot detail in **Fig A14 (c)**.

Figure	Starting point (X1)	Ending point (X2)	Number of peaks (N)	Interlayer distance (X2-X1)/N (nm)
c	0.13	5.38	19	0.27

2. Measurement of interlayer spacing (d -spacing) in WS₂ nanosheets:

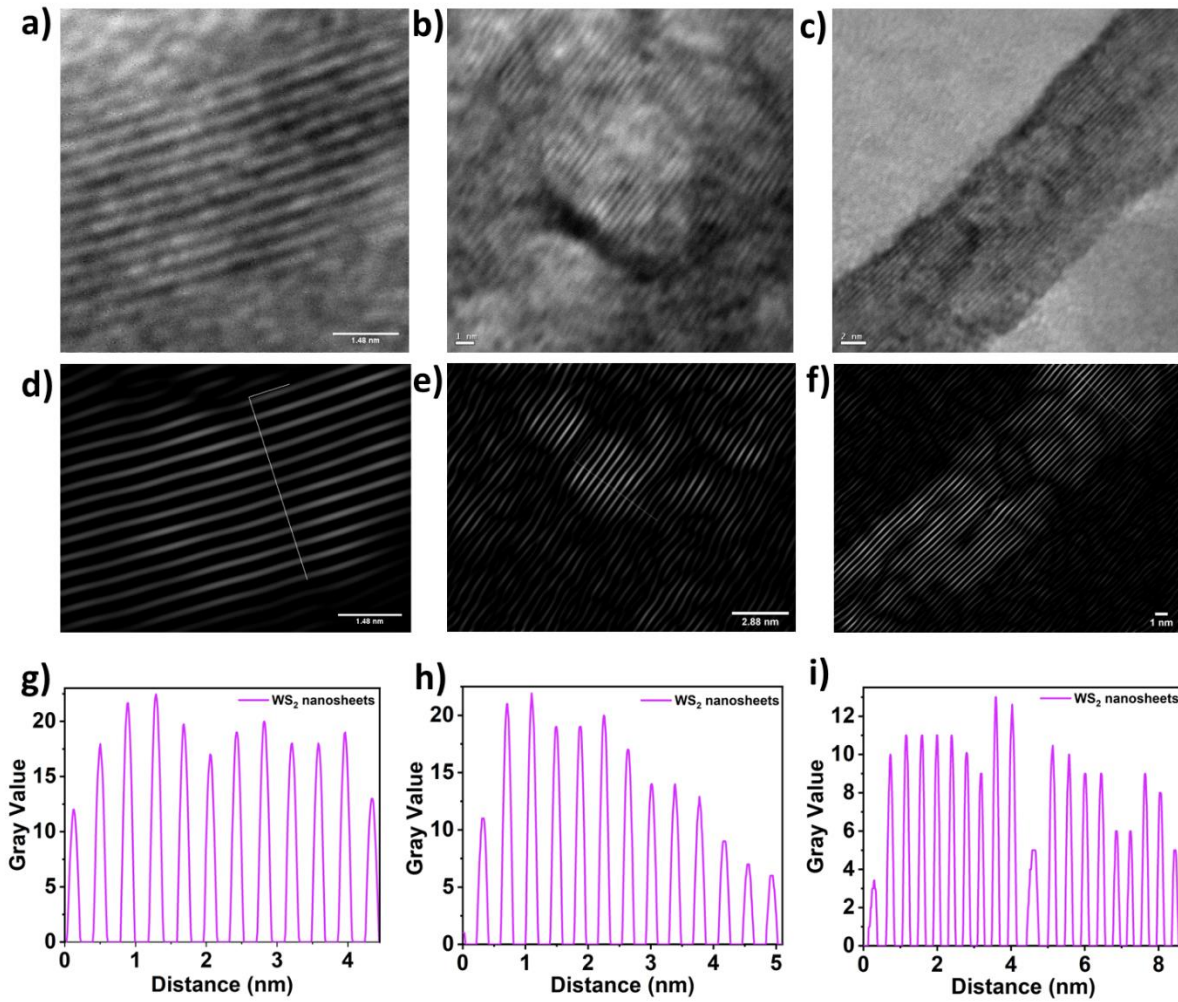


Fig A15. (a), (b), (c) HRTEM micrographs of WS₂ nanosheets taken at different positions; (d), (e), (f) IFFT of (a), (b) and (c) respectively; (g), (h), (i) gray value vs distance graphs of the straight lines drawn perpendicular to the fringe lines on (d), (e) and (f) respectively.

Table AT7. Calculation of d -spacing from the data obtained from plot detail in **Fig A15 (g), (h), (i)**.

Figure	Starting point (X1)	Ending point (X2)	Number of peaks (N)	Interlayer distance (X2-X1)/ N (nm) (rounding off)
g	0	4.45	12	0.37
h	0.19	4.87	13	0.37
i	0.11	8.58	20	0.42
Average d -spacing				0.39

