

Photocatalytic, antibacterial and electrochemical properties of novel rare earth metal oxides-based nano hybrids



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ABSTRACT

Mixed metal oxide nanocomposites (rare earth-based) have become irreplaceable and tend to display great functioning in all kinds of arenas like as photocatalytic, electrochemical, and biological. NiO-CGSO [NiO-Ce_{0.8}Gd_{0.2}O_{2-δ}-Ce_{0.8}Sm_{0.2}O_{2-δ}] nanomaterial was produced by the wet-chemical route for numerous purposes. The development of (FCC) face-centered cubic structure confirmed and there was no derivative phase was observed by XRD. Metal-Oxygen bond was revealed by FTIR analysis. The morphology and elemental composition of the sample were carried out using SEM with EDAX. The optical bandgap of prepared nanocomposite was studied using UV-Vis spectroscopy. Electrochemical behaviour was observed at conditions, voltage (1.3 V), and the frequency (42 Hz–5 kHz). Photocatalytic and antibacterial behavior of prepared NiO-CGSO nanocomposites also investigated. It was found that this novel composite catalyst decomposed 92% of toxic pollutants from wastewater. Further, NiO-CGSO composites showed superior antibacterial performance against *aeromonas hydrophila*, *E. coli*, and *S. epidermis* bacterial pathogens.

1. Introduction

Metal oxide nanocomposites (NCs) have extensive appliances for instance photocatalysis, chemical, biosensors, and anti-resistant bacteria and therapeutic agents [1]. In the value of ecological remediation, metal oxide based composites have broad and develop into competent material on account of their lesser bandgap, inexpensive, non-toxicity, thermal, and chemical stability [2]. Rare-earth doped composites were intentioned as feasible anodes for IT-SOFCs. They display typical assorted electronic conductivity [3,4]. It was originated to facilitate composites that could demonstrate exceptional high electron-transfer, conduction, and noxious dyes degradation properties, consecutively, which could sustain to achieve superior power output [5–8].

From literature, it can realize that several organic and inorganic nanoparticles including CuO, NiO, MgO, CeO₂, NiO have been widely utilized in biomedical and other purposes [9–12]. Nanomaterials attracted many young scientists as a result of their appreciable chemical stability, magnetic behaviour, and biocompatibility. The nanomaterials are also eminent in the antibacterial, antifungal, antioxidant, and anticancer activities [13,14].

In the meantime, the photocatalytic semiconductor has prompted scientific awareness owing to their prevalent purposes for the decrease of infectivity from the water and air. It is mostly utilized for H₂ production, odor control, and stimulation of bacteria and tumor cells. The pollutants released straight to the atmosphere need to be photo degraded not including the creation of added misuse and byproducts into H₂O and carbon dioxide [15]. It is an eminent authenticity that water effluence is an enormous confront for the existing and forthcoming generations. In the framework, dyes participate decisive function in the effluence of water bodies and are extremely noxious, which is frequently developed in several manufactories. As dyes show a composite structure, it is compli-

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cated to humiliate in nature. As a result, diverse semiconductor photocatalysis procedures have been implemented to humiliate the unrefined pollution integrated into the water resources [16]. Among many metal oxide nanoparticles (NPs), NiO NPs are widely applied on account of their large bandgap, chemical stability, non-toxicity, and electrochemical activity [8,17]. Such impending properties correlated with the NiO NPs in dissimilar fields like therapeutic, magnetic hyperthermia, sensing, and other applications [9,17–19].

Different techniques have been executed for nanocomposites (NCs) production such as ultra-sonic process, ball milling, microwave, mechanical alloying, sol–gel and chemical-precipitation, etc [21–23]. From above all well-framed methods, the coprecipitation technique is utilized as a valuable scheme for the synthesis of nanoparticles, due to a pleasant route. It targets a swift and harmonized method of reaction as an alternative of formulating from exterior resources. This technique is extra operative for capitulating homogeneity, awfully untainted model, miniature particle size, lesser time, and less exterior energy [25–28]. In this current work, Gd and Sm doped ceria material was inspected methodically using C-TAB surfactant. Cerium has so many applications and especially involved in catalysis and shown excellent features as reported earlier [29]. Also, rare-earth nanostructures have various applications as reported by many researchers [31–35].

In the present work, for the first time, novel NiO-CGSO nanohybrids were synthesized using a wet chemical method in the presence of CTAB surfactant. Synthesized nanohybrids were investigated to determine structural, morphological, antibacterial and electrochemical performance. Further, the study is broadened to inspect the removal of toxic pollutants through the photocatalytic process.

2. Experimental method

2.1. Materials

In this experimental study (NiO-CGSO NC), the aqueous solutions prepared and materials as similar to our earlier report [37,37].

