RASĀYAN *J. Chem.*
Vol. 19 | No. 1 | 174-179 | January-March | 2026
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

HYDROTHERMALLY SYNTHESIZED MoO_2/rGO NANOCOMPOSITE FOR SUPERCAPACITOR and ELECTROCHEMICAL SENSING OF HEAVY METAL IONS (Pb^{2+}) APPLICATIONS

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ABSTRACT
Reduced graphene and metal oxide nanocomposites have been a lucrative material for developing supercapacitors as well as electrochemical sensors. In the present study, we have prepared molybdenum oxide and molybdenum oxide/reduced graphene oxide nanocomposite (MoO_2/rGONC) by the method of hydrothermal synthesis. The structural and morphological analyses were conducted using X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM), and Scanning Electron Microscope (SEM). Electrochemical analysis was performed by using Cyclic Voltammetry (CV). Electrochemical characterisation yielded the specific capacitances (SC) of 179.1 F/g (MoO_2) and 340.1 F/g (MoO_2/rGONC) at 1 A/g, retaining 90–94.5 per cent of its capacitance after 2000 cycles. The lead (Pb^{2+}) sensor was constructed by modifying the MoO_2/rGONC. MoO_2/rGONC electrode demonstrated efficient lead ion detection, suggesting its potential for sensing applications. The dual functional usage of the prepared materials, which demonstrates their energy storage and effectiveness for sensors, is the vital outcome of the study and opens ways for sustainable and versatile devices.

Keywords: MoO_2; MoO_2/rGO nanocomposite; Pb^{2+} detection; energy storage; Electrochemical Sensing; Hydrothermal Synthesis.

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INTRODUCTION

Molybdenum oxide, also known as molybdic oxide or MoO_2, is an oxide of the transition metal molybdenum. MoO_2 is widely used in catalysis, fuel cells, sensors, and polishing agents due to its exceptional physicochemical properties.[1-3] It is also known as a strong oxidising agent and is accessible as molybdenum oxide combined with oxygen atoms.[4] These characteristics make molybdenum oxide a perfect option for a number of uses, especially when it's in the form of nanoparticles.[5] MoO_2-based composites have attracted a lot of attention due to their numerous applications in adsorption, hydrogen production, photo-catalysis, sensing, semiconductor devices, and the biomedical industry.[6] However, its useful electrochemical performance is limited by its comparatively low electrical conductivity; rGO is used as a conductive support to get around this restriction because of its greater surface area, superior conductivity, and chemical stability. Charge transfer and electrochemical responsiveness are improved by the MoO_2/rGONC synergistic combination[8] of the conductive network of rGO and the redox characteristics of MoO_2. Similar improvements employing reduced graphene oxide-based composites, such as CuO-NiO/rGO for energy storage and CuO@NiO/g-C_3N_4 for photocatalytic and electrochemical applications, have been shown in recent investigations.[9-11] rGO exhibits good electrical conductivity, thermal stability, and mechanical strength.[12] Nevertheless, only a few research have looked at the dual use of MoO_2/rGONC for sensor and supercapacitor functions.[13–15] MoO_2/rGONC combines the unique properties of rGO and MoO_2 to produce materials with enhanced performance for a variety of applications. Lead (Pb), cadmium, mercury, and other hazardous substances were often discovered in mining, electronics, lighting, and medical wastewater. Living beings are seriously harmed by these substances [16] Rapid developments in clean energy storage systems employing novel materials and design techniques are further highlighted in recent literature to achieve the United Nations Sustainable Development Goals.[17–18] These results strongly encourage the investigation of multifunctional MoO_2/rGONC,

http://doi.org/10.31788/RJC.2026.1919553

which acts as an effective supercapacitor material as well as sensor electrodes.[19–20] Despite these developments, not much research has been done on the dual use of MoO_2/rGONC in lead metal ion detection and supercapacitor performance. The present study focuses on the synthesis of MoO_2 and MoO_2/rGONC by the hydrothermal route and examines their structural, morphological and electrochemical analysis, Pb^{2+} ion electrochemical sensing capabilities. This study is related to the synthesis of materials for supercapacitor applications, which comes under the United Nations Sustainable Development Goal-7.

EXPERIMENTAL

Materials

All the substances involved in the synthesis were of analytical grade and didn't require further refinement. Hydrogen peroxide (H_2O_2), Molybdenum (Mo) powder, ethylene glycol ($C_2H_6O_2$) (>98%), reduced graphene oxide (rGO) (>99%), and ethanol (C_2H_5OH).