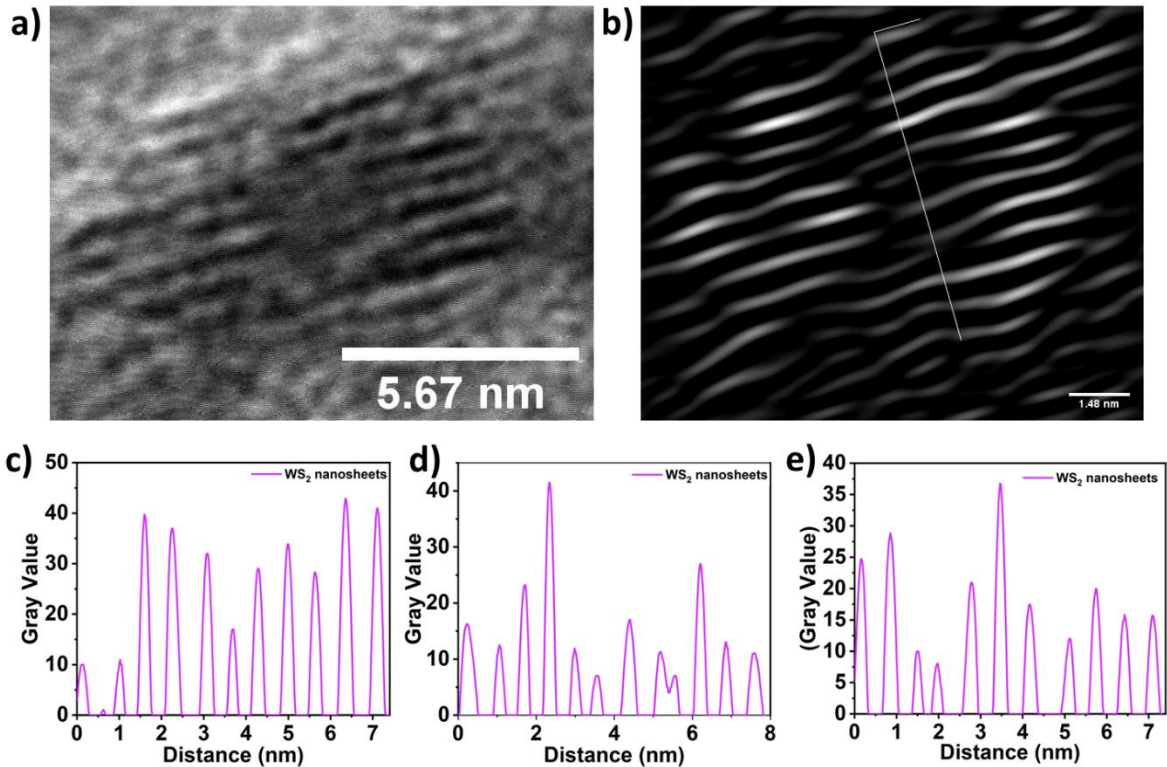


Fig A16. (a) Micrograph of WS₂ nanosheets (b) IFFT of (c), (d), (e) gray values vs distance graphs of three different straight lines drawn perpendicular to the fringe lines in the image (b).

Table AT8. Calculation of *d*-spacing from the data obtained from plot details in **Fig A16 (c), (d) and (e).**

Figure	Starting point (X1)	Ending point (X2)	Number of peaks (N)	Interlayer distance (X2-X1)/N (nm) (Rounding off)
c	0	7.31	12	0.61
d	0	7.85	12	0.65
e	0	7.29	11	0.66
Average <i>d</i> -spacing				0.64

List of Publications:

1. Neog, A., Biswas, R., Rather, M.A., Bardhan, P., Mandal, M. and Mazumder, N., 2024. Modulating mediation medium for Few layered Dichalcogenides enhances Inhibition of common pathogens. *Materials Advances*.
2. Neog, A., Deb, S. and Biswas, R. Atypical electrical behavior of few layered WS₂ nanosheets based platform subject to heavy metal ion treatment. *Materials Letters*, 268: 127597, 2020. <https://doi.org/10.1016/j.matlet.2020.127597>
3. Neog, A. and Biswas, R. WS₂ nanosheets as a potential candidate towards sensing heavy metal ions: A new dimension of 2D materials. *Materials Research Bulletin*, 144: 111471, 2021. <https://doi.org/10.1016/j.materresbull.2021.111471>
4. Neog, A., Das, P. and Biswas, R. A novel green approach towards synthesis of silver nanoparticles and it's comparative analysis with conventional methods. *Applied Physics A*, 127(12): 1-6, 2021. <https://doi.org/10.1007/s00339-021-05039-x>
5. Neog, A., Biswas, R. and Bharali, N. A novel electrochemical synthesis of graphene oxide: Converting waste product to utility. *Europhysics Letters*, 137(1): 16001, 2022. <https://doi.org/10.1209/0295-5075/ac40ea>
6. Neog, A., Biswas, R. Optical Study of Liquid Dispersed Few-Layered WS₂ Nanosheets. In: Sengupta, S., Dey, S., Das, S., Saikia, D.J., Panda, S., Podila, R. (editors) *Selected Progresses in Modern Physics*. Springer Proceedings in Physics, vol 265, 2021, Springer, Singapore. https://doi.org/10.1007/978-981-16-5141-0_25
7. Neog, A. and Biswas, R. Phase transition and formation of inorganic fullerene type nanostructure in WS₂ nanosheet system under laser excitation. *Scripta Materialia*, 216: 2022. <https://doi.org/10.1016/j.scriptamat.2022.114729>
8. Neog, A., Sarmah, H. J., Mahanta, D., Biswas R. Advances on layered semiconducting transition metal dichalcogenides in sensing applications, *Nanoscale matter and principles for sensing and labeling applications*, Springer, 2022
9. Neog, A. and Biswas, R. A novel route for sensing heavy metal ion in aqueous solution. *Europhysics Letters*, 139 :46002-p1, 2022. <https://doi.org/10.1209/0295-5075/ac76dc>
10. Neog, A. and Biswas, R. Evidence of Laser-Induced Amplification of Random Noise in WS₂ Nanosheets Based Resistive System. *Physica status solidi (RRL)–Rapid Research Letters*, 16(8) :2200142, 2022. <https://doi.org/10.1002/pssr.202200142>

11. Chakraborty, I., Banik, S., Govindaraju, I., Das, K., Mal, S.S., Zhuo, G.Y., Rather, M.A., Mandal, M., Neog, A., Biswas, R. and Managuli, V. Synthesis and detailed characterization of sustainable starch-based bioplastic. *Journal of Applied Polymer Science*, 139(39): e52924, 2022. <https://doi.org/10.1002/app.52924>
12. Barman, C., Neog, A., Biswas, S. and Biswas, R. A Preliminary Investigation Towards Detecting Heavy Metal Ions with a Cost-Effective Scheme. *Letters in Applied NanoBioScience*, 11(3): 2021. <https://doi.org/10.33263/LIANBS113.38223825>
13. Biswas, R., Bharali, N. and Neog, A. A Novel Route towards Sustainable Synthesis of Graphene. *Letters in Applied NanoBioScience*, 10(4):2760-2765,2021. <https://doi.org/10.33263/LIANBS104.27602765>
14. Dalal, N., Boruah, B.S., Neoh, A. and Biswas, R. Correlation of surface plasmon resonance wavelength (SPR) with size and concentration of noble metal nanoparticles. *Annals of Reviews and Research*, 5(2):1-6, 2019. <https://doi.org/10.19080/ARR.2019.05.5556568>