2.2. Method

The flowchart of the procedure for the fabrication of NiO-CGSO NC is revealed in Fig. 1.

2.3. Reactions involved in mechanism: Stepwise

Step by step reactions entailed in the fabrication of NiO-CGSO NC during the experiment can be shown below:

Reaction mechanism for NiO-CGSO

- (i) $7.6\text{NaOH} \xrightarrow{\text{H}_2\text{O}} 7.6\text{Na}^+_{(\text{aq})} + 7.6\text{OH}^-_{(\text{aq})}$
- (ii) $\text{Ni}(\text{NO}_3)_2 \xrightarrow{\text{H}_2\text{O}} \text{Ni}^{2+}_{(\text{aq})} + 2\text{NO}_3^-_{(\text{aq})}$
- (iii) $1.6\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}_{(\text{aq})} \xrightarrow{\text{H}_2\text{O}} 1.6\text{Ce}^{3+}_{(\text{aq})} + 4.8\text{NO}_3^-_{(\text{aq})} + 6\text{H}_2\text{O}_{(\text{aq})}$
- (iv) $0.2\text{Gd}(\text{NO}_3)_3 \xrightarrow{\text{H}_2\text{O}} 0.2\text{Gd}^{3+}_{(\text{aq})} + 0.6\text{NO}_3^-_{(\text{aq})}$
- (v) $0.2\text{Sm}(\text{NO}_3)_3 \xrightarrow{\text{H}_2\text{O}} 0.2\text{Sm}^{3+}_{(\text{aq})} + 0.6\text{NO}_3^-_{(\text{aq})}$
- (vi) $1.6\text{Ce}^{3+}_{(\text{aq})} + \text{Ni}^{2+}_{(\text{aq})} + 0.2\text{Gd}^{3+}_{(\text{aq})} + 0.2\text{Sm}^{3+}_{(\text{aq})} + 7.6\text{OH}^-_{(\text{aq})} + x\text{H}_2\text{O}_{(\text{aq})} \xrightarrow{\text{stirring}} 1.6\text{Ce}(\text{OH})_4 \cdot x\text{H}_2\text{O}_{(\text{s})}$
 $\downarrow + \text{Ni}(\text{OH})_2 \cdot x\text{H}_2\text{O}_{(\text{s})} \downarrow + 0.2\text{Gd}(\text{OH})_3 \cdot x\text{H}_2\text{O}_{(\text{s})}$
 $\downarrow + 0.2\text{Sm}(\text{OH})_3 \cdot x\text{H}_2\text{O}_{(\text{s})} \downarrow$
- (vii) $1.6\text{Ce}(\text{OH})_4 \cdot x\text{H}_2\text{O}_{(\text{s})} + \text{Ni}(\text{OH})_2 \cdot x\text{H}_2\text{O}_{(\text{s})} + 0.2\text{Gd}(\text{OH})_3 \cdot x\text{H}_2\text{O}_{(\text{s})} + 0.2\text{Sm}(\text{OH})_3 \cdot x\text{H}_2\text{O}_{(\text{s})} \xrightarrow{500-100^\circ\text{C}} 1.6\text{Ce}(\text{OH})_4 + \text{Ni}(\text{OH})_2 + 0.2\text{Gd}(\text{OH})_3 + 0.2\text{Sm}(\text{OH})_3 + x\text{H}_2\text{O}_{(\text{g})} \uparrow$
- (viii) $1.6\text{Ce}(\text{OH})_4 + \text{Ni}(\text{OH})_2 + 0.2\text{Gd}(\text{OH})_3 + 0.2\text{Sm}(\text{OH})_3 \times \xrightarrow{300,450,600\text{ and }750^\circ\text{C}} \text{NiO} - \text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta} - \text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{2-\delta(\text{s})} + x\text{H}_2\text{O}_{(\text{g})} \uparrow$

