

Short communication

Flexible rGO/Cu/Ni/ZnO nanocomposites with enhanced optoelectronic, anticorrosive, photocatalytic, and magnetic properties

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ABSTRACT

Reduced graphene oxide (rGO) was prepared by improved Hummer's method and ternary metal oxide nanocomposites (NCs) of Copper, Nickel, and Zinc oxide (CNZ) were synthesized by modified Sol-Gel techniques. rGO and ternary CNZ NCs were grown together to get a flexible film. Their synergistic performance was investigated in terms of anticorrosive, photocatalytic, magnetic, and opto-electric properties. A deeper comprehension of the structural and optical characteristics of rGO/CNZ NCs was made by UV-visible, FTIR, p-XRD, SEM, and Raman techniques. The average particle size observed from the XRD and SEM techniques was 80 nm. The NCs demonstrated significant corrosion inhibition efficiency for mild steel with 99.01 % inhibition efficiency in 1.0 M H₂SO₄ at 100 ppm concentration. A magnetic susceptibility (χ) of 0.257 indicates that the NCs are paramagnetic. The electrical-band gap of NCs was 3.05 eV as observed from UV-vis. spectroscopy. The remarkable photocatalytic efficiency for degrading Methylene Blue dye in 70 min. demonstrates the good photocatalytic efficiency of the NCs. The rGO/CNZ NCs were flexible (bendable and rollable) showing their highly flexible nature, hence finding application in flexible electronic materials with anticorrosive, photocatalytic, and magnetic properties.

1. Introduction

In the world of flexible electronic devices, the synergistic performance of rGO and ternary metal oxides (TMO) gives a new path for multifunctional applications [1–3]. In heterogeneous catalysis, graphene-based materials stimulated the interest of researchers in this field [4–7]. They have a huge surface area, excellent electron mobility, thermal stability, and chemical flexibility. Graphene-based materials offer great flexibility in adjusting catalyst characteristics to specific applications. For instance, a large surface area promotes performance by aiding in the dispersion of nanomaterials [8–11]. It also serves as a perfect substrate for metal NPs because of its exceptional thermal stability under reducing conditions [12].

rGO is a two-dimensional (2D) emerging compound in material science and nanotechnology because it has chemical stability and excellent conductivity. Its sheets are not as electrically conductive as pure graphene, but their adaptability makes them extremely valuable [13]. Chemical reduction procedure removes extra functional groups from graphene, yielding graphene sheets of rGO [14].

rGO can be synthesized from the oxidation of graphene flakes to synthesize graphene oxide (GO) followed by a reduction process of GO to rGO which contains conjugated carbon atoms. One of the most common methods for the formation of GO is modified Hummer's method [15], then reduced to rGO by different reducing agents [16]. Different transition metal oxide [17–23], main group metal oxide [24] or metal sulfide NPs [25] either adsorb on surface or entangled in layers of graphene which has potential to show a variety of application such as in flexible electronic devices, catalysis, bio-medical, sensors, photocatalytic activity, magnetism, anticorrosive agents, antibacterial activity, etc [26–36].

Metal oxide (MO) NCs are an emerging area of research due to their tunable band gap and enhanced surface area. These NCs find many applications in the fields of water remediation, adsorption, biomedical, and electronics [37–40]. MO NPs are versatile and have promising properties like opto-electrical, magnetic, and photocatalytic properties. We selected CuO, ZnO, and NiO NPs in our study because CuO and ZnO NPs show antibacterial and photocatalytic properties whereas NiO shows catalytic and charge storing capacity [24,28,29,31,33,34].

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When rGO and transition metal oxide (TMO) NPs are combined into NCs then their synergistic effects are observed [41–44]. rGO provides large surface area to metal oxide NPs for adsorption and prevents their agglomeration. Proper dispersion of NPs takes place in rGO sheets. rGO sheets prevent the recombination of reactive oxygen species produced by metal NPs in the presence of sunlight or UV light. Thus together rGO and NPs enhance antibacterial and photocatalytic properties. This combination opens up new possibilities to enhance electrical, mechanical, magnetic, anticorrosive, photocatalytic performance, and other properties. The potential to create multifunctional NCs with better properties than their constituent parts is made possible by the supplementary properties of these components.

Because of their superior electrical conductivity and flexibility, rGO/TMO NCs seem especially suitable for application in energy storage devices. Because of their greater conductivity, which enhances charge storage and retrieval, and mechanical reliability, which provides strength, these rGO-based NCs acts as a promising materials for improved electrode materials for rechargeable batteries and supercapacitors. Different rGO-based NCs have applications in energy storage devices, especially batteries, supercapacitors are explored by the scientific community. For instance, Hao Jiang *et al.* reported high-rate supercapacitors from a nanostructured composite of rGO/CNTs/MnO₂ [45]. Zulqar Nain Ahmed *et al.* reported binderless electrode of rGO/Fe₂O₃/PolyIndole nanocomposite for supercapacitor application [46]. Ning Yi *et al.* synthesized a highly sensitive, stretchable, and low-temperature NO₂ gas sensor based on MoS₂@rGO NCs [47].

Donghui Li *et al.* fabricated an NH₃ sensor, GaN/rGO NCs [48]. Love Dashairya *et al.* reported SnS₂/rGO NCs for the enhanced photocatalytic degradation of toxic dyes produced from industries under sunlight [8]. Yahya Sandali *et al.* synthesized WO₃/BiS₂/rGO composite for photodetector applications with improved opto-electronic performance [49]. Movlud Valian *et al.* reported that the electrochemical hydrogen storage could be significantly improved by using novel Li_{0.35}La_{0.55}TiO₃/rGO NCs [50]. Sharma *et al.* have investigated rGO@Al₂O₃/CuO/TiO₂ NCs as photocatalytic agents in UV light for degrading methylene blue dye with 94 % degrading efficiency [51]. Kumar *et al.* have reported different metal oxide NPs as electrochemical food sensors for detecting different preservatives, additives, and coloring agents in food samples [52]. Sharma *et al.* have investigated rGO@Al₂O₃/ZnO/TiO₂ NCs as impure water purification and environmental remediation agent [53]. Sharma *et al.* have investigated rGO@Al₂O₃/Mn₃O₄/Fe₂O₃ NCs as a multifunctional nanomaterials [54].

Karami *et al.*, synthesized CaSiO₃ nanostructure and enhanced photocatalytic performance for water treatment. The catalyst could efficiently degrade acid red, methyl orange, and thymol blue under visible light [55]. Yousefzadeh *et al.*, produced Sm₂CuO₄ nanophotocatalyst effectively decolorated organic pollutants (methylene blue dye) [56]. Hosseini *et al.*, reported Ni₂SiO₄ (NSO) nanostructures for the first time and achieved 91.8 % degradation of Eriochrome black T [57]. Seyed *et al.*, fabricated Dy₂Cu₂O₅ nanostructures for photocatalytic activity investigated through different organic coloring agents [58].

Most of the research is based on exploring single or binary MO with rGO-based NCs with one or two applications, but very few studies are based on the NCs of rGO/TMO. First time, we are reporting, the synergistic performance of the synthesized TMO NCs of Copper, Nickel, and Zinc oxide with rGO (rGO/CNZ). We have reported different properties like flexibility, optical, magnetic, photocatalytic, and anti-corrosive, followed by multiple applications. The NCs act as versatile, flexible, and conductive nanomaterials with promising multifunctional applications in magnetism, optoelectronics, corrosion inhibition, and photocatalytic activity for dye degradation. Photocatalytic properties of CuO and ZnO NPs are combined with the optoelectronic properties of NiO NPs. rGO acts as a supporting material for TMO NPs and increases the electrical properties of nanomaterials by reducing the optical band gap and helping in the separation of electrons and holes.