LIST OF CONFERENCES ATTENDED:

1. **“Electrical detection of heavy metal ions using multilayer WS₂ nanosheets”**, National Conference on Emerging Trends in Materials Science (**ETMS 2022**), May, 2022, Department of Physics, Tezpur University, Assam, India. (Oral Presentation).
2. **“Optical study of few layer WS₂ nanosheets”**, International Conference in Trends in Modern Physics, (**TiMP 2021**), February, 2021, Jyothi Engineering College, Kerala, India. (Virtual Mode), (Oral Presentation).
3. **“Light induced unique attributes of WS₂ nanosheets based device leading to generation and amplification of random noise”**, International Conference on Nanoscience and Nanotechnology (**ICONN 2021**), February, 2021, SRM Institute of Science and Technology, Tamil Nadu. (Virtual Mode), (Oral Presentation).
4. **“Laser induced oxidation of WS₂ nanosheets”**, International Conference on Advances in Nano-optoelectronics and its Application (**ICANOPA-2020**), October 2020, Department of Physics, Rajiv Gandhi University, Rono Hills, Arunachal Pradesh, (Virtual Mode), (Oral Presentation).
5. **“Effect of laser power on Raman studies of multilayered WS₂ nanosheets”**, Student Conference on Optics and Photonics (**SCOP 2020**), September 2020, Physical Research Laboratory (PRL), Ahmedabad, (Virtual Mode), (Oral Presentation).
6. **“Electrical properties of tungsten disulfide nanosheets deposited on copper electrodes (fabricated on copper clad)”**, Progress in Material Science research (**PMSR 2020**), February 4-6, 2020, Dibrugarh University, Dibrugarh, Assam. (Poster Presented. Best Poster Award.)

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My Family
and
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Modulating mediation medium for few layered dichalcogenides enhances inhibition of common pathogens†

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As mandated by the United Nations *Ad hoc* Interagency Coordination Group, there is a looming prospect of acute health crises and poverty by 2030 in the absence of action against microbial resistance. Nanomaterials possess the capability to disrupt pathogenic cell membranes or induce cell death through the production of reactive oxygen species and free radicals. Hence, nanomaterials have emerged as promising agents to combat the impending crises. While research on nanomaterial-based approaches for drug-resistant infections has commenced, it is imperative to conduct parallel investigations to ascertain the maximal effectiveness of nanomaterials against common pathogens. Transition metal dichalcogenides represent the next generation of antibiotics to counter common and multidrug-resistant infections. However, existing studies predominantly focus on a limited spectrum of microorganisms or pathogens, with minimal reports on their efficacy against pathogens such as *Pseudomonas aeruginosa* and *Candida albicans*. Notably, many studies have explored the functionalization, doping, or composite formation of these nanostructures to enhance their antipathogenic activity, overlooking the intrinsic antibiotic potential of the materials in their original form. Consequently, this study investigates the antipathogenic activity of non-functionalized few-layer WS₂ and MoS₂ nanosheets against a range of pathogens, including *Mycobacterium smegmatis*, *Staphylococcus aureus*, *Bacillus cereus*, *Pseudomonas aeruginosa*, *Yersinia pestis*, *Escherichia coli* and *Candida albicans*, in lysogeny broth (LB) and potato dextrose broth (PDB) media. Remarkably, few-layer MoS₂ and WS₂ exhibit significant antipathogenic activity against all tested pathogens, surpassing standard antibiotics in the case of *Pseudomonas aeruginosa* and *Candida albicans*.

1. Introduction

Transition metal dichalcogenides (TMDCs) have made significant strides in various applications, including photosensing,^{1–3} bio and chemical sensing,^{4–7} future electronic and valleytronic devices,^{8–11} catalysis,^{5,12,13} wastewater treatment, and toxic gas adsorption and removal,¹⁴ among others. Despite these advancements, research on TMDCs for biomedical applications is still nascent. Only in the past two decades have researchers begun exploring the cytotoxicity of these materials.^{15–17} Notably, inorganic fullerene-type and few-layer structures of WS₂ and MoS₂ have garnered attention due to their low cytotoxicity and genotoxicity, as assessed by various biocompatibility tests.¹⁸

These findings have spurred investigations into the anti-pathogenic activities of WS₂ and MoS₂. Although limited, several studies have yielded promising results. WS₂ nanosheets synthesized *via* the hydrothermal method have exhibited significant bactericidal activity, with a mortality rate of up to 99.97% against *Staphylococcus epidermidis*.¹⁹ Additionally, WS₂ nanosheets have shown efficacy against *Escherichia coli*, *Salmonella typhimurium*, and *Bacillus subtilis* at a concentration of 250 μg mL⁻¹, as assessed using the colony counting method.¹⁹ Furthermore, the antibacterial activity of WS₂ against Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus* was evaluated through colony-forming unit studies, resulting in nearly 0% bacterial viability at a concentration of 200 μg mL⁻¹.²⁰ The activity of WS₂ and the WS₂/ZnO nanohybrid against *Candida albicans* was investigated using the disc diffusion method, inhibiting fungal growth by up to 74% and 91%, respectively, at a concentration of 300 μg mL⁻¹.²¹

MoS₂ nanosheets synthesized *via* Li-intercalation exhibited a reduction in *Escherichia coli* viability of 91.8% ± 1.4% at a concentration of 80 μg mL⁻¹.²² The superior performance of

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† Electronic supplementary information (ESI) available. See DOI: <https://doi.org/10.1039/d3ma01128c>



Atypical electrical behavior of few layered WS₂ nanosheets based platform subject to heavy metal ion treatment

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ABSTRACT

We herein report an atypical electrical behavior of WS₂ nanosheets drop casted on Cu electrodes. The addition of heavy metal ions on this platform results in non-ohmic behavior, accompanied by a dramatic rise of current. Additionally, this atypical behavior is found to be reversible. The proposed platform regains its ohmic behavior upon removal of these ions from the nanosheets. It is envisioned that this unusual characteristic will pave way for more research in the field of sensing as well as in relevant fields.