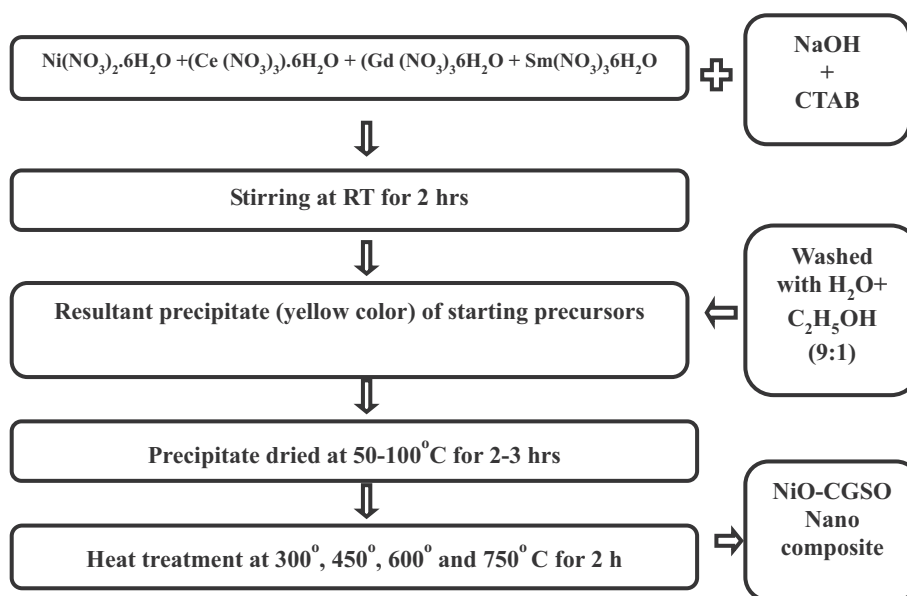
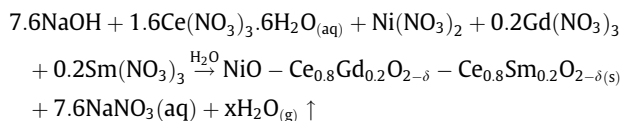


Fig. 1. Flowchart of NiO-CGSO NC preparation.

Overall balanced equation:



2.4. Characterization

To characterize the synthesized composite powders, the phase of the samples was investigated by XRD operating the Shimadzu XRD 6000 X-ray diffractometer K_α radiations ($\lambda = 1.5418 \text{ \AA}$). FTIR spectra were recorded using the JASCO 460 Plus spectrometer over the range from 4000 to 400 cm^{-1} . For this analysis, a small amount of Y_2O_3 samples was blended with KBr and then pressed into pellets for the measurement. The changes in surface morphology of the products were monitored by a scanning electron microscopy (JEOL Model JSM-6360 SEM) operated at 15 kV. Thermal behavior was examined on a Perkin Elmer TGA 7 under N_2 atmosphere at 10 $^\circ\text{C}/\text{min}$ of heating rate. The UV–vis absorption spectrum was obtained from the Agilent 8453 diode array UV–Vis spectrophotometer. The bulk conductivity was projected via impedance analysis. The photocatalytic behavior of the composite was studied under natural light irradiation, and the antimicrobial behavior of the composite was studied by the agar well diffusion method.

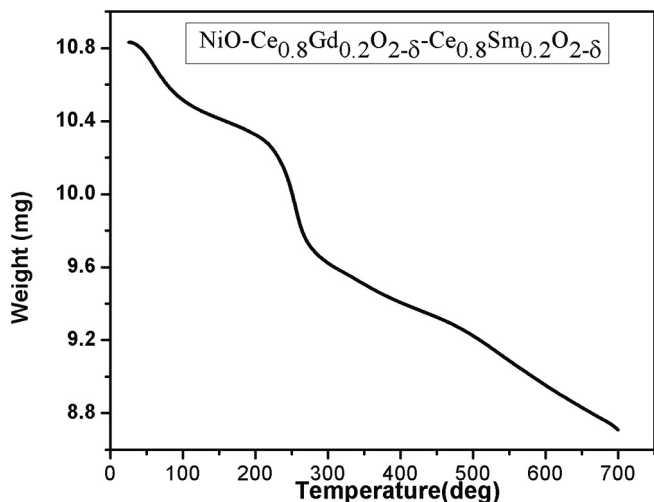


Fig. 2. TGA pattern of the precursor material.

Table 1
Weight loss regions of the forerunner material.

| Phase/Region | Temperature (°C) | Weight failure | Explanation |
|--------------|-------------------|---------------------------|---|
| I | 100 | 1–2% | Water molecule thrashing |
| II | 250 | 5–6% | Phase arrangement of NiO |
| III | Among 250 to 700 | Over 6% | carbon/nitrogen-based compounds decomposition |
| IV | Approximately 700 | Steadiness in Weight loss | Phase-pure nanocomposite configuration |

Table 2
Change in weight reduction recorded from the TGA of precursor material.

| Sample | Primary weight (mg) at 25 $^\circ\text{C}$ | Final weight (mg) at 700 $^\circ\text{C}$ | Total weight loss (mg) | Total Weight loss (%) |
|----------|--|---|------------------------|-----------------------|
| NiO-CGSO | 10.83 | 8.70 | 2.13 | 22% |

3. Results and discussion

3.1. Thermal properties

The prepared forerunner material (Hydroxides of Ni, Ce, Gd, and Sm) by a prelude mass of 8–12 mg was positioned (Pt crucible) and proceed for examination and the output was shown in Fig. 2.