2. Experimental

AR-grade graphite flakes, Min. assay ≥99 % (Sigma-Aldrich), Mol. wt. 12.01, C. No. 7782-42-5. Zinc Oxide, Min. assay 99.0 % (CDH) Mol. wt. 81.38, C. No. 1314-13-2. Oxalic Acid, Min. assay 99.5 % (CDH) Mol. wt. 126.07, C. No. 6153-56-6. Hydrochloric acid, Min. assay 37.0 % (CDH) Mol. wt. 36.46, C. No. 7647-01-0. Nitric acid, Min. assay 70.0 % (Sigma-Aldrich) Mol. wt. 63.01, C. No. 7697-37-2. Ethanol 99.9 % (CSS). Sulphuric Acid, Min. assay of ≥98 % (CDH), Mol. Wt. 98.08, C. No. 7664-93-9. Sodium Hydroxide Pellets, Mol. wt. 40.00, C. No. 1310-73-2. Zn(NO₃)₂ Min. assay 98.0 % (Sigma-Aldrich) Mol. Wt. 297.49, C. No. 10196-18-6, Cu(NO₃)₂, Min. assay 99.0 %, M. Wt. 241.6. KMnO₄, NiNO₃, and all the above used chemicals were of AR Grade. The anti-corrosive study, on Mild Steel (MS) in the 1.0 M H₂SO₄ acidic medium, of rGO/ZNC NCs was investigated by wt. loss technique (ASTM-D2688). Distilled water (DW) was utilized for preparing 1.0 M sulfuric acid as a corroding medium. The photocatalytic activity is performed under UV light using methylene red dye (MRD). Magnetic properties of synthesized NCs were observed using Gouy's balance.

2.1. Synthesis of CNZ MO NPs using Sol-gel approach

Sol A was prepared from CuNO₃, NiNO₃, and ZnNO₃ in an acidic medium by maintaining a pH of 1–2 using dilute HNO₃. Then in a dropwise manner, Sol B, which is composed of ethanol (CH₃CH₂OH) and oxalic acid (C₂H₂O₄) in a ratio of 5:1, was added to Sol A, with a continuous stirring (650 rpm) at 70 °C for 6 h, until the precipitates (light blue colored) were formed. Washing of the precipitates was carried by two solvents, i.e., distilled water (DW) and acetone. Finally, the ppt. formed were dried overnight at 70 °C and then stored.

2.2. Synthesis of rGO

rGO was synthesized using an improved Hummer's method, in which, graphite powder is mixed with KMnO₄ and then pure H₂SO₄ is gradually mixed with the H₃PO₄ under a controlled temperature for the oxidation process. To quench the process and reduce the manganese compounds DW and H₂O₂ were added. The solid component was kept undisturbed for a day which was further centrifuged and purified by using HCl to remove the metal ions. Washing was carried out with ethyl alcohol and then with distilled water. Thus a viscous solution of rGO was prepared.

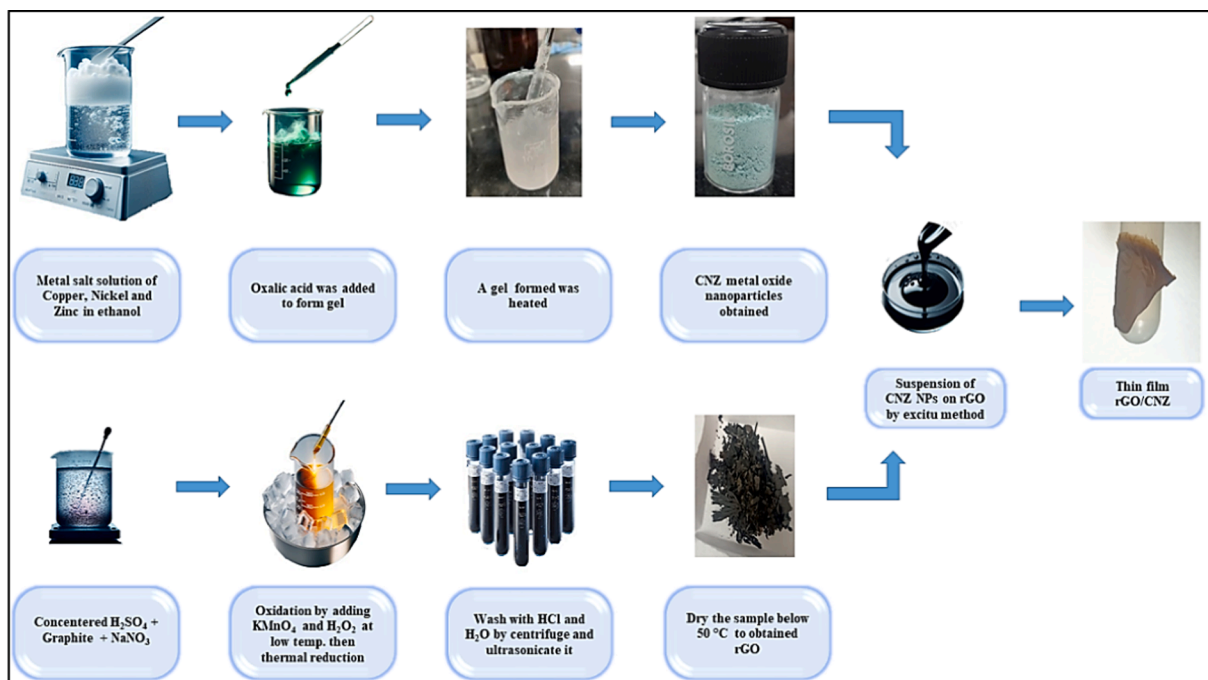
2.3. Loading of CNZ NPs in rGO matrix by ex-situ method

For the loading of ternary CNZ NPs in the rGO matrix, the *ex-situ* method was adopted as shown in Scheme 1. A suspension of 0.050 g of CNZ NPs was added to the dispersion of rGO. The dispersion containing suspension of NPs was heated to 70 °C and ultrasonicated for 30 min. Thereafter, the suspension was kept in the petri dish and was dried at 55 °C. After 24 h of drying, rGO/CNZ NCs were obtained as a flexible film.

3. Results and discussion

3.1. UV-visible investigation

The absorption spectrum of rGO/CNZ NCs was observed using UV-visible spectroscopic technique and shown in Fig. 1. The MO NPs of Cu, Ni, and ZnO were found to have the maximum absorbance at 205 nm, which is slightly shifted towards a shorter wavelength in the rGO/CNZ NCs showing blue shift which satisfy the chemistry of synthesized NCs. An absorption band at 260 nm was associated with π-π* transition because of the double bond present in hexagonal rings of rGO.



Scheme 1. Schematic representation for the synthesis of metal oxide NPs by Sol-gel method and rGO/CNZ thin film by *ex-situ* method.