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1. Introduction

The advent of semimetal Graphene [1] and its exotic behavior [2,3] have inspired the exploration of other 2D materials. Transition Metal Dichalcogenides (TMDC) are some of the post-graphene 2D layered materials having identical structural properties with Graphene [4]. TMDCs are characterized by formula MX₂ where *M* represents transition metals including Molybdenum (Mo), Tungsten (W) etc. with *X* denoting chalcogenides. In monolayer, the transition metals are sandwiched between the chalcogens; thereby possessing strong intra-layer covalent bond in between them. However, the inter-layer interaction is weak Van der Waals attraction. This facilitates the exfoliation of these materials into single and few layers, analogous to Graphene. The exfoliation of these materials into mono or few layers considerably preserves the bulk properties and gains additional properties due to quantum confinement [5–6]. Consequently, WS₂ nanosheets have drawn more attention for its exceptional electronic properties, with wide use in optoelectronics, catalysis, thermal batteries and sensing applications [7,8,9,10]. Several detection schemes e.g. fluorescence based [11,12], Field effect transistor based [13,14] etc. have been devised for the detection of heavy metal ions with the help of TMDCs. However, electrical modulation of two terminal WS₂ nanosheets based electric platform in the context of heavy metal ion treatment is hardly explored so far. Generally, WS₂ possesses intrinsic Sulfur(S) atoms being suitable coordination sites for certain heavy metal ions [15]. Very few studies

address the basic affinity of WS₂ towards heavy metal ion treatment. Therefore, in this work, we have attempted to fabricate a two terminal sensing platform as shown in the Scheme 1. Further, we analyze the effect of heavy metal ions on it in order to qualitatively investigate the sensing ability.

2. Experimental details

1.5 mg/ml of bulk WS₂ (99.9%, Sigma Aldrich) was dispersed in N-methyl-2 pyrrolidone and sonicated for 3 hrs with periodic shaking. Then, the dispersion was centrifuged for 15 mins (3000 rpm) and the obtained pellets were washed; followed by 12 hrs of drying to attain the desired nanosheets.

To prepare 1 ppm solution of the ions, CoCl₂, FeCl₃, HgCl₂, NaAsO₂, SnCl₂ and ZnCl₂ were purchased from Merck, PbCl₂ from Loba Chemie and preformed dilution process in DD water. The crystal structure of as-synthesized sample was examined by X-ray diffraction (XRD, Rigaku) at a rate of 1° min⁻¹. The surface morphology was observed by SEM (JEOL). Optical characterization was done by Raman Spectroscopy (RENISHAW, model no– Basis Series), where, 514 nm Ar⁺ laser line was used.

The as-synthesized WS₂ nanosheets were dispersed in DD water and ultrasonicated for 20 mins and then drop casted on top of finger like Cu electrodes. Electrical measurements were done using two probe arrangement attached to a Keithley 2400© sourceme-ter at room temperature. The effects were monitored up to 20 mins at an interval of 5 min for Co²⁺ and Fe³⁺ ions and for other curves, the response were taken 20 mins after addition of the ions. The whole prototype is shown in Scheme 1.

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Research Papers

WS₂ nanosheets as a potential candidate towards sensing heavy metal ions: A new dimension of 2D materials

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ABSTRACT

Here we present unique features of metal ion treated tungsten disulphide (WS₂) nanosheets. Through chemical exfoliation of bulk WS₂, multilayered WS₂ nanosheets were prepared and drop casted on finger like Cu (Copper) electrodes, thereby fabricating 2D material-based sensor units. Later, I-V characteristics of the units were investigated with and without heavy metal ion treatment. In case of Zn²⁺ ions, the sensor unit shows prominent negative resistance in the positive potential region of the I-V characteristics, which was further quantified by taking the difference between the peak and valley point of the negative resistance region to selectively detect Zn²⁺ ions. Accordingly, the response of the novel scheme was visualized by plotting calibration curve for Zn²⁺ ions, accompanied by analysis of the influence from other interfering ions through I-V characteristics. Excellent selectivity towards Zn²⁺ ions, with limit of detection 0.94 ± 0.05 ppb was found. Sensitivity of 0.63 ± 0.05 μ A per ppb was achieved, accompanied by a rapid response.

1. Introduction

The attention towards two-dimensional (2D) Transition Metal Dichalcogenides (TMDCs) has escalated right after the successful synthesis of Graphene in 2004 [1–3]. The exploitation of the unique material characteristics of TMDCs has laid down the base for new generation nano-optoelectronics devices. In TMDCs, transition metals such as Ti, Mo, W etc. and group VI-A chalcogen elements such as S, Se, Te crystallize into layered structure; thereby offering diverse material properties from that of an insulator to a metal. The strong intra-layer covalent bond between the chalcogenides and weak inter-layer Van der Waal attraction between the layers facilitates the transition of these materials from three dimensional (3D) to 2D, analogous to Graphene [4–6]. Among the semiconducting TMDCs, WS₂ has caught considerable attention owing to unique features such as higher stability and wider operational temperature. Moreover, it possesses the direct band gap and breaking of spatial inversion symmetry with further accompaniment of high excitonic binding energy in the monolayers [7–10].

Apart from that, most of the semiconducting (SC) 2D materials including WS₂ can react with metallic and nonmetallic ions, nanoparticles, organometallic materials etc. and can change the electronic surface states of 2D materials [11–13]. The effect of such chemistry can be clearly observed in the electronic behavior of the system. Therefore,

such chemical modifications have been used to develop different electronic units and sensors [14,15]. Heavy metal ions (HMIs) which are considered as toxic to living body, can also be used to functionalize semiconducting 2D materials. However, such functionalization techniques have not been properly utilized to detect HMI itself. Therefore, this work endeavors to systematically study the modification of I-V characteristic behavior of layered WS₂ system due to surface functionalization by HMIs as well as find a way to selectively detect HMIs.