From the above curve, it was assumed that the loss of weight starts to show from the earlier phase itself. The NC thermal decomposition can be allocated into 4 split sections as explained in the earlier reports [5,38] and also the loss of weight modification detected for the forerunner material attained from the TGA data displayed in Tables 1 and 2 respectively.

3.2. Morphological and structural studies

3.2.1. Crystallinity

The XRD of the NiO-CGSO NC discloses the design of FCC (fluorite cubic) well crystalline single-phase as shown in Fig. 3 [40–41]. There are no contamination peaks or any other secondary phase was examined in the XRD pattern of NiO-CGSO NC and the crystallographic planes viewed at (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2), (4 0 0), (3 3 1), (4 2 0) and (4 2 2) as per JCPDS No: 81-0792 are designated in CeO_2 phase [42]. The crystallographic planes observed at (1 1 1), (2 0 0) and (2 2 0) as per JCPDS No: 75-0197 are designated in the NiO phase [43].

The structural parameters of the samples were computed (from the XRD data) as reported earlier [20,25,44] and the obtained values shown in Table 3.

3.2.2. FTIR studies

FTIR spectrum of NiO-CGSO NC was exposed in Fig. 4. The significant peaks noticed from the FTIR spectrum ascribed for distinguishing peaks have been listed in Table 4.

3.2.3. Morphological studies

The SEM characterization and EDAX analysis of images acquired on NiO-CGSO NC calcined at 750 $^\circ\text{C}$ are presented in Fig. 5. SEM

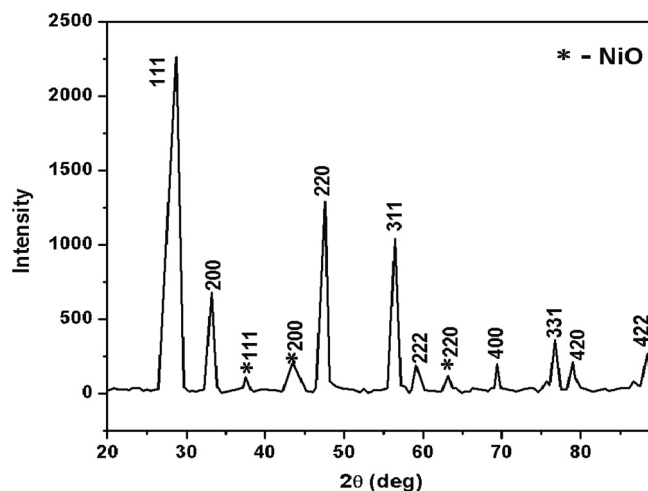


Fig. 3. The obtained XRD pattern of NiO-CGSO NC.

Table 3
The structural parameters of NiO- NC.

| Lattice parameter | CeO ₂ phase (JCPDS No. 81–0792) | Doped CeO ₂ phase of NiO-CGSO | NiO phase (JCPDS No. 75–0792) | NiO phase of NiO-CGSO |
|----------------------------------|--|--|-------------------------------|-----------------------|
| Crystallite structure | Cubic (FCC) | Cubic (FCC) | Cubic (FCC) | Cubic (FCC) |
| Lattice parameter 'a' (Å) | 5.412 | 5.412 | 4.170 | 4.161 |
| Lattice volume (Å ³) | 158.516 | 158.516 | 72.511 | 72.043 |
| Speculative density (g/cc) | 7.2110 | 8.139 | 6.8430 | 6.885 |
| Crystallite size (nm) | 20.8 | – | 20.8 | – |