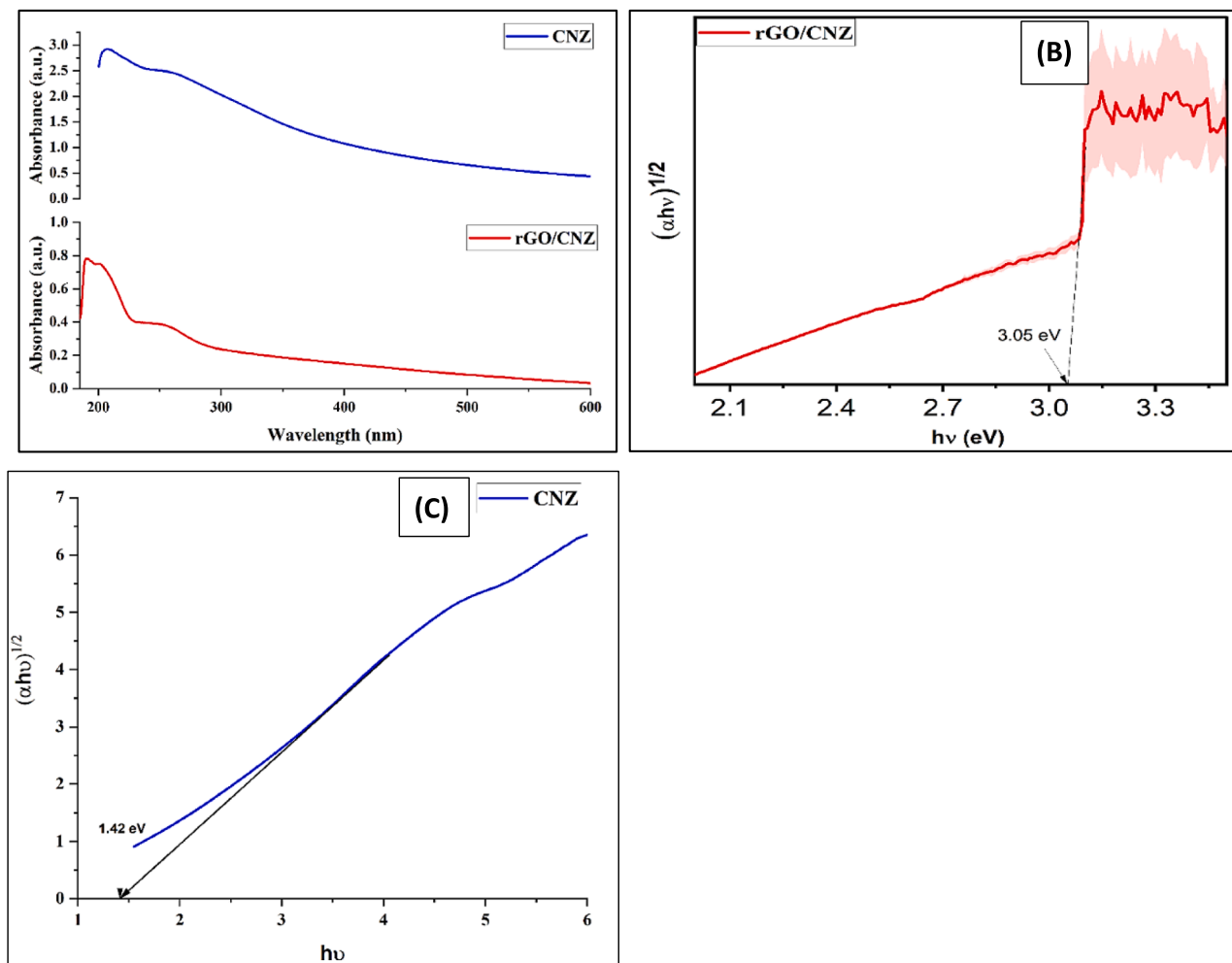


Fig. 1. UV spectra of CNZ NCs (blue) and rGO/CNZ NCs (red) [A], Tauc plot for rGO/CNZ NCs (B) and CNZ NCs (C) for direct band gap.

3.2. FT-IR spectra of rGO/CNZ NCs

FT-IR technique was utilized to determine skeleton and functional peaks in rGO/CNZ NCs. The FT-IR spectra of CNZ NCs and rGO/CNZ NCs are shown in Fig. 2. The rGO/CNZ NCs' FT-IR spectra revealed large and wide absorption peaks at 3394 cm^{-1} which was associated with -OH stretching modes present in rGO. Also, the absorption peak at 1619 cm^{-1} is associated with the peak of the carbonyl groups of COOH present in rGO/CNZ NCs. The absorption peaks between $1600\text{--}1400\text{ cm}^{-1}$ were observed due to the C–O stretching vibrations. The peaks at 819 , 594 , and 430 cm^{-1} were observed as corresponding peaks of the Cu–O, Ni–O, and Zn–O which confirmed the MO NPs were entangled between rGO layers.

3.3. Raman spectroscopic study

Fig. 3 depicts the Raman spectrum for rGO/CNZ NCs. Two strong peaks in the range $1200\text{--}1350\text{ cm}^{-1}$ and $1580\text{--}1600\text{ cm}^{-1}$ represent D and G bands due to rGO. Both D and G bands are common in rGO which provide information about structural integrity and reduction of graphitic oxide to rGO. A medium peak at 2750 cm^{-1} corresponds to the 2nd-order 2D band of rGO. The 2D band is the overtone of the D band which gives information about the thickness and stacking of layers in rGO. 2nd order overtone of the D band (2750 cm^{-1}) provides insights into the stacking and thickness of rGO layers. Many small intensity peaks in the region of $1700\text{--}3500\text{ cm}^{-1}$ correspond to metal oxide components (CuO, NiO, and ZnO) in the rGO matrix. Hydroxyl groups (OH) and carboxyl functional groups present at the surface of rGO also show bands in this region.

3.4. SEM study

The synthesized rGO/CNZ NCs were examined for surface morphology by SEM technique. SEM images of CNZ NCs are shown in Fig. 4(a), whereas the images (Fig. 4b to f) are of rGO/CNZ NCs at different resolutions. NCs had an average particle size of 115 nm . The SEM pictures show that the NCs, which are made of rGO and CNZ NCs

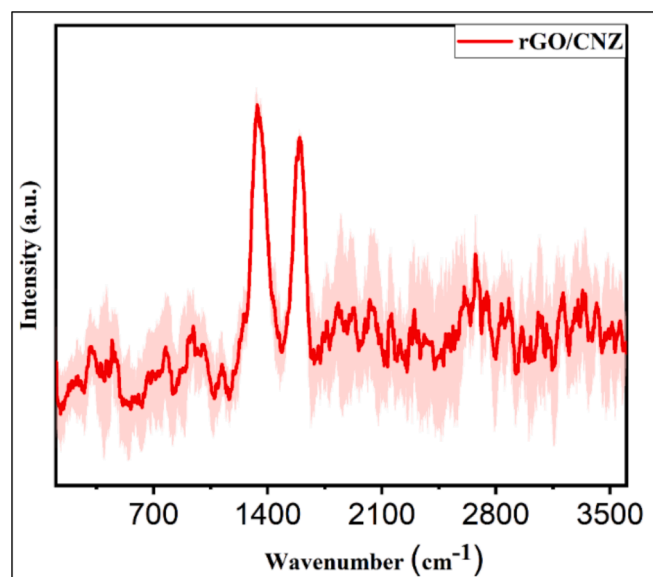


Fig. 3. Raman spectra for rGO/CNZ NCs.

film, had an amorphous surface and an overall sheet-like structure. In the flexible layers of rGO, CNZ NCs were perfectly settled over, which increases the mechanical strength of layers and the conductivity of the film.