Synthesis of MoO_2 NPs and MoO_2/rGONC

MoO_2 nanoparticles produced using the hydrothermal method. 10 mL of 25% H_2O_2 was added to 1 gram of Molybdenum powder while continuously stirring until a clear orange solution was achieved. Next, the mixture was combined with 40 mL of deionised water (DW) and 20 mL of $C_2H_6O_2$. After stirring the mixture for roughly 40 minutes until it became clear yellow slurry, it was immediately placed in an autoclave lined with Teflon and heated to 200 ^{0}C for 72 hours. Eventually, the samples were subjected to centrifugation and cleaned using C_2H_5OH and DW. Black granules were produced after a 24-hour vacuum drying at 65 °C. The MoO_2/rGO nanocomposite was also synthesised via the hydrothermal route. An optimisation procedure was carried out to get ideal precursor concentrations. 30 mL 1 M $C_2H_6O_2$ was ultrasonicated with 0.04 grams of reduced graphene oxide for 60 minutes. Later, 0.06 grams of MoO_2 was placed upon completion of 30 minutes of stirring time. It was immediately placed in an autoclave lined with Teflon and heated to 200 ^{0}C for 72 hours. After cooling, the black material underwent centrifugation and was rinsed multiple times with DW and C_2H_5OH until it became translucent. Finally, the prepared material underwent drying in an oven at 60 °C for 24 hours. In order to prepare the electrode, 80 weight per cent MoO_2 or MoO_2/rGONC, 15 weight per cent acetylene black, and 5 weight percent polyvinyliden were combined in an agate mortar. After this, the slurry was evenly coated on copper foil and subjected to drying at 85 ^{0}C for 6 hours.

Fig.-1: Schematic Diagram for the Preparation of MoO_2/rGONC

To guarantee correct contact with the electrolyte, the electrode was submerged in 3 M KOH for approximately half an hour before use.[21]

RESULTS AND DISCUSSION

XRD Study

Figure-2 corroborates the identified diffraction patterns of MoO_2/rGO, (011) at ~26.27°, (002) at ~37.2°, (220) at ~53.9°, (031) at ~60.34°, (131) at ~66.9°, which fit very nicely with JCPDS No. 65-5787.

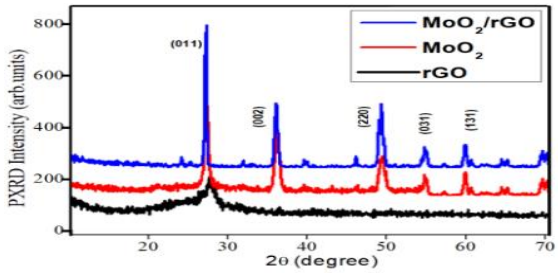


Fig.-2: XRD Spectra of Prepared MoO_2/rGONC, MoO_2, and rGO

The synthesised MoO_2/rGONC possesses a simple monoclinic structure with high crystallinity. The average nanocomposite particle size was 12.2 nm.

Morphological Studies

Figure-3(a, b) represents the SEM analyses of the prepared MoO_2/rGO nanocomposite. The particle size distribution was obtained by using ImageJ, an image analysis program, to measure particles that are about 10 nm in size.

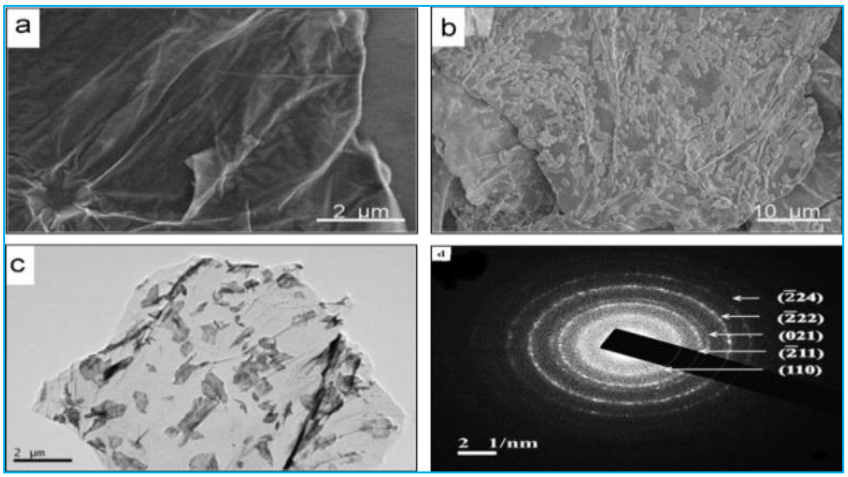


Fig.-3: Images of (a, b) SEM, (c) HR-TEM, (d) SAED of MoO_2/rGONC

The SAED pattern, in Fig.-3(d), shows the material crystalline structure by the appearance of concentric rings related to (224), (222), (021), (211), and (110) planes. The HR-TEM pictures unmistakably showed widely distributed MoO_2 affixed on thin, transparent, rGO layers. The frontier between the MoO_2 and the rGO support is distinctly observable, signifying robust contact and effective dispersion attained via the hydrothermal method.