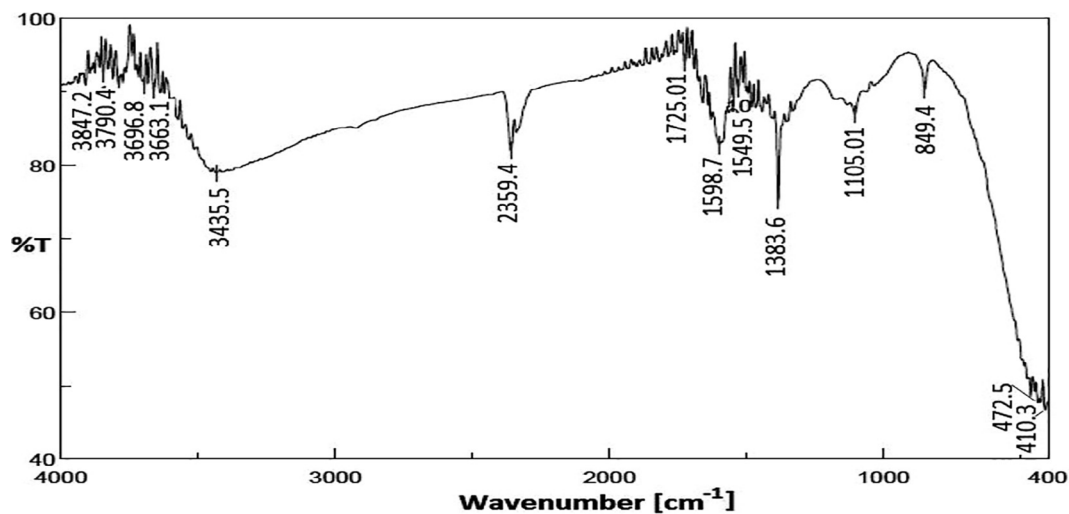


Fig. 4. FTIR spectrum obtained on NiO-CGSO NC.

Table 4
FTIR consignments of the prepared sample.

| Material | Obligation of distinctive peaks (Cm ⁻¹) | | | | |
|-----------------|---|----------|----------------|-------------------|---------------|
| | Ce-O | Ni-O | Carbon dioxide | δ (H-O-H) bending | OH stretching |
| Reference Peaks | 1383.2 | Near 400 | 2360.5 | 1600.7 | 3400.8 |
| NiO-CGSO | 1384 | 410 | 2359 | 1599 | 3435 |
| Ref. | [46] | [45] | [47] | [46] | [45] |

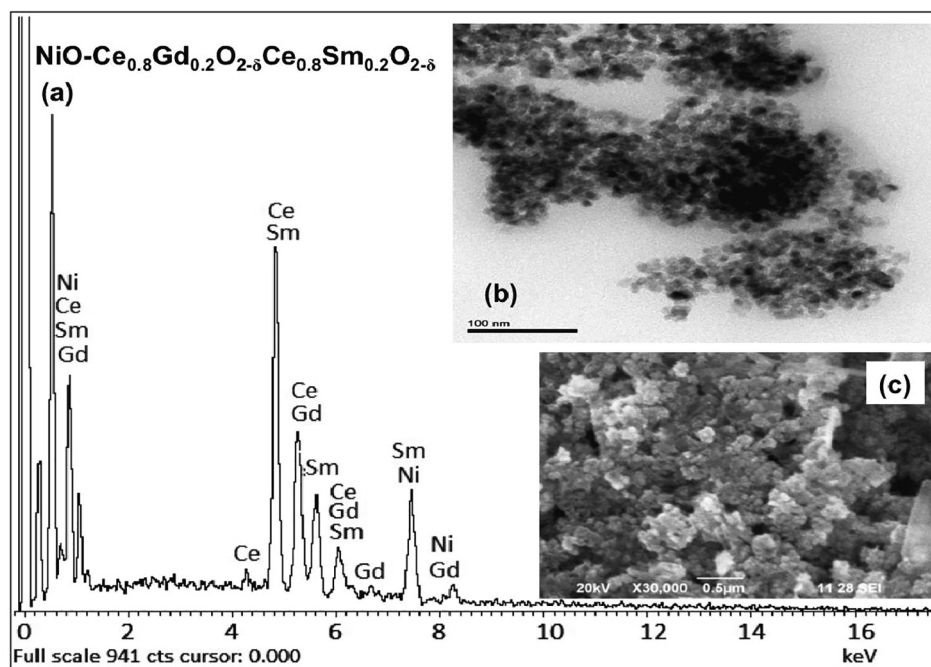


Fig. 5. (a) EDAX, (b) TEM, and (c) SEM data obtained on NiO-CGSO nanocomposite.

photographs (Fig. 5c) and TEM images (Fig. 5b) were displayed in Fig. 5.

TEM and SEM pictures obtained on NiO-CGSO NC reveal the clear spherical shaped particles and the size was shown in the range of 30 to 60 nm. It is also observed that the minority microparticles showed owing to cluster [25,25]. The supplement of the CTAB (surfactant) prohibited the chance of remarkable cluster to achieve well NC. EDAX disclosed the occurrence of the elemental composition obtained from the NiO-CGSO NC as Ni (13.4%), Ce (49.4%), Gd (5.8%), Sm (4.2%), and O (27.2%). There are no contamination peaks observed in EDAX spectra and also suggested the purity of the composite.