In recent years, the growing concern about environmental impact of Heavy metal pollution has triggered frequent monitoring of heavy metal ions in water, soil etc. which has encouraged plenty of researchers to develop cost effective and sustainable heavy metal ion detectors [16–19]. Among the HMs, Zinc (Zn) is one of the most essential trace elements important for neuron activity, learning activity as well as memory. Interestingly, both deficiency and overdose of Zn²⁺ ions can adversely affect the human health. Zinc deficiency can impair cognitive performance, neuropsychological behavior, attention and other brain activities. Studies suggest that overdose of Zinc can strongly impair consolidation of hippocampal-dependent memory [20–22]. However, according to guidelines for drinking water quality (fourth edition incorporating the first addendum, WHO, 2017), no health-based guideline value has been proposed for zinc in drinking water. According to WHO, Zinc only imparts an undesirable taste to water and makes

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A novel route for sensing heavy metal ions in aqueous solution

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Abstract – Heavy metal ions are some of the major aquatic pollutants which are held responsible for many ailments as well as undesired effects on flora and fauna. The World Health Organization has prescribed some permissible levels for each of the heavy metal ions. The presence of ions above permissible limits results in toxicity in the aquatic bodies as well as for other water-dependent living organisms. Therefore, it is necessary to make monitoring schemes to keep a check on the concentration of heavy metal ions. Considering this, here we report a unique route to detect heavy metal ions. Through chemical exfoliation, tungsten disulphide (WS₂) nanosheets were prepared and drop casted on finger-like Cu electrodes to build the sensing unit. When the sensing unit was treated with heavy metal ions like Cu²⁺, Co²⁺, Pb²⁺, Hg²⁺, Sn²⁺ ions, rise in the current across the unit was observed. The concentrations of all the heavy metal ions were the same, *i.e.*, 1 ppm. The maximum current gain was calculated for Cu²⁺ ions. It was found that the current gain for different ions is different in the voltage range 4–6 V and remains constant except for Cu²⁺ and Co²⁺ ions. It is believed that this novel route will pave the way for rapid and simultaneous sensing of different pervasive heavy metal ions in aqueous solutions.

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Introduction. – Water pollutants are causing havoc in the entire globe. Due to rampant industrialization in small as well as large scale in almost all developing nations, there is a rapid growth of heavy metal ion pollution in aquatic bodies, caused by unplanned and untreated disposal of these industrial effluents and wastes [1,2]. The fact sheets on water displayed by the World Health Organization (WHO) elucidate the present global status of drinking water [3]. While establishing a framework for safe drinking water, WHO has incorporated different levels of information starting from microbial, chemical up to radiological pollution of water. In the guidelines of drinking water quality, WHO has included the guideline values for different organic and inorganic materials, thereby raising the awareness and proposing a research problem for the scientific community [4,5]. Along with WHO, the Environmental Protection Agency (EPA) has also set drinking water regulations, suggesting their own permissible limits for heavy metal the ions [6,7].

In order to combat the heavy metal ion pollution in water, effective and rapid sensing technologies are desired. The standard laboratory procedures such as inductively coupled mass plasma spectroscopy [8], high-performance liquid chromatography [9], mass spectroscopy [10], atomic

fluorescence spectroscopy [11], etc., involve sophistication and require trained personnel. The delicate instrumentation and handling of such systems introduce further cost to the system and make these unsuitable for being deployed in field analysis. These impairments of the conventional procedures motivate researchers to explore alternatives. In that direction, there are copious works in the literature dealing with various sensing procedures. For instance, colorimetric procedures [12], being solely a visual identification scheme, suffer from lack of accuracy. Similarly, electrochemical analysis is also impaired by degradation of the electrodes [13–15] with time. Likewise, surface plasmon enabled analysis [16] suffers from signal loss, if not controlled properly. All these lacunae necessitate an innovative way of sensing pollutants in aqueous solutions. In this regard, researchers have exploited several materials and techniques —out of which use of 2D layered semi-conducting transition metal dichalcogenides (TMDCs) is comparatively a recent approach. 2D layered TMDCs are advanced multifunctional materials having the generic formula MX₂, where M represents a transition metal and X denotes a chalcogen. A single layer of typical TMDC consists of three atomic planes. The plane consisting of the metal atom is hexagonally packed. One metal atom is 6-fold coordinated with the chalcogen atoms. They are usually found in three polymorphs [17]. Adoption of par-

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Evidence of Laser-Induced Amplification of Random Noise in WS₂ Nanosheets Based Resistive System

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Electronic noises are indispensable part of stochastic resonance (SR) and other molecular neuromorphic devices. Considering the paramount significance of such devices, investigation of artificial noise generation with further amplification attributes of WS₂ nanosheets based resistive unit is endeavored. To realize it, Cu interdigital electrodes (IDE) are fabricated on copper clad and chemically exfoliated hexagonal WS₂ nanosheets are drop-casted on the surface of as-fabricated IDE. In ambient conditions as well as in the presence of resistance fluctuations and defects in the layered material, the resistive unit is found to develop noise with an average peak-to-peak fluctuation of ≈ 13 nA on the linear current–voltage (*I*–*V*) characteristics of the device. On performing irradiation with 5 mW red, green, and blue laser on to the very fabricated unit, a maximum ≈ 60 nA current fluctuations in the *I*–*V* characteristics are detected. Additionally, the current fluctuation is found to be more prominent for green laser than that of red and blue laser.

1. Introduction


The 2D semiconducting transition metal dichalcogenides (TMDCs) have received considerable attention for their strong light–matter interaction. They have high quantum luminescence efficiency^[1] and have the ability to perform the role of optical resonators—thereby strengthening the coupling process.^[2] They can be integrated into an electronic system to absorb or detect the incident photons.^[3,4] It was observed that multilayer TMDCs can provide wider spectral response.^[5] The photocurrent for the shorter wavelength is found to be higher in comparison to the longer wavelength.^[6] Although, copious research articles have reported light–matter interaction in TMDCs, very few reports address the usefulness of the same in neuromorphic device applications.