3.5. X-ray diffraction study

XRD is a very useful technique to understand the structural properties of composites. In the case of rGO with Cu, Ni, and ZnO NPs, XRD analysis helps in identifying the phase and crystallinity of the rGO and the NPs, as well as revealing any interactions between them. Fig. 5 shows the X-ray diffraction pattern for rGO/CNZ NCs. A strong peak at 12° (002) is due to the reflection of XRD rays from large inter-layer spacing in the rGO layers. In pristine graphite, this characteristic

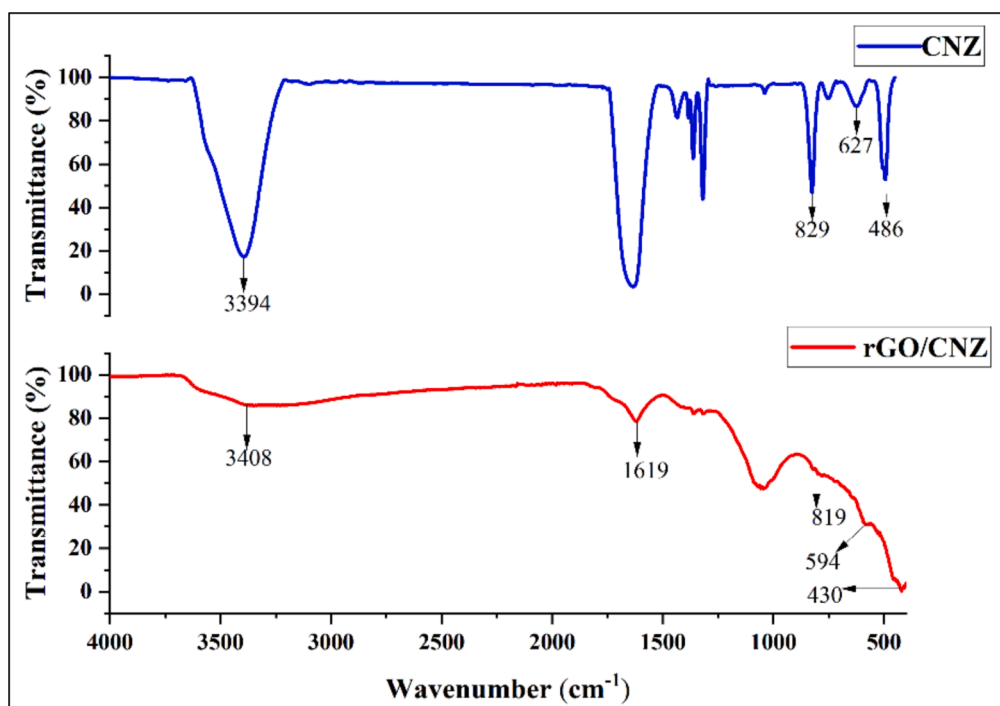


Fig. 2. FTIR Spectra of CNZ NCs (blue) and rGO/CNZ NCs (red).

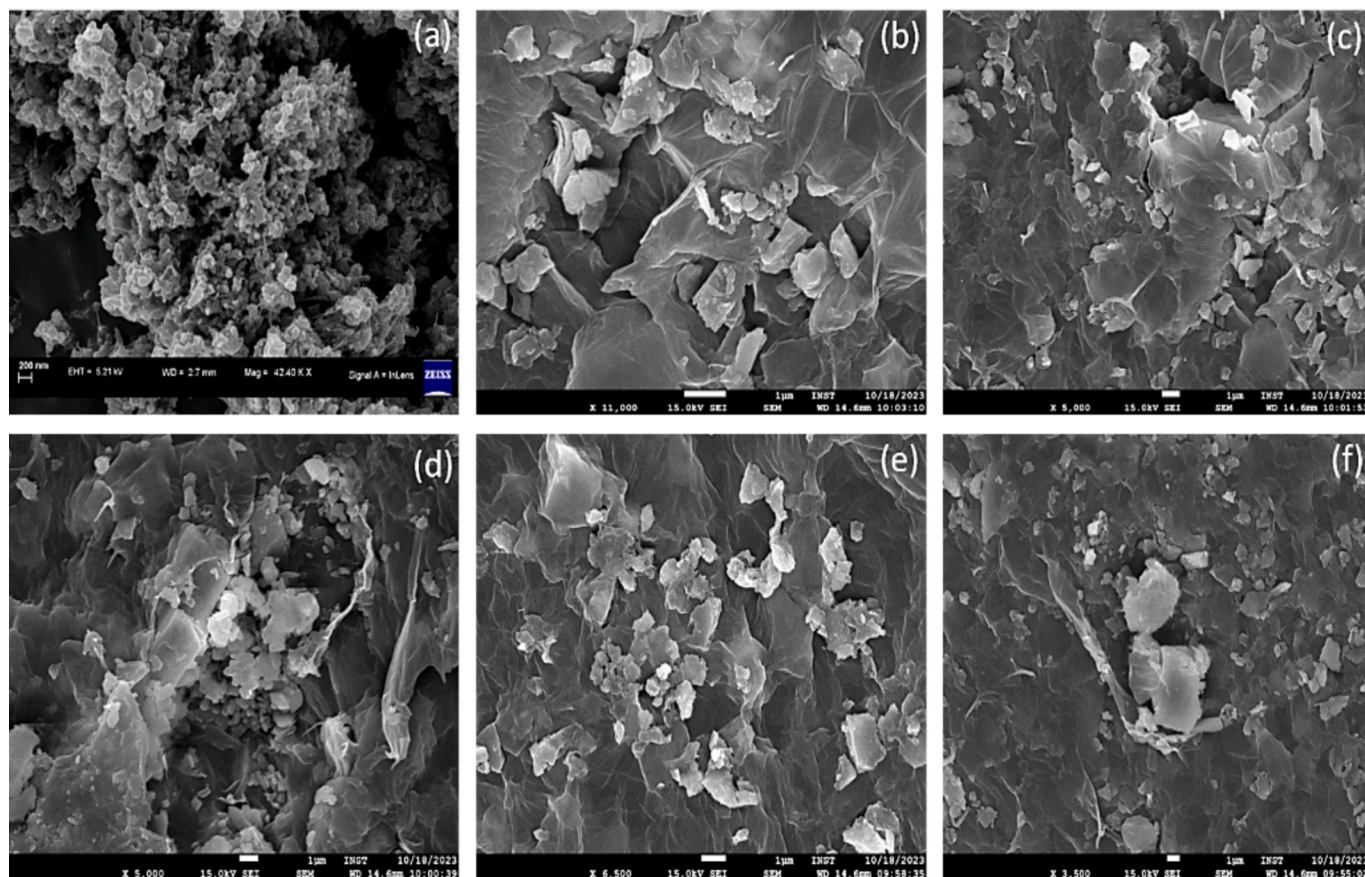


Fig. 4. SEM images of CNZ NCs (a) and rGO/CNZ NCs (b-f).

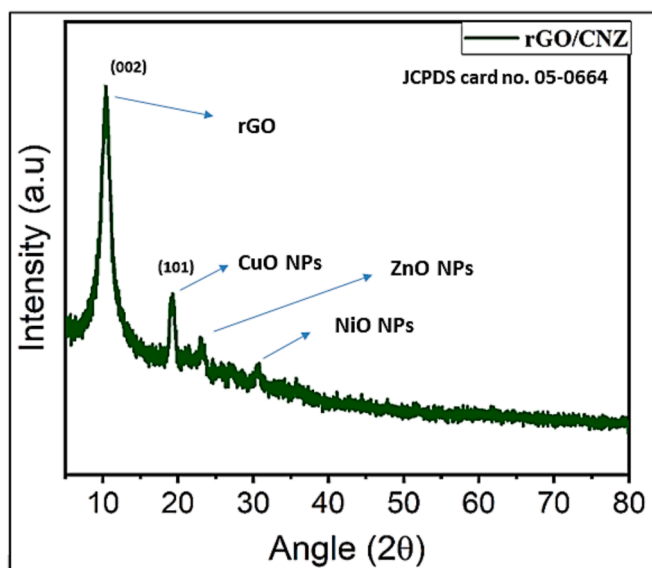


Fig. 5. X-ray diffraction pattern for rGO/CNZ NCs.

peak appears at 26 degrees, however, in rGO these peaks shift to a lower angle that is 10–12 degrees indicating less crystalline character in rGO.