3.2.4. Optical studies

The optical studies of NiO-CGSO NC were deliberated by UV-Visible spectroscopy. The spectrum was recorded in the region

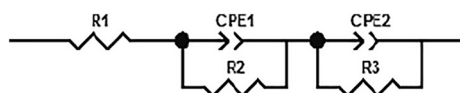


Fig. 6. Corresponding circuit (2RQR) employed for fitting data.

from 200 to 800 nm wavelength at RT. The energy bandgap of the NC can be determined from the E_g measurements via tau's relation formula. The nanocomposite exhibits a strong absorbance peak (350 nm) wavelength. The analyzed bandgap of NiO-CGSO composite was initiated to be 3.54 eV [27–28].

3.3. Electrochemical behavior

The circular compacts of prepared the NC, diameter (10 mm), thickness (2 mm), and pressure (1.2 ton) using hydraulic pressure pelletizer. To achieve a more densified state, the sintering temperature was applied at 750 °C for 3 h to diminish pores.

Table 5
The calculated conductivity values of NiO-CGSO NC at diverse temperatures.

| Temperature (K) | Conductivity (S/cm) |
|-----------------|--------------------------|
| 310 | 7.8151×10^{-06} |
| 573 | 7.3273×10^{-06} |
| 673 | 1.2710×10^{-04} |
| 773 | 4.3843×10^{-04} |
| 873 | 1.2501×10^{-03} |

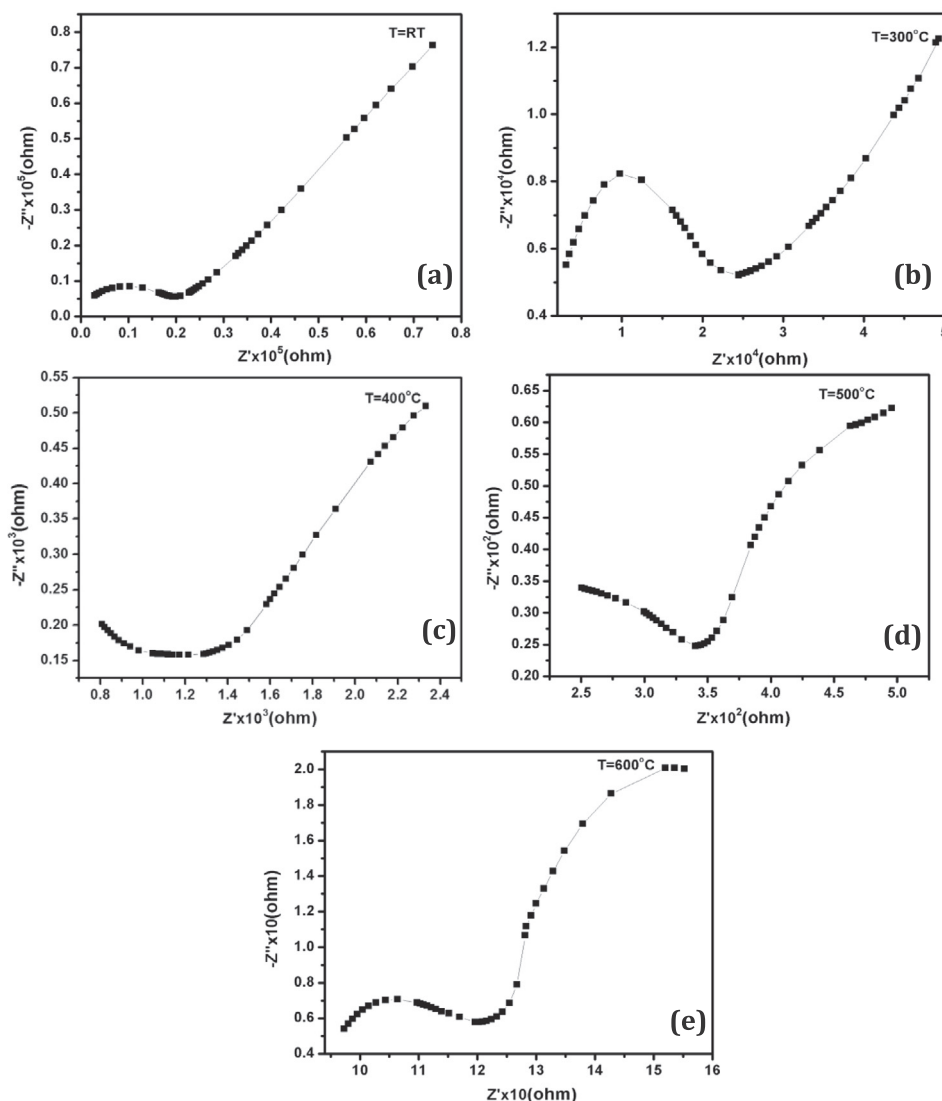


Fig. 7. (a–e) Impedance curves of NiO-CGSO NC at different temperatures.