In general, neuromorphic devices constitute the basic components of neuromorphic computing. Precisely, neuromorphic computing is the next-generation computing, which promises efficient processing of complex information than the conventional von Neumann Computing.^[7] The concept of

neuromorphic devices is based on the functioning of a human brain involving synapses and neurons. So far, this idea of neuromorphic computing was realized using machine learning and complementary metal–oxide semiconductor-based technology. However, toward achieving effective emulation of the brain, the electronic devices should function as artificial synapses and neurons.^[8] As such, there has been going on extensive research in the quest for better material and device architecture.^[9–11] One of the biological phenomena emulating the electronic system as well as having impact in neuromorphic devices is the stochastic resonance. This is a phenomenon where a threshold value of noise is used to extract the information from a weak signal.^[12] The first stochastic resonance phenomenon in an electronic

system was observed in a Schmitt trigger circuit^[13] and thereafter it was reported to be observed in many electronic devices such as superconducting quantum interference devices (SQUID),^[14] field-effect transistors (FETs),^[15–17] photodetectors,^[12,18,19] etc. Generally, these devices engage external noise generators,^[20,21] which may set limit to the dimensionality and applicability of the system. Accordingly, researchers designed experiments for *in situ* generation of noise.^[22,23] Despite this, very few focused works exist in this direction. As such, the present work illustrates the process of fabrication of an electrical unit for intrinsic generation and amplification of random noise. To realize the same, the present work gives importance to the consideration of the fact that 2D electrical system generates $1/f$ noise. Studies on low-frequency $1/f$ noise in 2D systems suggest that the fluctuations in 2D electronic system can be tuned by changing the ambient condition and post-fabrication processes as performed on the system. As for instance, upon exposure to ambient condition or annealing, characteristics of noise can be altered in MoS₂-based FET.^[24–26] Again, due to atomically thin nature and having great surface to volume ratio, these materials are sensitive to the surrounding. Hence, their electrical properties are influenced by substrates, unlike the bulk counterparts and noise fluctuations can be witnessed accordingly.^[27] Apart from these, noise generation in layered materials also takes place due to lattice defects and grain boundaries.^[28,29] Although noise in electrical systems is not desired, characterizations of noise have been performed with multitude of objectives, which include detection of chemicals,^[30] exploration of physics of resistance fluctuations, localization physics, etc.^[31] Thus, in this work, one of the most promising semiconducting layered TMDC materials, that is, WS₂ was used

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Phase transition and formation of inorganic fullerene type nanostructure in WS₂ nanosheet system under laser excitation

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ABSTRACT

Here in, laser power and energy dependant chemical and structural changes in WS₂ nanosheets have been reported. The nanosheets were obtained through liquid phase exfoliation in N-methyl pyrrolidone (NMP). Simultaneous laser irradiation and Raman spectral analysis were performed on the nanosheets. It was observed that laser power enhancement leads to further exfoliation and red shift of prominent phonon modes E_{12g}¹ and A_{1g} in the nanosheets. Oxidation of the nanosheets was also witnessed with a minimum 50 mJ of laser irradiation. Raman spectral analysis also reveals the transition of WS₂ from 2H (hexagonal symmetry, one layer per repeat, trigonal prismatic coordination to 1T (tetragonal symmetry, one layer per repeat, octahedral coordination) crystallographic arrangements with a minimum 50 mJ of laser energy. Formation of incipient inorganic fullerene type nanostructures was also confirmed in the laser irradiated WS₂ nanosheets. It is anticipated that such experimental findings will boost further laser induced treatment of transition metal dichalcogenides for novel applications.

Few and multilayered semiconducting two-dimensional transition metal di chalcogenides (2D TMDCs) have been extensively studied due to their intrinsic properties as well as their use in various applications [1–5]. TMDCs are usually found in several polymorphs, namely, 1T, 2H, 3R, 1T' etc. Here, the numbers stand for the number of layers and the letters indicate symmetry (*T*-trigonal, *H*-hexagonal, *R*-rhombohedral, *T'*-monoclinic) [6,7]. In TMDCs, polymorphism is supposed to be an excellent mean for improvisation of the material properties [8–11]. Recently, amongst the TMDCs, WS₂ has earned a lot of attention in this regard [12,13]. WS₂ has two distinct symmetries depending on the arrangement of S atoms, namely, 2H phase (hexagonal symmetry) and 1T phase (tetragonal symmetry). Both phases possess their own characteristic properties—1T phase has superior catalytic activity and conductivity than 2H phase [12]. With the aid of different synthesis procedures, phase transition can be triggered in WS₂ systems. The most established synthesis technique is the lithium intercalation method. Voiry et al. used this method in order to increase the 1T sites—thereby enhancing the catalytic activities and conductivity [12]. Phase transition from 1H to 1T' phase can also be initiated by applying pressure (~GPa) on WS₂ system [14]. Further, local structural phase transition can also be observed in WS₂ nanosheets in a WS₂/Au/mica system via thermal annealing [15]. 2H to 1T phase transition of WS₂ has also been


observed for electrochemical synthesis of WS₂ nanosheets [13]. Apart from these, phase transition from 1T to 1H phase in WS₂ system has also been introduced with the help of light and laser driven techniques which can be explained by atomic distortion mechanism [16]. Amongst all these different phase transition techniques, laser assisted processes are generally considered to be robust because they can generate local effect and are of non-contact mode e.g. high quality heterophase homo-junction ohmic contact can be fabricated between hexagonal (2H) and monoclinic (1T') MoTe₂ by laser induced phase patterning [7]. Although, laser driven phase transition have been introduced to different TMDCs and WS₂ system [16–19], laser induced 2H to 1T phase transition has not been reported so far.

Apart from polymorphism, laser induced oxidation is another interesting area for manipulation of material properties [20]. Laser induced formation of sub-stoichiometric WO_{3-x} polycrystals substantially increases the conductivity of WSe₂ [21]. Along with phase transition and oxidation, laser assisted thinning is another phenomenon which occurs simultaneously with other allied phenomena [7,18,22]. Besides, phonon shifts are inevitable in such processes [23,24]. Laser-ablation can also generate Inorganic Fullerene (IF) like structures in 2D materials [25–27] which is considered to be the superior lubricants for harsh conditions [15].

