Tuning the structure, optical, and magnetic properties of nanostructured NiMoO₄ by nitrogen plasma treatment

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Abstract
Herein, the nitrogen plasma treatment with different time irradiation (0, 90, 120, and 150 min) is used to tune the structure, optical, and magnetic properties of nanostructured NiMoO₄ NMO NPs. The XRD patterns revealed that the crystallinity of NMO samples increases with an increase in the N₂ plasma exposure time. The notable reduce in this peak' intensity for the sample at dose of 120 min may be attributed to the energy dissipated in the defect generation. Also, the crystallite size for NMO samples was found in the range (23.9–26.7) nm. Further, EPR is used to evaluate the impact of the treatment duration on the oxygen vacancy density. The total number of spins rises as plasma irradiation duration increases, revealing that the NMO NPs can be used as a dosimeter for plasma irradiation. The optical bandgap ranged from 2.92 eV to 3.24 eV as the N₂ plasma treatment duration changed. The saturation magnetization was enhanced with the rise of plasma treatment time. Furthermore, the Hc increases from 16.67 G for untreated NMO NPs to 128.41 G for N₂ plasma-treated NMO NPs for 150 min. The resulted optical and magnetic properties of N₂ plasma-treated NMO NPs make it candidate material for photocatalysis applications.

Keywords Nitrogen plasma · Nickel molybdate · Dosimeter · Optical properties · Magnetic behavior

1 Introduction
Due to their complex electronic configuration and cost-effectiveness, transition metal oxides (TMOs) are numerous interesting active materials. Nevertheless, their low usage effectiveness, low electron conductivity, and limited functional site density restricted their practical applications [1–3]. Binary metal molybdates (MMO₄; M = Zn, Bi, Cu, Ni,.) have recently been demonstrated to have exceptional characteristics that make them promising materials for significant areas such as water treatment [4], corrosion science [5], water splitting [6], antimicrobial agents [7], electrochemical electrode [8], and energy storage [9]. Between these, the NiMoO₄ NMO nanoparticles NPs have piqued the interest of scientists due to features like chemical stability, better electrochemical display, luminescence, magnetic performance, optics, and decolorization abilities at a lower cost [10–14]. Many investigations have been conducted to increase NMO properties, including as surface functionalization and elemental doping, with the notable results [15–17]. Also, NMO suffers from its high resistivity, limiting its uses in electrochemical applications. As a result, doping NMO with a suitable metal cation may boost the electrochemical characteristics. NMO NPs has mostly been used in the photoelectric area, including electrodes, supercapacitors, and photocatalytic purposes. As a photocatalyst, NMO was employed to decompose methyl orange. After 1 h of ultraviolet light irradiation, the degradation value was estimated to be 67 percent [14]. Also, due to Ni’s excellent electrochemical efficiency and Mo’s high electrical conductivity, NMO might be
a realistic choice for pseudo-capacitive active materials [18]. Sol–gel [13, 19], solid-state reaction [20], hydrothermal [21], co-precipitation [22, 23], and sonochemical [24] techniques have all been described for NMO production.

When an electric field is applied to a gas-forming plasma in a reactor, plasma is generated as a confined ionized gas. Plasma can be hot or cold and have high or low pressure [25]. The plasma treatment technique, which has recently been extensively utilized in surface modification/treatment, is a successful method for defect engineering and substituents injection. The plasma-assistant reaction yields free radicals, developing crystalline vacancies on inorganic materials, modifying the surface electronic redistribution, and providing appropriate active sites [26–30]. Furthermore, it is required to adjust the bandgap and enhance the ion/electron conductivities. Currently, hetero atom doping (N, B, F atoms) has been often used to reduce the bandgap, which may serve as an electron donor in the material’s bandgap and generate a mid-gap state, for enhanced usage in energy storage and conversion. In the same context, several oxygen vacancies and species might considerably improve electrical conductivity. Plasma treatment might be an effective technique for hetero atom doping in the material under investigation. Plasma comprises highly excited atoms with enough energy to attack the surface layer and break the chemical bond. The extremely engaged atoms then react with the investigated material to introduce a unique bond, resulting in effective hetero atom doping and the existence of multiple oxygen vacancies [31–34]. Recently, Liu et al.[35] developed a unique method for synthesizing significant atomic defects on NMO-like nanosheets using N2 plasma treatment, then filling these defects with heterocation dopants, and stabilizing them with sintering.