Table 6
The calculated activation energies for NiO-CGSO NC.

| Material | Temperature (°C) | 1000/T (K ⁻¹) | logσT (Scm ⁻¹ K) | slope | Activation energy (eV) |
|----------|------------------|---------------------------|-----------------------------|--------|------------------------|
| NiO-CGSO | 400 | 1.492 | -3.895 | -2.887 | 0.249 |
| | 500 | 1.298 | -3.358 | | |
| | 600 | 1.149 | -2.903 | | |

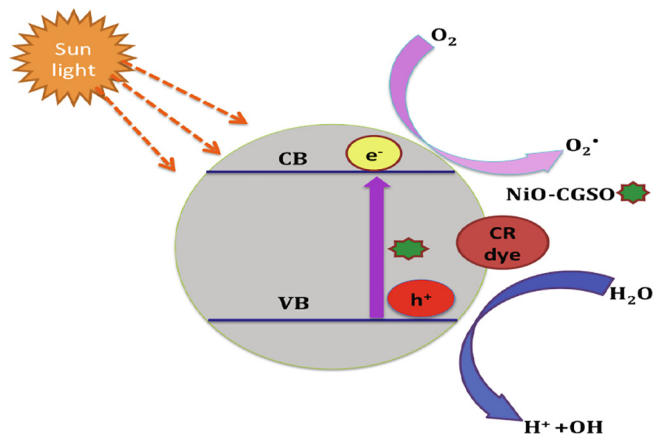


Fig. 8. Schematic mechanisms of CR dye degradation of NiO-CGSO NC.

At various temperatures (RT, 300, 400, 500, and 600 °C), ac impedance measurements were taken with the standard conditions. Data fitting for the measures was completed with the ZVIEW software by applying the following equivalent circuit (2RQR) and it is represented in Fig. 6. The plots obtained from the sample at diverse temperatures showed in Fig. 7(a–e).

From the data, it is observed that NiO-CGSO NC exhibited the optimum value of conductivity at high temperatures and the data shown in Table 5. Petrovsky et al reported innovative nanomaterial; Sm-doped ZrO₂ for application in ITSOFC [30–43].

The Arrhenius linear fit relationship is employed to estimate the activation energy of the prepared composite. When the conductivity enhances the activation energy also increased and the obtained values are shown in Table 6.

3.4. Photocatalytic performance

The photocatalytic nature of the NC was analyzed from the photodegradation performance of CR dye beneath the natural light irradiation process and as shown in Fig. 8. The CR degradation efficiency compared with earlier reports is shown in Table 8.

The CR dye activity of NiO-CGSO NC was evaluated under natural light irradiation. The absorption spectrum of CR is an illustration (Fig. 9) and the distinguishing absorption of CR (664 nm) diminishes hurriedly through enhanced coverage time. This species to facilitate the solute dye concentration declines promptly and virtually disappears in 120 min.

The degradation percentage is 92 at 120 min. The photodegradation of CR dye obeys pseudo-first-order kinetics. From the kinetic

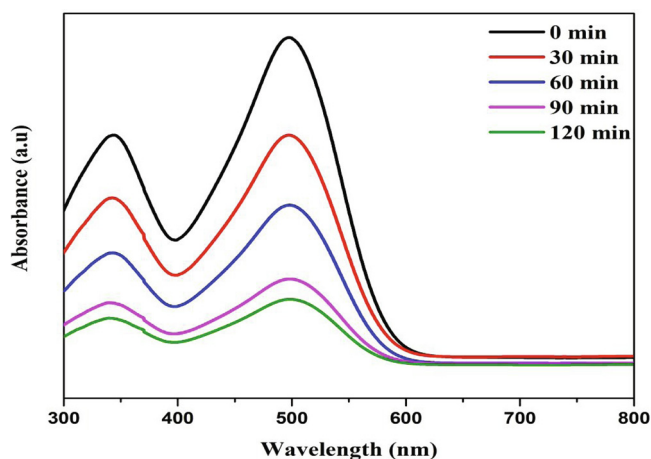


Fig. 9. Absorbance spectrum of NiO-CGSO NC for CR under nature sunlight irradiation.

study, NiO-CGSO NC reveals an excellent CR dye action and the kinetic constant (k) is 0.0056 min⁻¹.

From Table 7, NiO-CGSO nanohybrids show importantly higher catalytic activity under natural light irradiation than the existing commercial catalyst.

3.5. Antibacterial behavior

The antimicrobial nature was assessed alongside gram-negative such as *Aeromonas hydrophila*, *E. coli*, and *S.epidermis* bacterial pathogens using NiO-CGSO NC (Fig. 10). Table 8 illustrates the Zone of inhibition (ZOI) of NiO-CGSO alongside pathogenic bacteria.