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A novel electrochemical synthesis of graphene oxide: Converting waste product to utility

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Abstract – We comprehensively report here a green route for synthesizing graphene oxide (GO) from waste Zn-C dry cell batteries. Here LiCl was used as electrolyte for the electrochemical synthesis of GO from graphite rods extracted from waste batteries. The satisfactory yield of 0.0305 g/h, formulated wholly through basic medium (at pH 9.36), was validated through confirmatory characterizations such as X-ray diffraction (XRD) analysis, Fourier-transform infrared spectroscopy (FTIR), UV-Vis and Raman spectroscopy, etc. A prominent diffraction peak around 26.70° in the XRD spectra and two prominent excitonic peaks around 1581.31 cm^{-1} and 2720.25 cm^{-1} in the Raman spectra were observed for the obtained samples. The overall scheme turned out to be engaging less reactants as well as facile; thus, converting a waste material to a useful product.

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Introduction. – Graphene and its derivatives have been attracting researchers with their unique chemical and physical properties [1–8]. One of these chemically active derivatives of graphene is GO, which finds use in several applications such as catalysis, sensing [9–12], etc. It can be easily reduced to graphene by different methods [13,14]. Regarding the synthesis of GO, several groups resorted to various processes of synthesis. While some researchers have opted for conventional chemical methods such as hummer's method [14] and electrochemical (EC) methods [13,15], others have engaged with either modifying the existing methods [16] or implementing unconventional ideas [17] which can address the problems involving the efficiency and environmental impact of the existing synthesis processes. Driven by the idea of greener synthesis, several groups have considered discharged battery rods/graphite rods to be the source of GO and graphene in the electrochemical synthesis of GO and graphene where H_2SO_4 [13], sodium dodecyl benzenesulfonate (SDBS) [15], mixture of KOH and $(\text{NH}_4)_2\text{SO}_4$ [18], etc., have been used as electrolytes. As analyzed from the data reported in the literature, the maximum yield was for the H_2SO_4 (acidic) electrolyte [13] which was $\sim 1.114\text{ g/h}$.

Molten alkali chlorides such as LiCl, KCl, NaCl, etc., and their mixtures have been used as electrolytes to produce nanostructured carbonaceous products including graphene [19–21]. The reaction at the cathode involves the intercalation of the alkali metal into the graphite electrode

and destabilization of the layered graphite structure, thus initiating the formation of carbonaceous nanostructured species [22]. Although the quality and quantity of the nanostructures are believed to be dependent on the nature of the molten state of the alkali chlorides [19], in order to develop a synthesis process of GO in a basic environment with high yield through EC exfoliation of graphite rods, LiCl at room temperature was employed as the electrolyte in the current investigation. EC synthesis of GO from graphite rods (extracted from drained battery) in LiCl was performed in both acidic and basic environment at room temperature. In both cases the exfoliation of graphite rods was observed. In the case of basic environment, the exfoliation was higher than in the acidic.

Experimental details. –

Chemicals and instrumentation. Waste Zn-C batteries were collected from electronic shops and gadgets. HCl, LiCl and buffer (pH-10 \pm 0.02, KCl + boric acid + NaOH) were purchased from Sigma Aldrich. The pHs of the solutions were measured with the help of pH 700 (tabletop pH meter) by Eutech instruments. The crystal structure of the as-synthesized sample was examined by X-ray diffraction (XRD, Rigaku) at a rate of 1° min^{-1} . The optical characterizations were performed with the Shimadzu UV-Vis spectrophotometer with quartz cuvette having pathlength 10 mm, FTIR spectrometer and Raman spectroscopy (RENISHAW, model no- Basis Series, 514 nm Ar + laser line).

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A novel green approach towards synthesis of silver nanoparticles and its comparative analysis with conventional methods

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Abstract

The quest for novel synthesis method of silver nanoparticles (AgNPs) is still an active field of research due to their usefulness in different levels of therapeutic and sensing applications. Owing to the better biodegradability and biocompatibility, plant extract-assisted synthesis processes of AgNPs have gained considerable attention in recent years. Here, we report a novel synthesis approach for AgNPs using banana root bulb (BRB) extract. The BRB extract was used as solvent as well as reducing agent in this synthesis process. The nanoparticles obtained through this process possessed hydrodynamic size of the order of 40 nm and a broad surface plasmon resonance peak at ~411 nm. In order to validate this process, the properties of the product were compared with properties of the nanoparticles obtained from other two established approaches viz- chemical reduction method involving trisodium citrate as reducing agent and green synthesis method involving mixed fruit peel waste extract as solvent and reducing agent. It was observed that surface plasmon resonance peaks entitled to AgNPs prepared through all the three approaches were quite consistent with each other. Besides, the broad surface plasmon resonance peaks suggest that the nanoparticles obtained through all the approaches were polydispersed.

1 Introduction

Amid several metal nanoparticles (NPs), silver nanoparticles (AgNPs) have received great attention due to their antimicrobial and antibacterial activities as well as therapeutic applications (as virucidal agents, photosensitizers, antiangiogenic agents, etc.) [1, 2]. Due to their large surface-to-volume ratio, they are sensitive to changes in the environmental conditions. As such, they have been used in different sensing applications. Owing to their plasmonic nature, they are often used in optical sensing applications, imaging, etc. [3–5]. Silver nanoparticles can be produced by various physical and chemical methods. Laser ablation and evaporation–condensation methods are some of the well-known physical methods for synthesis of silver nanoparticles. There are chemical methods where only organic or inorganic reducing agents are sufficient for synthesis of silver nanoparticles. However, in order to attain particular shape and size

and to improve stability, other agents like capping agents or stabilizing agents can be used in the synthesis process. There are numerous chemical methods where particle irradiation, microwave and ultraviolet irradiation, visible light irradiation are used. In electrochemical reduction technique, electrical energy is used for initiating the synthesis process [6]. Though these processes can effectively produce silver nanoparticles, researchers are in quest of greener techniques; so that, the whole process becomes more environment friendly. Recently, numerous green techniques have been investigated where various plants, algae (green, brown, red, blue), fungi, yeast, bacteria, actinomycetes, etc. have been used to produce AgNPs with different sizes and shapes [1, 7, 8]. The proteins [9] and biopolymers [10] found in various plants and microorganisms help in reduction of the metal salt as well as nucleation of the metal nanoparticles. It has also been reported that the biopolymers such as polysaccharides extracted from plants have appreciable biological activities [11–14]. Therefore, using plant extracts can be a great choice for production of metal nanoparticles with enhanced biological activities. In this regard, different parts of the banana plant have already been extensively exploited for synthesis of AgNPs. Preparation of silver nanoparticles using banana peel extract has been carried out by several groups. It turns out that the surface plasmon resonance (SPR) peaks of such