To our knowledge, there is no evidence of the use of nitrogen plasma to enhance the structural, optical, or magnetic characteristics of nickel molybdate NMO NPs. Herein, for the first time, nitrogen plasma treatment with different time irradiation (0, 90, 120, and 150 min) is used to tune the structure, optical, and magnetic properties of nanostructured NMO. The sol–gel technique was utilized to synthesize NMO as a facile and low-cost approach. The untreated and N2 plasma-treated NMO NPs were characterized via numerous tools: XRD, EDX, SEM, Electron paramagnetic resonance (EPR) spectroscopy, diffuse reflectance spectroscopy (DRS), and vibrating sample magnetometer (VSM).

2 Materials and methods

2.1 Materials

Ni(NO3)2·6H2O and (NH4)2MoO4 were considered sources for Ni and Mo, respectively. The citric acid monohydrate (C6H5O7·H2O) and ethylene glycol (C2H6O2) were used as fuel and gel initiation [36–40].

2.2 Preparation of NMO NPs

The NMO NPs were synthesized via the facile sol–gel technique, as illustrated in [41]. The as-synthesized NMO NPs were dried at 300 °C [36–40, 42]. Then 2 g of NMO NPs was pressed into the shape of a disk.

2.3 N2 plasma treatment

Four disks of as-synthesized NMO NPs were exposed to nitrogen plasma at different time irradiation (0, 90, 120, and 150 min). A plasma source is used to irradiate the NMO NPs in the charged particles Lap., Radiation Physics Department, National Center for Radiation Research and Technology, Egyptian Atomic Energy Authority. The vacuum system consists of rotary and diffusion pumps that are used to evacuate the system up to 10–4 mbar. The plasma source is placed inside the system where the backpressure inside the plasma source chamber is in the range 10–4 mbar, while the operating pressure is 2 × 10–3 mbar. The plasma source is DC plasma source where the cathode is cold cathode connected to the earth, and the anode is connected to positive voltage up to 4 kV, the discharge current is 2 mA. The NMO samples are placed inside the plasma source between the anode and the cathode, where the plasma is immersed in the NMO samples. Figure 1 depicts the fabrication of a cold d.c. plasma source. It has a stainless steel cylindrical anode and stainless steel disk cathode and a 5 mm inner aperture diameter. Teflon disks separate the anode from the cathode.

2.4 Characterization of NMO NPs

XRD (Shimadzu 6000) was used to illustrate the phase analyses of NMO NPs. SEM and EDX (JEOL JSM-5600 LV, Japan) were utilized to give information about surface morphology and elemental composition of NMO NPs. Fourier transform infrared FTIR spectroscopy (NICOLET iS10) is measured in range between 400 and 4000 cm−1. The EPR signals were acquired at room temperature using a CW X-band EMX EPR spectrometer (Bruker, Germany) and an ER 4102 standard rectangular cavity. During the EPR experiment, the following operating parameters were used: microwave power, 5.053 mW; modulation amplitude, 12.00 Gauss; modulation frequency, 100 kHz; the number of x-scans, 1; resolution in x, 1024 sweep width, 10,000 Gauss; microwave frequency, 9.668 GHz; time constant, 163.84 ms; conversion time, 163.84 ms; and sweep time, 167.77 s. A Jasco UV–visible spectrophotometer (V-670 PC) had utilized to present UV diffusion reflectance spectra.
3 Results and discussion

3.1 Structural analyses

Figure 2 exhibits EDX spectra and elemental mapping images of pristine NMO NPs. The images confirmed the existence of fundamental elements of NMO NPs (i.e., Ni, Mo, and O) without any foreign elements. Further, the elemental mapping images revealed the uniform distribution of all the investigated elements over the whole NMO sample [41].