The peak at around 12° corresponds to the (002) reflection of rGO. This peak is characteristic of the rGO structure, and it is shifted to a lower angle compared to the graphite (26° peak). In pristine graphite, the interlayer spacing between graphene sheets is very small, leading to the (002) peak at ~26°. However, in rGO, the reduction of oxygen-

containing groups (such as epoxides, hydroxyls, and carboxyls) causes an increase in the interlayer spacing. This results in a shift of the (002) peak to a lower angle (around 12°), indicating a less crystalline structure and increased interlayer spacing. The increased interlayer spacing in rGO provides a better environment for the intercalation or incorporation of metal NPs. This shift suggests that the reduction of GO to rGO has created more accessible sites for the metal NPs to interact with and potentially be intercalated into the rGO sheets.

A medium peak at 18 degrees (101) corresponds to an additional rGO staking configuration. It indicates the intercalation of metal oxide NPs in rGO layers. The peak at 18° is assigned to the (101) plane of rGO, corresponding to a stacking configuration within the rGO sheets. This peak suggests that metal oxide NPs (Cu, Ni, ZnO) have been incorporated into the rGO layers. The shift and appearance of this peak can indicate changes in the stacking arrangement of rGO layers upon intercalation of the metal NPs. The appearance of this peak and the associated intercalation of metal oxide NPs indicates that the metal oxide NPs are likely filling the interlayer space between rGO sheets. This suggests a strong interaction between the rGO and the metal oxide NPs, which may enhance properties like electrical conductivity, stability, or catalytic activity.

Very small intensity peaks at 22 degrees (110) may be due to ZnO or CuO (Tenorite) NPs. The Wurtzite structure of ZnO NPs shows a peak of 22 degrees. The peak at around 22° is typically observed for ZnO NPs, which crystallize in the Wurtzite structure. This peak may also appear for CuO (Tenorite) NPs. ZnO nanoparticles crystallize in the Wurtzite structure, and the characteristic XRD peak of ZnO is typically at around $2\theta = 22^\circ$ (for the (110) reflection). CuO NPs, also known as Tenorite, can show peaks around 22°, corresponding to the (110) reflection of the monoclinic structure of CuO. The appearance of this peak in the XRD pattern indicates that ZnO or CuO NPs are indeed present in the NCs. It

also suggests that these metal oxide NPs are well-dispersed on the rGO sheets or may even be incorporated within the rGO layers. The interaction between the rGO and these metal oxide NPs can lead to increased catalytic or electrochemical properties of the NCs.

A peak at 30 degrees corresponds to the Bunsenite structure of NiO NPs. The presence of this peak indicates that NiO NPs are incorporated into the rGO matrix. The interaction between NiO NPs and rGO could involve the adsorption of NiO on the surface of rGO or even intercalation between the rGO layers. The interaction of NiO with rGO may enhance the performance of the NCs in applications like energy storage or sensors. The average particle size range as observed from the Scherer equation was 80 nm. Table 1 depicts p-XRD data of rGO/Cu/Ni/ZnO NCs.

4. Applications study of rGO/CNZ NCs

4.1. Anticorrosive nature of rGO/CNZ NCs

By using the weight loss technique, the anticorrosive properties of the synthesized rGO/CNZ NCs were examined against MS in 1.0 M H₂SO₄. This approach involved exposing MS strips in 1.0 M H₂SO₄ solution for 6.0 h at different conc. of NCs (Table 2). The CIE and CR were calculated based on the weight loss of the strips [59]. An increase in weight loss of the strips and corrosion rate was observed along with a decrease in CIE as the NC conc. was raised. Fig. 6 illustrates that as the conc. of rGO/CNZ NCs was increased, the CIE was decreased. Consequently, for MS in the acidic medium (1.0 M H₂SO₄), the synthesized rGO/CNZ NCs were found as a good anticorrosive nanomaterial at a lower conc. of 10 ppm. Table 3 depicts a comparative study of CIE shown by different green and chemical corrosion inhibitors with our rGO/CNZ NCs [63–75]. It was observed that the CIE shown by our NCs was comparable to most of the corrosion inhibitors reported in the literature.

Mechanism of action of rGO/CNZ NCs as corrosion inhibitor

Fig. 11 depicts the corrosion inhibition protection mechanism for MS in 1.0 M H₂SO₄ by the rGO/CNZ NCs. The corrosion resistance of the rGO/CNZ NCs was due to several mechanisms that involve both the physical properties of rGO and the chemical properties of the metal oxide NPs. The corrosion study data shows how the rGO/CNZ NCs effectively inhibit corrosion of MS in acidic media (1.0 M H₂SO₄) and how its anticorrosive performance changes with concentration. The rGO, when used in composites, forms a protective layer on the MS surface. This barrier layer prevents direct contact between the MS and the aggressive acidic medium (H₂SO₄). rGO has excellent adhesion properties and is known to form a highly stable, dense network, which acts as a barrier, reducing the penetration of aggressive ions like H⁺ from the acidic solution. This reduces the likelihood of corrosion occurring at the MS surface.

ZnO, CuO, and NiO NPs are known to adsorb metal cations and form complexes with H⁺ or other corrosive species in the solution. This helps neutralize the acidic environment near the metal surface, thereby reducing the rate of corrosion. The metal oxide NPs can accept or donate electrons, modifying the electrochemical behavior at the MS surface. This shifts the corrosion potential and inhibits the electrochemical processes responsible for corrosion. ZnO, CuO, and NiO can form protective oxide layers on the metal surface, which act as a physical and electrochemical barrier that prevents the further attack of the metal by the acidic medium. This is especially important in acidic environments,

Table 1
p-XRD data of rGO/Cu/Ni/ZnO NCs.

2θ degree	d-spacing (Å)	Intensity	Indexing	Phase
12.0	7.37	Strong	rGO (002)	Amorphous
18.0	4.92	Medium	rGO (101)	Amorphous
22.0	4.04	Weak	ZnO (110)/CuO	Tenorite
30.0	2.97	Medium	NiO	Bunsenite

Table 2

The weight loss, corrosion rate (CR), and corrosion inhibition efficiency (CIE %) determined through the weight loss technique across different concentrations of NCs.