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A Preliminary Investigation Towards Detecting Heavy Metal Ions with a Cost-Effective Scheme

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Abstract: We report the findings of preliminary investigation corresponding to an optical detection technique implementing smartphone as our receiver towards quantitative assessment of heavy metal ions, namely, Cu, Zn, and Ni. Using intensity modulation, the optical responses are attained through a user-friendly app. The sensing region is made up of optical fiber whose cladding portion has been etched. Subject to varying concentrations of these metal ions, the modulated responses are attained, which reveal a declining trend. The absence of traditional parts such as a spectrophotometer makes the reported scheme cost-effective as well as field-portable.

Keywords: citrus species; cultivars; electrochemical biosensor.

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1. Introduction

Aquatic pollutants in the form of heavy metal ions are of major concern when they exceed the permissible limit, as mandated by the World Health Organisation and Environmental Protection Agency [1-4]. The rampant industrialization, as well as mushrooming anthropogenic sources, has aggravated the problem of aquatic pollution. Starting from the ailments to causing the undesirable affects in flora and fauna, researchers have been engaged in constant efforts to curb this form of aquatic pollution. Accordingly, the first goal is to identify/select the heavy metal ions present in water bodies. Removal procedures then follow this. Until and unless there is an effective assessment of these hazardous metal ions, the diminution process remains ineffective. Arsenic, Cobalt, Zinc, Nickel, and Copper deserve specific mention [5-7]. Because of their inevitable nature of bioaccumulation, they pose a serious threat to living creatures, including human beings.

As far as the detection of heavy metal ions is concerned, there exist several standard techniques such as atomic absorption spectroscopy [8-9], atomic emission spectroscopy [10-12], inductively coupled plasma mass spectrometry [13]. These standard procedures require trained personnel and the designated labs, incurring huge expenditures in terms of cost and maintenance. On the contrary, colorimetric as well as optical detection techniques happen to have cost expensive. They can be assembled in a small space along with minimal logistics. Recently, Biswas and his group have done extensive research in detecting heavy metal ions implementing colorimetric, optical techniques as well as electrochemical techniques [14-15]. An optical detector was used in most of these implementations, which was then integrated with a readout system. Although cost-effective, the intensity fluctuations may induce certain errors

A Preliminary Investigation Towards Detecting Heavy Metal Ions with a Cost-Effective Scheme

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Correlation of Surface Plasmon Resonance Wavelength (SPR) with Size and Concentration of Noble Metal Nanoparticles



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Abstract

Surface plasmon resonance SPR basically refers to collective oscillation of conduction electrons. Having the intrinsic property of sensing the surface properties, SPR have already received tremendous attention in the field of biomedical, Nano sensors, photovoltaics etc. Scientific data have also revealed that it is also highly characterized by the structure, type and dimension of the host nanoparticles, which eventually can lead us to a systematic correlation between different physical attributes of the host nanoparticles and SPR. In this work, we try to make a simplistic approach of correlating size, shape of gold (Au) and silver (Ag) nanoparticles (NPs) with their corresponding peak wavelength. It has also been attempted to bring the concentrations in the same frame. The computed results and the observed results are compared and matching up to a considerable extent has been shown in this work. It is envisioned that this small-scale correlation will help in giving a first insight towards characterization of Au and AgNPs just based only on SPR peak.

Keywords: SPR; Nanoparticles; MATLAB

Introduction

Every property of a material has a characteristic or critical length associated with it; e.g. scattering wavelength for electrical conductivity, plasma frequency (can be converted easily to wavelength) for optical property etc. The fundamental physics and chemistry changes when the dimensions of a solid become comparable to one or more of its characteristic wavelengths, many of which are in the nanometer range. Therefore, in the nano regime, novel physical and chemical properties can be experienced, e.g. yellow shiny bulk gold turn into red when the diameter of it is restricted to 10nm [1-15]. One of the very tempting properties of the nanoparticles is that their properties depend on their size, shape and density. As for instance, in medical applications, the size of the nanoparticles should be less than 100nm. This is because, above this dimension, the nanoparticles become toxic to the living body. So, to implement nanoparticles in different applications, having reliable information about the size, shape and density is indeed imperative. In order to determine the size, shape and density we usually incorporate different techniques. TEM, SEM are used for determining the size and morphology, respectively. Similarly, AFM provides information of shape. These techniques are not in situ, some of them are costly and some processes are time consuming. But with the help of some extraordinary properties of metal nanopar-

ticles called Surface Plasmon Resonance (SPR), the aspects like-size, shape and density of the metal nanoparticles can be feasibly extracted, saving of energy and time.

When total internal reflection occurs in a metal surface, then a wave moves along the surface of incidence which is referred to evanescent wave [2-5]. This wave occurs due to the interaction of electrons in the surface with the electric field of light. In case of metal bulk, the electrons absorb the light and oscillate exactly opposite to that of the incident wave and we perceive this as reflection of light. Therefore, metal appears to be shiny. But in case of metal nanoparticles, if the incident wave is nearly equal to that of the resonance frequency of the NPs, then they oscillate along with the electric field which is called Surface Plasmon Resonance. As such, they absorb a wavelength of light which we call SPR wavelength [1-7]. As the size of the nanoparticle changes, the resonance frequency of the particle changes. SPR wavelength also changes. The above descriptions eventually imply that there may be a quite sound correlation among the SPR wavelength, size, shape and density which can turn out to be standard equation [5-8].

Standardization of the equation can help the researchers to predict the size, shape and density of the sample without going into further investigations. So, this study is mainly aimed to find