Figure 3 shows XRD patterns of untreated and N₂ plasma-treated NMO NPs. The patterns confirmed the successful synthesis of NMO NPs without any exotic phases. The diffraction peaks of untreated and N₂ plasma-treated NMO NPs also matched (JCPDS cards No: 01-086-0361 & 45-0142) [43–45]. Similar results are presented by Keerthana et al. [46].

Further, the figure displayed the enhancement in the peak intensity of the preferred plane ($2\theta = 29.19^\circ$) as the exposure time for N₂ plasma was increased. This means that the crystallinity of NMO samples increases with an increase in the N₂ plasma exposure time. In other words, this energy
is dissipated to improve the ordered phases. The notable decrease in this peak’s intensity at the dose of 120 min may be attributable to the energy dissipated in the defect generation, resulting in an increase in the strain in the sample as presented in Williamson-Hall plots (Fig. 4) [47]. Further, the crystallite size for untreated and N₂ plasma-treated NMO NPs was calculated and is found to be 23.9, 24.3, 26.1, and 26.7 nm with the increase in the N₂ plasma exposure time.

Also, the peak at 2θ = 29.19° with a preferred orientation at the plane (220) was shifted toward the lower diffraction angles for the treated NMO samples (see Fig. 5). The interpretation of appearing this shift can be due to the unit cell expansion of NMO NPs, which means the growth of the lattice constant of the treated NMO sample [39].

Figure 6 shows the FTIR spectra of untreated and N₂ plasma-treated NMO NPs. In the case of pristine NMO NPs, the peaks that appeared at 647 cm⁻¹ can be attributable to the vibrations of the distorted tetrahedron (MoO₄) presenting in NMO NPs. Further, the bands observed at 3465 and 1685 cm⁻¹ may be ascribed to the stretching vibration and the flexing modes for O–H due to the absorption of water in NMO NPs surfaces, respectively. The peak noticed at 1440 cm⁻¹, which is attributable to the C–O stretching vibration, suggests the presence of carbon (from citric acid) in NMO NPs. The absorption characteristics of the –CH₃ and –CH₂ stretching are given by the peaks at 2994 cm⁻¹.

The bands at 970 cm⁻¹ can be assigned to the symmetric and antisymmetric stretching of the Mo = O linkage. The bands at 828 cm⁻¹ are assigned to the vibrations of the Mo–O–Ni.

Besides, the bands at 590 and 454 cm⁻¹ could be associated with the bending vibration of Mo–O–Mo attachment. Our
The shift in the position of the peak at $2\theta = 29.19^\circ$ for untreated and N$_2$ plasma-treated NMO NPs

The effect of N-plasma on the surface morphology of NMO NPs is illustrated in Fig. 7. SEM images of untreated and N$_2$ plasma-treated NMO NPs are presented in Fig. 7. Also, it is demonstrated that the untreated NMO NPs comprising agglomerated particles owned a considerable size. The figure revealed that the N-plasma possesses a marked effect on the surface morphology of NMO NPs. The remarkable discrepancy was caused by the variable surface reactivities caused by the different exposure times for N$_2$ plasma.

Furthermore, there were intragranular pores in the treated samples, which caused the density to rise. Microstructural holes are pores trapped within grains as a consequence of increasing grain development.

### 3.3 Electron paramagnetic resonance (EPR) spectroscopy

Because the nitrogen plasma treatment is predicted to create a large number of crystallographic defects and vacancies on the NMO NPs surface [30, 35], EPR is used to evaluate the effect of the treatment duration on the oxygen vacancy density [50]. The intensity of oxygen vacancies signal identified at $g = 2.175$ (Fig. 8) grows progressively as the nitrogen plasma treatment duration is extended, showing that nitrogen-based free radicals are gradually eroding the crystalline surface. We calculated the total number of spins formed in each sample (Table 1). We found that it increases as a function of plasma irradiation time, indicating the possible use of this material as a dosimeter for plasma irradiation.

### 3.4 Optical properties

Figure 9 shows the typical DRS spectra of the untreated and N$_2$ plasma-treated NMO NPs. It is marked from the figure that the wavelength at maximum reflection ($\lambda_{\text{max}}$) for the untreated and N$_2$ plasma-treated NMO NPs was seen at ranges between 200 and 300 nm.