Concentration (ppm) of rGO/CNZ NCs	Wt. loss (mg)	Corrosion rate (mpy)	Inhibition efficiency (%)
10	11.4	185.44	99.01
20	20.3	330.20	98.24
50	49.5	805.18	95.71
70	56.6	920.67	95.09
100	75.8	1232.98	93.43
Blank	1153.3	18759.87	–

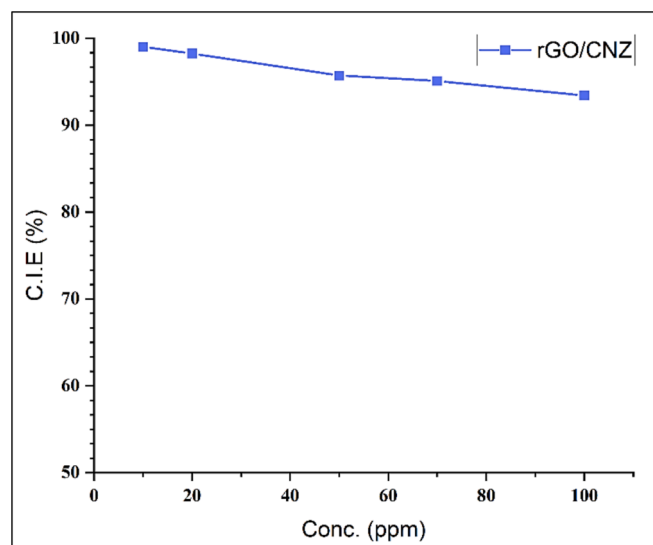


Fig. 6. Variation in CIE shown by rGO/CNZ NCs w.r.t. concentrations at room temperature.

showing a high corrosion rate. The metal oxide NPs (ZnO, CuO, NiO) are well dispersed on the rGO surface. rGO provides high specific surface (SSA) to the NCs. High SSA allows better adsorption of the NCs on the MS surface thus improving its corrosion inhibition efficiency. Hence, rGO and CNZ NPs act synergistically with each other and help in improving CIE. These NPs can form strong interactions with the rGO sheets, which improves adhesion to the MS surface.

4.2. Photocatalytic activity of rGO/CNZ NCs

Using a pH meter, the photocatalytic properties of rGO/CNZ NCs were studied on methylene blue dye (MBD). We subjected MBD to rGO/CNZ NCs of 20 ppm concentration and exposed it to visible light for the degrading MBD. A gradual fall in the pH indicates the degradation of dye into mineral acids. Fig. 7 depicts the pH of the nanocomposite/dye solution at the interval of every 10.0 min. The pH of the solution decreases continuously for 70 min confirming the degradation of MBD into mineral acids by the synthesized rGO/CNZ NCs (Fig. 7).

Fig. 8 depicts the absorbance of the NCs/dye solution at different intervals of time by using a UV-vis. spectrophotometer. The absorbance of the solution decreases which indicates that the color of the dye disappears as the dye degrades. Thus the synthesized rGO/CNZ NCs showed photocatalytic activity under visible light and hence can be used as a waste water remediation process.

4.2.1. Mechanism for photocatalytic degradation of MBD

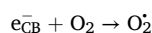
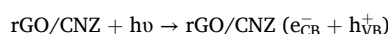


Table 3

A comparative study of corrosion inhibition efficiency shown by different green, chemical, and NCS-based corrosion inhibitors.

S. No.	Types of inhibitor	Corroding medium	Nature of metal	CIE (%)	References
1.	Chalcone + KI derivatives	1.0 M HCl	Mild steel	85.4	[63]
2.	Epoxy modified Cellulose/ Polyaniline/ternary metal oxide NCS	1.0 M H ₂ SO ₄	Mild steel	83.9	[64]
3.	Sulfate-functionalized ternary metal oxide/ polypyrrole NCS	1.0 M HCl	Mild steel	85	[65]
4.	Pyrazole Schiff bases	1.0 M HCl	Mild steel	94.6	[66]
5.	Petals of <i>B. glabra</i> as a green corrosion inhibitor	1.0 M H ₂ SO ₄	Mild steel	93	[67]
6.	Polyvinyl caprolactam	H ₂ S and CO ₂ media	Mild steel	96.9	[68]
7.	<i>Azadirachta indica</i> extract as a green corrosion inhibitor	5.0 M HCl	Mild steel	89.2	[69]
8.	<i>Morus nigra</i> as a green corrosion inhibitor	0.5 M HCl	Mild steel	91.6	[70]
9.	Polyvinyl pyrrolidone	1.0 M HCl	Mild steel	85.9	[71]
10.	Ammonium oxalate	0.5 M HCl	Mild steel	94.9	[72]
11.	N,N'-dimethylaniline (DMA)	2.0 N HCl	Carbon steel	84.0	[73]
12.	<i>Allium-sativum L.</i> as a green corrosion inhibitor	5.0 M HCl	Mild steel	94.7	[74]
13.	Acyclic and cyclic hydrocarbons	2.0 N HCl	Carbon steel	85.13	[75]

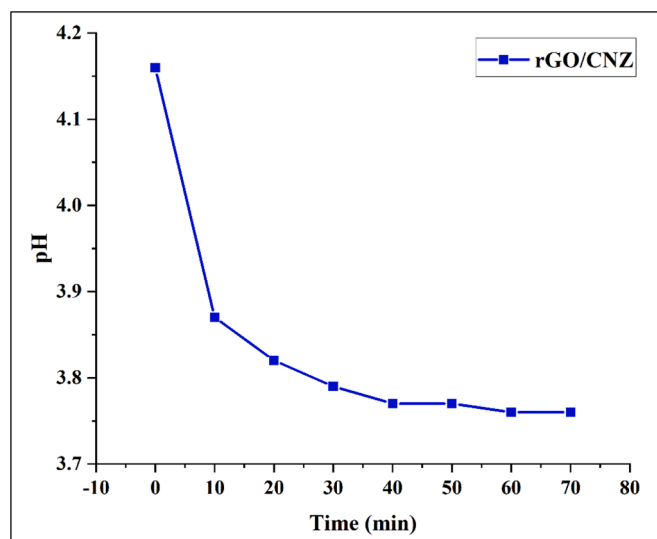
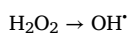
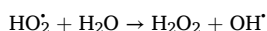
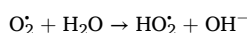


Fig. 7. pH variation shown by MBD solution containing rGO/CNZ NCs.



In this mechanism, the NCs produce electron-hole pairs by absorbing

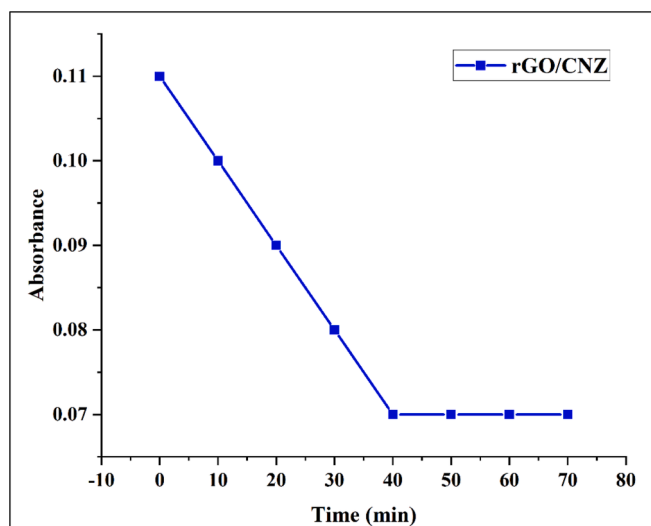


Fig. 8. Absorbance shown by rGO/CNZ NCs at different intervals of time.

light in the ultraviolet spectrum (Fig. 9). To create the reactive species O_2^- and $\text{OH}^\cdot$, these photo-generated e-h pairs, respectively, take part in the redox reactions of the H_2O molecule. The MBD is broken down by these reactive species into mineral acids CO_2 and H_2O [60]. Cu/Ni/ZnO NCs photocatalytic efficiency is enhanced as more reactive oxygen species (ROS) are generated on a higher surface area provided by the rGO sheet. Thus, rGO and CNZ NPs act synergistically with each other in increasing photocatalytic efficiency.