EDX Analysis

The sample contains molybdenum (Mo), oxygen (O), and carbon (C), according to the EDX analysis, which is displayed in Fig.-4.

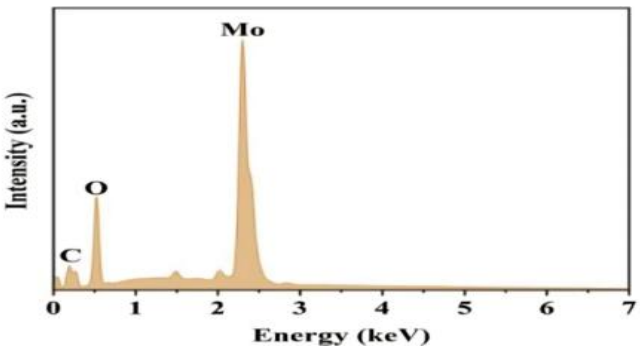


Fig.-4: EDX Spectra of MoO_2/Rgonc

The measured ratios of Molybdenum and Oxygen atoms differ from the expected stoichiometry of MoO_2 (1:2) because of the impact of reduced graphene oxide and semi-quantitative aspects of EDX. However, in spite of these constraints, the findings provide a qualitative confirmation of the hybrid characteristics of the material.[22]

Electrochemical Activity

For cyclic voltammetry (CV) investigations, rGO, MoO_2 and MoO_2/rGONC were employed as modified working electrodes. The electrodes employ 1 M KOH as their electrolyte, and scan rates are done between 10 and 40 mVs^{-1}. The CV graphs in Fig.-5 (a, b and c) are within the possible window between 0.0 - 0.5 V. MoO_2 and MoO_2/rGONC have shown mini oxidation peaks at 0.2V and 0.32V. The substantial fluctuation in the dispositions of the electrode anodic peaks across successive cycles indicates wonderful electrode stability.[23]

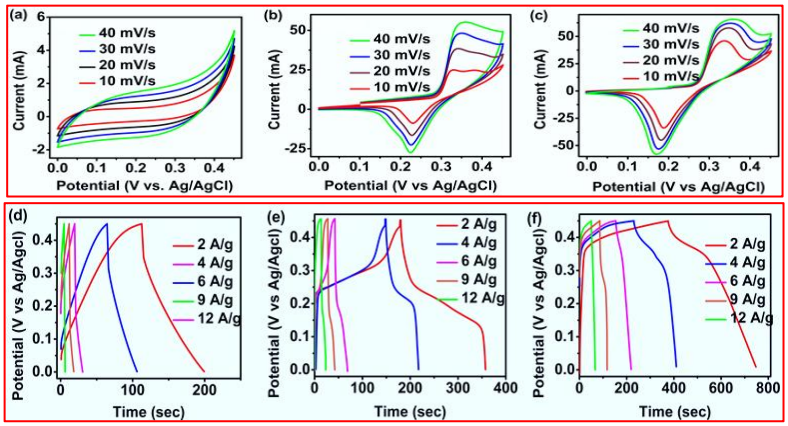
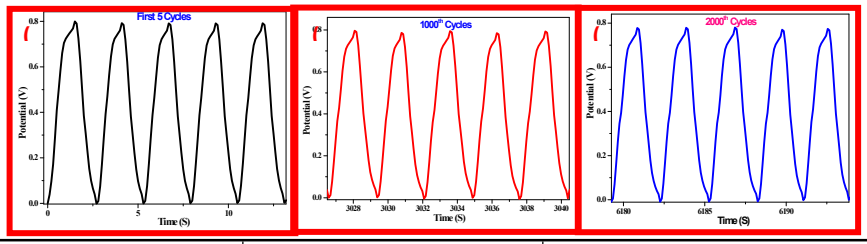


Fig.-5: Cyclic Voltammograms of Electrode (a) rGO, (b) MoO_2, and (c) MoO_2/rGONC. GCD curves of (d) rGO (e) MoO_2 and (f) MoO_2/Rgonc

The CV graphs of MoO_2 and MoO_2/rGONC exhibited remarkable redox peaks, signifying a pseudocapacitive characteristic. After 2000 cycles, the MoO_2/rGONC electrode maintained around 94.5% of its initial current response, showing good electrode stability. The potential produced in the MoO_2/rGONC electrode showed greater cyclic stability, preserving 95%, 93%, and 90% of the beginning capacity after the fifth, 1000th, and 2000th cycle, respectively, according to Fig.-6.