The optical band gap can be evaluated using the reflectance results via Kubelka–Munk theory and Tauc’s equation [51, 52]. The optical bandgap ($E_g$) for untreated and N$_2$ plasma-treated NMO NPs decreases from 3.11 eV for pristine NMO NPs to 2.92 eV for N$_2$ plasma-treated NMO NPs at (150 min). The remarkable increase in optical bandgap (3.24 eV) for N$_2$ plasma-treated NMO NPs at (120 min) can be ascribed to the defect that occurs as presented in Fig. 10. The obtained optical behavior was matched well with XRD data.

### 3.5 Magnetic properties

Figure 11 presents the M-H curves for the untreated and N$_2$ plasma-treated NMO NPs with the field sweeping from $-20,000$ to $+20,000$ G at room temperature. The untreated and N$_2$ plasma-treated NMO NPs show superparamagnetic behavior. Table 2 summarizes primary magnetic parameters, which include saturation magnetization (Ms), remanence (Mr), coercivity (Hc), and squareness (Mr/Ms) estimated out from curves. The number of magnetic molecules in a single magnetic domain is known to be proportional to the energy of a magnetic particle through an external field [53, 54]. The pristine NMO NPs possess 0.0331 emu/g, $0.4370 \times 10^{-3}$ emu/g, and 16.6700 G
as the value of Ms, Mr, and Hc, respectively. These values increase with the increase of N2 plasma exposure time. The saturation magnetization (Ms) and remanence (Mr) values depend on the crystallite size. Both Ms and Mr increased to 0.1057 emu/g and 2.463 × 10⁻³ emu/g, respectively, for N2 plasma-treated NMO NPs at (150 min). The increase in Ms and Mr for N2 plasma-treated NMO NPs compared to untreated NMO has been mostly due to the increased particle size and spin canting at the NMO surface [51, 53]. The extraordinary drop in the value of Ms and Mr for N2 plasma-treated NMO at 120 min matched well with XRD and optical properties. The Hc is also affected by crystallite size, as shown in Fig. 12. The aggregation of magnetic nanoparticles enhanced their interaction [53]. The Hc is increased from 16.67 G for untreated NMO NPs to 128.41 G for N2 plasma-treated NMO NPs at (150 min). The optical and magnetic properties of N2 plasma-treated NMO NPs make them promising candidates materials for photocatalysis applications.

4 Conclusion

In this study, a sol–gel technique was utilized to synthesize NiMoO4. The impact of nitrogen plasma treatment on the structure, optical, and magnetic properties of nanostructured NMO NPs was investigated. The crystallite size increases from 23.9 nm to 26.7 nm with the increase in the N2 plasma exposure time. EDX spectra and elemental mapping images confirmed the existence of fundamental elements of NMO NPs without any foreign elements. The total number of spins
increases as a function of plasma irradiation time, indicating the possible use of NMO NPs as a dosimeter for plasma irradiation. Also, the values of Ms and Mr for N$_2$ plasma-treated NMO NPs increased than those for untreated NMO due to the increased particle size and spin canting at the NMO surface. Overall, the obtained optical and magnetic
properties of N₂ plasma-treated NMO NPs make them promising materials for photocatalysis applications.

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**Data availability** Not applicable.

**Declarations**

**Conflict of interest** The authors declare that they have no conflict of interest.

**Ethical approval** Not required.

**Consent to publish** Not applicable.

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**References**

1. P. Zhai, Y. Zhang, Y. Wu, J. Gao, B. Zhang, S. Cao, Y. Zhang, Z. Li, L. Sun, J. Hou, Engineering active sites on hierarchical transition bimetal oxides/sulfides heterostructure array enabling robust overall water splitting. Nat. Commun. 11(1), 1–12 (2020)

2. L. Hu, X. Zeng, X. Wei, H. Wang, Y. Wu, W. Gu, L. Shi, C. Zhu, Interface engineering for enhancing electrocatalytic oxygen evolution of NiFe LDH/NiTe heterostructures. Appl. Cat. B: Environ. 273, 119014 (2020)