4.3. Magnetic property by rGO/CNZ NCs

Magnetic susceptibility (χ) was measured to ascertain the magnetic behavior of rGO/CNZ NCs. Gouy's balance technique was utilized to calculate the magnetic susceptibility. The weight of rGO/CNZ NCs varies with the applied magnetic field (H^2), as seen in Fig. 10. As the strength of H increased, the weight of rGO/CNZ NCs decreased. This demonstrates unequivocally that rGO/CNZ NCs were paramagnetic. Additionally support to this conclusion was the magnetic susceptibility (χ) measured by Eq. (1).

$$\chi = \frac{2g \times \Delta m}{A(\Delta H^2)} \quad (1)$$

The magnetic susceptibility of the synthesized NCs ($\chi = 0.257$) showed that the synthesized rGO/CNZ NCs are paramagnetic. Consequently, USB flash drives, super magnets, or magnetic recording tapes may be made from rGO/CNZ NCs.

4.4. Optical study of rGO/CNZ NCs

The combination of NiO, CuO, ZnO, and rGO in the form of rGO/metal oxide NCs has a significant impact on the electronic band structure, and this can be understood through several key concepts, including band gap modifications, electronic transitions, and conductivity changes.

The indirect band gap between the molecular orbitals of the HOMO and LUMO of rGO/CNZ NCs was calculated by utilizing the Tauc relation given in Eqn. (2) [41,61,65]

$$\alpha h\nu = B(h\nu - E_g)^n \quad (2)$$

The coefficient of absorption (α) was estimated by the Lambert's-Beer relation [62].

The Tauc relation, used to estimate the optical band gap (E_g), based on the absorption coefficient (α) and photon energy ($h\nu$). The plot of $(\alpha h\nu)^{1/2}$ vs. $h\nu$ is used to determine the indirect band gap, which is the

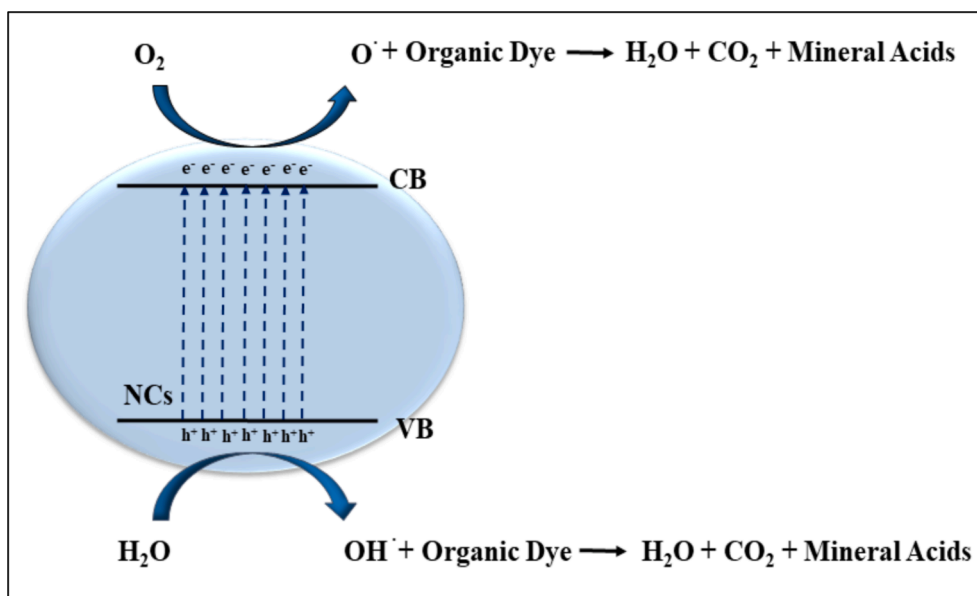


Fig. 9. Photocatalytic degradation mechanism of MBD.

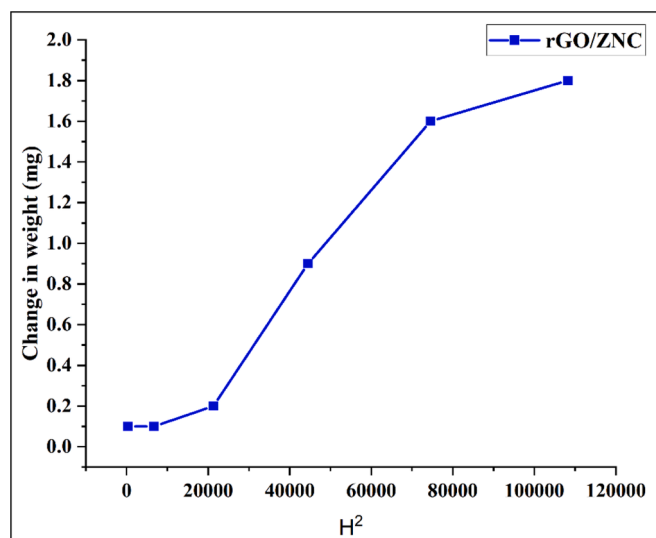


Fig. 10. Magnetic study of rGO/CNZ NCs by Gouy's balance method.

energy difference between the VB and the CB of the material.

A plot between $(ah\nu)^{1/2}$ and $h\nu$ was drawn (Fig. 1B and C), to measure the indirect band gap of rGO/CNZ NCs. The energy band gap (E_g) of rGO/CNZ NCs was found to be 3.05 eV, which is attributed to π - π^* transition to CB from VB, while for CNZ NCs this was 1.42 eV. That indicates the addition of CNZ NCs to rGO, E_g increases from 1.42 to 3.05 eV. Consequently, with the addition of CNZ NCs, the conductivity of rGO/CNZ NCs-based film was slightly decreased.

The band gap of rGO/CNZ NCs (where CNZ likely refers to the metal oxide composite of NiO, CuO, and ZnO) was calculated to be 3.05 eV, which was due to the π - π^* transition from the VB to the CB. This is typical for materials that have a semiconductor nature, where electrons in the VB are excited to the CB. The band gap of the pure CNZ NCs (metal oxides such as NiO, CuO, ZnO) was found to be 1.42 eV. This indicates a much smaller band gap compared to the rGO-based composite, suggesting that the metal oxide components have a narrower band gap and may exhibit more metallic or semi-conducting behavior.

The band gap of the rGO/CNZ NCs being higher (3.05 eV) than the

metal oxides (1.42 eV) suggests that the incorporation of rGO (which has a reduced band gap due to its conductive nature) leads to an increase in the overall band gap of the composite. The interaction between rGO and metal oxide NPs alters the electronic structure, likely resulting in the formation of new electronic states, or possibly a type of hybridization between the rGO π -bands and the metal oxide CB.

Each metal oxide NPs has a unique electronic structure that influences the overall properties of the rGO/metal oxide composite. For example, NiO typically exhibits p-type conductivity with a wide band gap (around 3.6 eV). CuO has a band gap of about 1.2–2.5 eV, showing mixed p-type and n-type behavior. ZnO has a wide band gap (~3.37 eV) and is a typical n-type semiconductor. When these metal oxide NPs are combined with rGO, heterojunctions can form at the interfaces between the rGO and the metal oxide. This could lead to an alignment of energy levels that modifies the electronic structure of the composite. The metal oxides could influence the electronic properties by either donating or accepting charge carriers, which affects the charge transport properties of the NCs.