Fig.-6: (a,b,c) GCD Profiles of MoO_2/rGONC Electrode at 1 A/g for 5, 1000, 2000 Cycles

Table-1: The Specific Capacitance of MoO_2 and MoO_2/rGONC Electrodes by using C-D Graph



Current Density (A/g)	MoO_2 Specific Capacitance (F/g)	MoO_2/rGONC Specific Capacitance (F/g)
5	111.5	220.6
4	126.6	260.1
3	147.5	280.5
2	158.5	314.4
1	179.1	340.1

Table-2 displays the important parameters obtained from the fitted Nyquist charts in Fig.-7. The MoO_2/rGONC showed a noticeably reduced R_s = 5.13 Ω and R_{ct} = 0.000323 Ω in comparison with pure MoO_2 (R_s = 6.48 Ω and R_{ct} = 0.0095 Ω), showing excellent conductivity.[25]

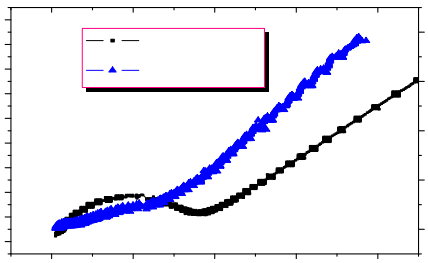


Fig.-7: Nyquist Plot of MoO_2 and MoO_2/rGONC

Electrochemical Sensing Analysis

Differential Pulse Voltammetry (DPV) analyses were employed to detect Pb^{2+} concentrations between 0.02 µM – 0.2 µM on the MoO_2/rGONC electrode, as illustrated in Fig.-8. The results showed that the electrodes' performance to lead in the range of 0.02 µM – 0.2 µM, with observed peak currents. The

linear curves confirmed the excellent sensing capability of the hybrid electrode for efficient lead concentration detection. [26]

Table-2: Parameters from Nyquist Plot

Electrodes Name	Rs (Ω)	Rct (Ω)	Cdl (F)
MoO_2	6.48	0.0095	0.0005846
MoO_2/rGONCs	5.13	0.000323	0.000962

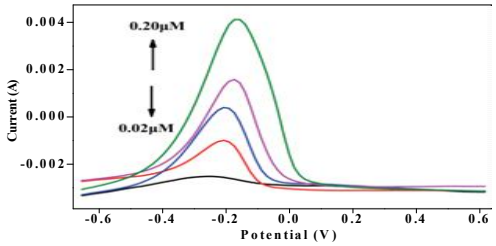


Fig.-8: DPV Curves of the MoO_2 /rGO for Pb(II) Detection in the Range of 0.02 - 0.2 μM

The electrode material exhibits capacitance retention of 94.5% following 2000 cycles, alongside an SC of 340.1 F/g at 1 A/g. The findings in the current study are comparable to the studies reported for MoO_2/rGO and other MoO_2-based materials produced by using different synthesis techniques.[27-28] As the main finding of this work is a high-performance supercapacitor material, which comes under the United Nations Sustainable Development Goals (SDGs), particularly SDG 7, Affordable and Clean Energy.

CONCLUSION

MoO_2 and MoO_2/rGO nanocomposite were prepared by the hydrothermal route and structurally confirmed by various characterisations. Exceptional capacitive behaviour was shown by electrochemical examination, with MoO_2/rGONC achieving a specific capacitance of 340 F/g at 1 A/g in comparison with 179.1 F/g for MoO_2. The Composite electrode exhibited excellent cycling stability upon completion of 2000 cycles. The DPV studies of MoO_2/rGONC were found to be valid as an electrochemical sensor for the detection of Pb^{2+}. These findings validate that MoO_2/rGONC is a multifunctional material with prospective uses in electrochemical sensors and supercapacitors, providing promise for the creation of sustainable energy devices. The present work is related to SDG-7: *Affordable and Clean Energy*, since this study is related to supercapacitors and sensors.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge JNTUA College of Engineering, Ananthapuramu, and JNTUA for support and facilities.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-7: Affordable and Clean Energy*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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Cite this article as: V. Ramanjaneyulu and T. Bala Narsaiah, *Rasayan J. Chem.*, 19(1), 174-179(2026), https://doi.org/10.317 88/RJC.2026.1919553.

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