3. J. Ji, L.L. Zhang, H. Ji, Y. Li, X. Zhao, X. Bai, X. Fan, F. Zhang, R.S. Ruoff, Nanoporous Ni (OH) 2 thin film on 3D ultrathin-graphite foam for asymmetric supercapacitor. ACS Nano 7(7), 6237–6243 (2013)

4. G. Yang, Y. Liang, K. Li, J. Yang, K. Wang, R. Xu, X. Xie, Engineering the dimension and crystal structure of bismuth molybdate photocatalysts via a molten salt-assisted assembly approach. J. Alloys Compd 844, 156231 (2020)

5. M. Wu, J. Shi, Beneficial and detrimental impacts of molybdate on corrosion resistance of steels in alkaline concrete pore solution with high chloride contamination. Corros. Sci. 183, 109326 (2021)

6. S.P. Keerthana, B.J. Rani, R. Yuvakkumar, G. Ravi, Y. Shiva-tharsiny, E.S. Babu, H.S. Almoallim, S.A. Alharbi, D. Velayuthapillai, Copper molybdate nanoparticles for electrochemical water splitting application. Int. J. Hydrogen Energy 46(11), 7701–7711 (2021)

7. Z. Xia, J. Min, S. Zhou, H. Ma, B. Zhang, X. Tang, Photocatalytic performance and antibacterial mechanism of Cu/Ag-molybdate powder material. Ceram. Int. 47(9), 12667–12679 (2021)

8. T. Ghiassi, S. Ahmad, E. Ahmad, M.R.T. Baval Olyai, Z. Khodadadi, Novel electrochemical sensor based on modified glassy carbon electrode with graphene quantum dots, chitosan and nickel molybdate nanocomposites for diazinon and optimal design by the Taguchi method. Microchem. J. 160, 105628 (2021)

9. M.I.A. Abdel Maksoud, R.A. Fahim, A.E. Shalan, M. Abd Elkodous, S.O. Olojede, A.I. Osman, C. Farrell, A.H. Al-Muhtaseb, A.S. Awed, A.H. Ashour, D.W. Rooney, Advanced materials and technologies for supercapacitors used in energy conversion and storage: a review. Environ. Chem. Lett. 19(1), 375–439 (2021)

10. B. Ramulu, S. Chandra Sekhar, G. Nagaraju, J.S. Yu, Rational design and construction of nickel molybdate nanohybrid composite for high-performance supercapaciters. Appl. Surf. Sci. 515, 146023 (2020)

11. C. Karami, M.A. Taher, A novel enzyme-less amperometric sensor for hydrogen peroxide based on nickel molybdate nanoparticles. J. Electroanal. Chem. 847, 113219 (2019)

12. W. Zhang, J. Yin, F. Min, L. Jia, D. Zhang, Q. Zhang, J. Xie, Preparation and photoluminescence properties of MMoO₄ (M = Cu, Ni, Zn) nano-particles synthesized via electrolysis. J. Mol. Struct. 1127, 777–783 (2017)

13. V. Unapathy, P. Neeraja, A. Manikandan, P. Ramu, Synthesis of NiMoO₄ nanoparticles by sol–gel method and their structural, morphological, optical, magnetic and photocatalytic properties. Trans. Nonferrous Metals Soc. China 27(8), 1785–1793 (2017)

14. A. Alborzi, S. Khademholhoseini, Nickel molybdate nanocomposites: synthesis, characterization, optical and photocatalytic properties. J. Mater. Sci.: Mater. Electron. 27(4), 3963–3967 (2016)

15. D. Muthu, S. Vargheese, Y. Haldorai, R.T. Rajendra Kumar, NiMoO₄/reduced graphene oxide composite as an electrode material for hybrid supercapacitor. Mater. Sci. Semicond. Process. 135, 106078 (2021)

16. T. Wang, H. Wu, C. Feng, Y. Ding, H. Mei, Ni, N-codoped NiMoO₄ grown on 3D nickel foam as bifunctional electrocatalysts for hydrogen production in urea-water electrolysis. Electrochim. Acta 391, 138931 (2021)