The data mentions that the conductivity of rGO/CNZ NCs is slightly decreased compared to pure rGO or the metal oxide NPs alone. This decrease in conductivity is likely due to the increase in the band gap from 1.42 eV to 3.05 eV. A larger band gap typically implies that fewer charge carriers are available for conduction at a given temperature, leading to a decrease in the material's overall conductivity. This is consistent with the data, where the conductivity of rGO/CNZ NCs is slightly reduced despite the presence of rGO (which is a good conductor). The probable reason for the decrease in optical band gap on the addition of rGO in NCs may be due to the quantum confinement effect. The addition of rGO in CNZ NCs leads to a reduction in the particle size of metal oxide NCs. This leads to an increase in the optical band gap due to the small size of NPs and exhibits a widening of the band structure due to the confinement of charge carriers. The other probable reason for an increase in the optical band gap is due to charge transfer between rGO and CNZ-based NCs leading to modification in the band structure.

π^* transition from the VB to the CB in the rGO/CNZ NCs implies that the composite material undergoes electronic excitations similar to those observed in pure graphene-based materials. However, due to the presence of metal oxide NPs, these transitions may be modified by interactions with the metal oxide components, such as charge transfer at the interfaces or electronic state modifications.

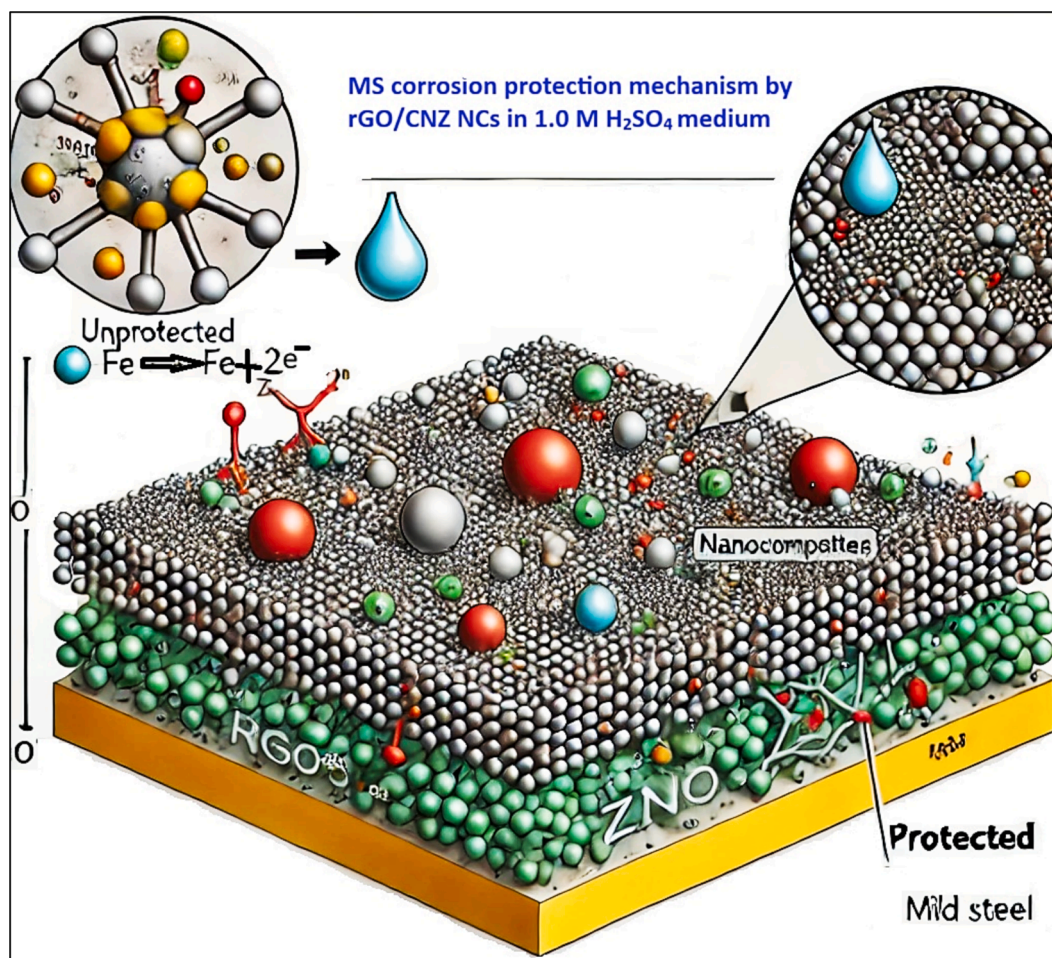


Fig. 11. Corrosion inhibition protection mechanism for MS in 1.0 M H₂SO₄ by the rGO/CNZ NCs.

4.5. Flexibility and durability of rGO/CNZ NCs

The flexibility and durability of rGO/CNZ NCs thin film were checked by rolling it on a glass tube and glass rod. It was found that the film did not break on rolling both on the glass tube and glass rod. However, due to the opaque color of the film the solution filled in a glass tube was not visible through the film.

Table 4 depicts a comparative study of anticorrosive, magnetic, optical, photocatalytic, and flexibility studies of rGO/CNZ NCs with reported literature [76–79]. Anticorrosive, magnetic, and electrical studies are better than reported in the literature.

5. Conclusions

The potential of rGO/CNZ NCs as adaptable materials was explored with multiple applications. The loading of CNZ NPs in the rGO matrix was done by the *ex-situ* method. The NCs demonstrated significant CIE i. e., 99.01 % for MS in 1.0 M H₂SO₄. A magnetic susceptibility ($\chi = 0.257$)

indicates that the NCs fall within the paramagnetic material range, suggesting their suitability for magnetic applications. The NCs' direct optical band gap i.e., 3.05 eV proved their suitability in semiconductor devices and will act as a novel material of nanomaterial science with multifunctional applications. The remarkable photocatalytic activity for degrading methylene blue dye in 70 min demonstrated its promising role in environmental remediation and wastewater pollution. The synthesized rGO/CNZ NCs were flexible and rollable and, hence can be used in flexible electronic materials. The rGO/CNZ NCs serve as a multifunctional material by providing a protective covering to material, photo-catalytic agent in environmental protection, paramagnetic, and flexible optoelectronic applications. Optimization techniques to improve their performance and customize them for certain specific applications can be explored in more detail in the future.

CRediT authorship contribution statement

Gaman Kumar: Writing – original draft, Methodology,

Table 4

Comparative study of anticorrosive, magnetic, optical, photocatalytic, and flexibility study of rGO/CNZ NCs with reported literature [76–79].

Nanocomposites	Anticorrosive	Magnetic (χ)	Optical band gap (eV)	Photocatalytic	Flexibility	References
ZnO/rGO	NR	NR	NR	98.9 %	NR	[76]
CuFe ₂ O ₄ /Bi ₂ O ₃	NR	NR	1.62	91 %	NR	[77]
D-CNFO@G	NR	NR	2.75	71.8 %	NR	[78]
Al ₂ O ₃ -ZnO-TiO ₂ @rGO	54.51 %	0.0204	2.46	82.2 %	NR	[41]
Al ₂ O ₃ -CuO-ZnO@rGO	95.9 %	0.0941	2.653	89.21 %	NR	[79]
rGO/CNZ	99.01 %	0.257	3.05	57.14 %	Flexible	Present Work

Investigation. **Harish Kumar**: Writing – review & editing, Validation, Supervision, Investigation, Conceptualization. **Rajni Kumari**: Visualization, Validation. **Aarti Tundwal**: Visualization, Validation. **Ankit Dhayal**: Visualization, Validation. **Abhiruchi Yadav**: Visualization, Validation, Investigation. **Rahul Sharma**: Visualization, Validation.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Harish Kumar reports administrative support and equipment, drugs, or supplies were provided by Central University of Haryana. Harish Kumar reports a relationship with Central University of Haryana that includes: employment. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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