17. S.K. Ray, B. Joshi, S. Ramani, S. Park, J. Hur, Multicolor and white light upconversion luminescence in α-NiMoO₄·Yb₃⁺/Lu₃⁺ (Ln = Tm, Ho, Tm/Ho) nanoparticles. J. Alloys Compd. 892, 162101 (2022)

18. K. Xiao, L. Xia, G. Liu, S. Wang, L.-X. Ding, H. Wang, Honeycomb-like NiMoO₄ 4 ultrathin nanosheet arrays for high-performance electrochemical energy storage. J. Mater. Chem. A 3(11), 6128–6135 (2015)

19. A. Maione, M. Devillers, Solid solutions of Ni and Co molybdates in silica-dispersed and bulk catalysts prepared by sol–gel and citrate methods. J. Solid State Chem. 177(7), 2339–2349 (2004)

20. X. Lu, W. Jia, H. Chai, J. Hu, S. Wang, Y. Cao, Solid-state chemical fabrication of one-dimensional mesoporous β-nickel molybdate nanorods as remarkable electrode material for supercapacitors. J. Colloid Interface Sci. 534, 322–331 (2019)

21. K. Eda, Y. Kato, Y. Ohshiro, T. Sagitani, M.S. Whittingham, Synthesis, crystal structure, and structural conversion of Ni molybdate hydrate NiMoO₄·nH₂O. J. Solid State Chem. 183(6), 1334–1339 (2010)

22. A. Kaddouri, E. Tempesti, C. Mazucchia, Comparative study of β-nickel molybdate phase obtained by conventional precipitation and the sol-gel method. Mater. Res. Bull. 39(4–5), 695–706 (2004)
23. V. Jesseenthanari, A. Dayalan, K. Nagaraja, Nanocrystalline composite compositions of transition metal molybdate (Ni1–xCo xMoO4; x = 0, 0.3, 0.5, 0.7, 1) synthesized by a co-precipitation method as humidity sensors and their photoluminescence properties. J. Phys. Chem. Solids 115, 75–83 (2018)

24. G. Kianpour, M. Salavati-Niasari, H. Emadi, Sonochemical synthesis and characterization of NiMoO4 nanorods. Ultrason. Sonochem. 20(1), 418–424 (2013)

25. L. Minatti, C. Migliaretti, L. Lunelli, G. Viero, M. Dalla Serra, G. Sperranza, Plasma assisted surface treatments of biomaterials. Biophys. Chem. 229, 151–164 (2017)

26. Z. Xiao, Y. Wang, Y.-C. Huang, Z. Wei, C.-L. Dong, J. Ma, S. Shen, Y. Li, S. Wang. Filling the oxygen vacancies in Co 3 O 4 with phosphorus: an ultra-efficient electrocatalyst for overall water splitting. Energy Environ. Sci. 10(12), 2563–2569 (2017)

27. W. Zhang, Y. Tang, L. Yu, X.-Y. Yu, Activating the alkaline hydrogen evolution performance of Mo-incorporated Ni (OH) 2 by plasma-induced heterostructure. Appl. Catal. B: Environ. 260, 118154 (2020)

28. Y. Tang, Q. Liu, L. Dong, H.B. Wu, X.-Y. Yu, Activating the hydrogen evolution and overall water splitting performance of NiFe LDH by cation doping and plasma reduction. Appl. Catal. B: Environ. 266, 118627 (2020)

29. L. Meng, X. Zhou, S. Wang, Y. Zhou, W. Tian, P. Kidkunthod, S. Tunmee, Y. Tang, R. Long, Y. Xin, A plasma-triggered O– S bond and P– N junction near the surface of a SnS2 nanosheet array to enable efficient solar water oxidation. Angew. Chem. Int. Ed. 58(46), 16665–16675 (2019)

30. E.M. Liston, Plasma treatment for improved bonding: a review. J. Adhes. 30(1–4), 199–218 (1989)

31. D.-H. Wang, L. Jia, X.-L. Wu, L.-Q. Lu, A.-W. Xu, One-step hydrothermal synthesis of N-doped TiO2 2C nanocomposites with high visible light photocatalytic activity. Nanoscale 4(2), 576–584 (2012)

32. S. Dou, L. Tao, R. Wang, S. El Hankari, R. Chen, S. Wang, Plasma-assisted synthesis and surface modification of electrode materials for renewable energy. Adv. Mater. 30(21), 1705850 (2018)

33. R. Sharma, P.P. Das, M. Misra, V. Mahajan, J.P. Bock, S. Trigwell, A.S. Biris, M.K. Mazumder, Enhancement of the photoelectrochemical conversion efficiency of nanotubular TiO2 photoanodes using nitrogen plasma assisted surface modification. Nanotechnology 20(7), 075704 (2009)

34. J. Hu, L. Fu, R. Rajagopalan, Q. Zhang, J. Luan, H. Zhang, Y. Tang, Z. Peng, H. Wang. Nitrogen Plasma-Treated Core-Bishell Si@SiO x@TiO2–8: Nanoparticles with Significantly Improved Lithium Storage Performance. ACS Appl. Mater. Interfaces. 11(31), 27658–27666 (2019)

35. X. Liu, P. Liu, F. Wang, X. Lv, T. Yang, W. Tian, C. Wang. S. Tan, J. Ji, Plasma-induced defect engineering and cation refilling of NiMoO4 parallel arrays for overall water splitting. ACS Appl. Mater. Interfaces. 13(35), 41545–41544 (2021)

36. A.H. Ashour, A.I. El-Batel, M.I.A.A. Maksoud, G.S. El-Sayyad, S. Labib, E. Abdelwab, M.M. El-Okr, Antimicrobial activity of metal-substituted cobalt ferrite nanoparticles synthesized by sol–gel technique. Particulology 40, 141–151 (2018)

37. M.I.A.A. Maksoud, G.S. El-Sayyad, A.H. Ashour, A.I. El-Batel, M.A. Elsayed, M. Gobara, A.M. El-Khawaga, E.K. Abdel-Khalak, M.M. El-Okr, Antibacterial, antibiofilm, and photocatalytic activities of metals-substituted spinel cobalt ferrite nanoparticles. Microb. Pathog. 127, 144–158 (2019)

38. M.I.A. Abdel Maksoud, G.S. El-Sayyad, A.H. Ashour, A.I. El-Batel, M.S. Abd-Elmonem, H.A.M. Hendawy, E.K. Abdel-Khalak, S. Labib, E. Abdelwab, M.M. El-Okr, Synthesis and characterization of metals-substituted cobalt ferrite [Mx Co(1–x) Fe2O4; (M = Zn, Cu and Mn; x = 0 and 0.5)] nanoparticles as antimicrobial agents and sensors for Anagrelide determination in biological samples. Mater. Sci. Eng.: C 92, 644–656 (2018)

39. M.I.A.A. Maksoud, A. El-ghandour, G.S. El-Sayyad, A.S. Awed, R.A. Fahim, M.M. Atta, A.H. Ashour, A.I. El-Batel, M. Gobara, E.K. Abdel-Khalak, M.M. El-Okr, Tunable structures of copper substituted cobalt nanoferrites with prospective electrical and magnetic applications. J. Mater. Sci.: Mater. Electron. 30(5), 4908–4919 (2019)

40. M.I.A. Abdel Maksoud, A. El-ghandour, G.S. El-Sayyad, A.S. Awed, A.H. Ashour, A.I. El-Batel, M. Gobara, E.K. Abdel-Khalak, M.M. El-Okr, Incorporation of Mn2+ into cobalt ferrite via sol–gel method: insights on induced changes in the structural, thermal, dielectric, and magnetic properties. J. Sol-Gel Sci. Technol. 90(3), 631–642 (2019)

41. M.I.A. Abdel Maksoud, G.S. El-Sayyad, E. Fayad, A. Alyamani, O.A. Abu Ali, A.A. Elshamy, Gamma irradiation assisted the Sol-Gel method for silver modified-nickel molybdate nanoparticles synthesis: unveiling the antimicrobial, and antibiofilm activities against some pathogenic microbes. J. Inorg. Organomet. Polym. Mater. 32(2), 728–740 (2021)

42. S. Balasurya, A. Syed, L.L. Raju, S. Al-Rashed, A.M. Thomas, A. Das, S.S. Khan, Elucidation of photocatalysis, photoluminescence and antibacterial studies of Ag2MoO4 decorated NiMoO4 nanoheterostructure. Op. Mater. 113, 110856 (2021)

43. M. Kumar, R. Singh, H. Khajuria, H.N. Sheikh, Facile hydrothermal synthesis of nanocomposites of nitrogen doped graphene with metal molybdates (NG-MMoO 4)(M= Mn Co, and Ni) for enhanced photodegradation of methylene blue. J. Mater. Sci.: Mater. Electron. 28(13), 9423–9434 (2017)

44. B. Senthilkumar, K.V. Sankar, R.K. Selvan, M. Danielle, M. Manickam, Nano α-NiMoO 4 as a new electrode for electrochemical supercapacitors. RSC Adv. 3(2), 352–357 (2013)

45. J. Yesuraj, O. Padmaraj, S.A. Suthanthiraraj, Synthesis, Characterization, and Improvement of Supercapacitor Properties of NiMoO4 Nanocrystals with Polyanirole. J. Inorg. Organomet. Polym Mater. 30(2), 310–321 (2020)

46. S. Keerthana, B.J. Rani, R. Yuvakkumar, G. Ravi, B. Saravanakumar, M. Pannipara, A.G. Al-Sehemi, D. Velauthapillai, NiMoO4 nanorods photocatalytic activity comparison under UV and visible light. Environ. Res. 197, 111073 (2021)

47. A.V. Anupama, R. Kumar, H.K. Choudhury, V.J. Angadi, H.M. Somashekarakappa, B. Rudraswamy, B. Sahoo, Gamma-irradiation induced modifications in structural and magnetic properties of nanocrystalline Mn0.5Zn0.5SmxFe2-xO4 ceramics. Radiat. Phys. Chem. 166, 108506 (2020)

48. M.B. Rammal, S. Omanovic, Synthesis and characterization of NiO, MoO3, and NiMoO4 nanostuctures through a green, facile method and their potential use as electrocatalysts for water splitting. Mater. Chem. Phys. 255, 123570 (2020)

49. A.P. de Moura, L.H. de Oliveira, I.L.V. Rosa, C.S. Xavier, P.N. Lisboa-Filho, M.S. Li, F.A. La Porta, E. Longo, J.A. Varela, Structural, optical, and magnetic properties of NiMoO4 nanorods prepared by microwave sintering. ScientificWorldJournal 2015, 315084–315084 (2015)

50. J. Huang, Y. Xiong, Z. Peng, L. Chen, L. Wang, Y. Xu, L. Tan, K. Yuan, Y. Chen, A general electrodeposition strategy for fabricating ultrathin nickel cobalt phosphate nanosheets with ultrahigh capacity and rate performance. ACS Nano 14(10), 14201–14212 (2020)

51. B. Alshahrani, H.I. ElSaedery, S. Fares, A.H. Korna, H.A. Yako ut, M.I.A.A. Maksoud, R.A. Fahim, M. Gobara, A.H. Ashour, The effect of Ce3+ doping on structural, optical, ferromagnetic resonance, and magnetic properties of ZnFe2O4 nanoparticles. J. Mater. Sci.: Mater. Electron. 32(1), 780–797 (2021)
52. J. Tauc, R. Grigorovici, A. Vancu, Optical properties and electronic structure of amorphous germanium. Phys. Stat. Sol. (b) 15(2), 627–637 (1966)

53. H. El moussaoui, O. Mounkachi, R. Masrour, M. Hamedoun, E.K. Hhil, A. Benyoussef, Synthesis and super-paramagnetic properties of neodymium ferrites nanorods. J. Alloys Compd. 581, 776–781 (2013)

54. Q. Li, C.W. Kartikowati, S. Horie, T. Ogi, T. Iwaki, K. Okuyama, Correlation between particle size/domain structure and magnetic properties of highly crystalline Fe_3O_4 nanoparticles. Sci. Rep. 7(1), 1–7 (2017)

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