From Table 8, chemical precipitated NiO-CGSO NC show an exceptional antimicrobial activity against foodborne pathogens and also analogous with the customary antibiotics (Streptomycin).

Photolytic production of reactive oxygen species (ROS) on the surface of NiO-CGSO NC. NiO-CGSO NC is due to the formation of hydroxyl, superoxide radicals, and H₂O₂ (ROS) by the Fenton reaction leading to lipid peroxidation, DNA injure and protein decay can exterminate bacteria without destructive nonbacterial cells.

There are additional probable steps engaged in the antimicrobial activity. NiO-CGSO NC impedes the bacteria cell membrane and connects with mesosome (cellular inhalation, DNA reproduction, cell partition). These intracellular functional alterations are commenced by the oxidative stress manipulated by ROS foremost

Table 8
Antibacterial activity of NiO-CGSO NC at diverse concentrations.

| Tested bacteria | Gram reaction | ZOI (mm) | | | | Positive control (Streptomycin) | Negative control |
|-----------------------------|---------------|----------|----------|----------|----------|---------------------------------|------------------|
| | | 20 µg/mL | 40 µg/mL | 60 µg/mL | 80 µg/mL | | |
| <i>S. epidermis</i> | -ve | 12 | 15 | 17 | 21 | 28 | - |
| <i>E. coli</i> | -ve | 14 | 16 | 19 | 22 | 25 | - |
| <i>Aeromonas hydrophila</i> | -ve | 10 | 12 | 15 | 20 | 30 | - |

Table 7
Photocatalytic performance (CR dye) of NiO-CGSO NC related to metal oxide nanocomposites.

| Photocatalyst | Preparation technique | Concentration | Degradation percentage (%) | Reaction time (min) | Source | Ref. |
|--|-------------------------------|---------------|----------------------------|---------------------|-------------------------------|--------------|
| Mg-TiO ₂ -P25/PMS | Sonochemical | 50 mg | 95 | 120 | 150 W tungsten halogen lamp | [44] |
| Mg-TiO ₂ - P25/PDS | Modified auto combustion | 1 g/L | 75 | 180 | 100 W tungsten visible lamp | [45] |
| Fe-CeO ₂ (P) | | | 87 | | | |
| CeO ₂ | | | 82 | | | |
| Fe-CeO ₂ (Sg) | Hydrothermal | 50 mg | 48 | 90 | Visible light | [46] |
| MnFe ₂ O ₄ /ZnO | | | 54.4 | | | |
| ZnO/TA/ MnFe ₂ O ₄ | | | 70.2 | | | |
| MnFe ₂ O ₄ /TA/ZnO | Hydrothermal | 10 mg | 84.2 | 60 | LED-30 W | [47] |
| g-C ₃ N ₄ /RGO/ Bi ₂ Fe ₄ O ₉ | | | 87.65 | | | |
| ZnMnO ₃ /Fe ₃ O ₄ | | | 0.1 g | | | |
| SnS ₂ -CdO | Cost-effective chemical route | 6 mg | 92.86 | 210 | 350 W Xenon arc lamp | [49] |
| Ag/1.0 Mn ₃ O ₄ | Sol-gel method | 3 mg | 78 | 120 | 40 W UV-Vis light irradiation | [50] |
| NiO-CGSO | Wet chemical route | 5 mg | 92 | 120 | Sun light irradiation | Present work |

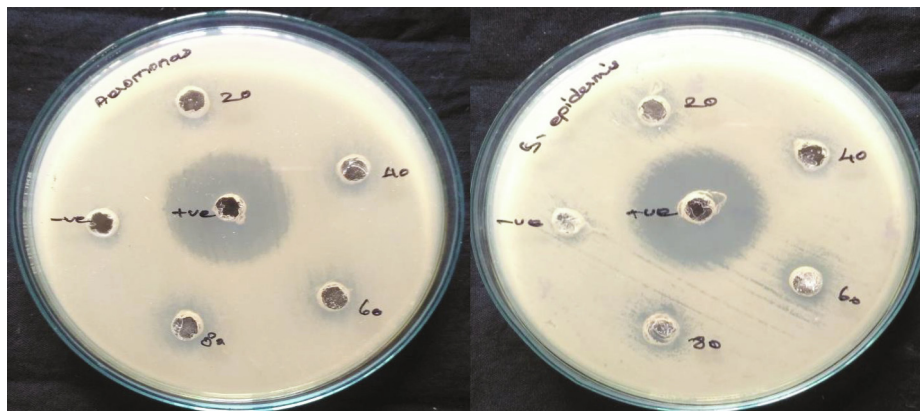


Fig. 10. Plate photos of antibacterial activity of NiO-CGSO NC.

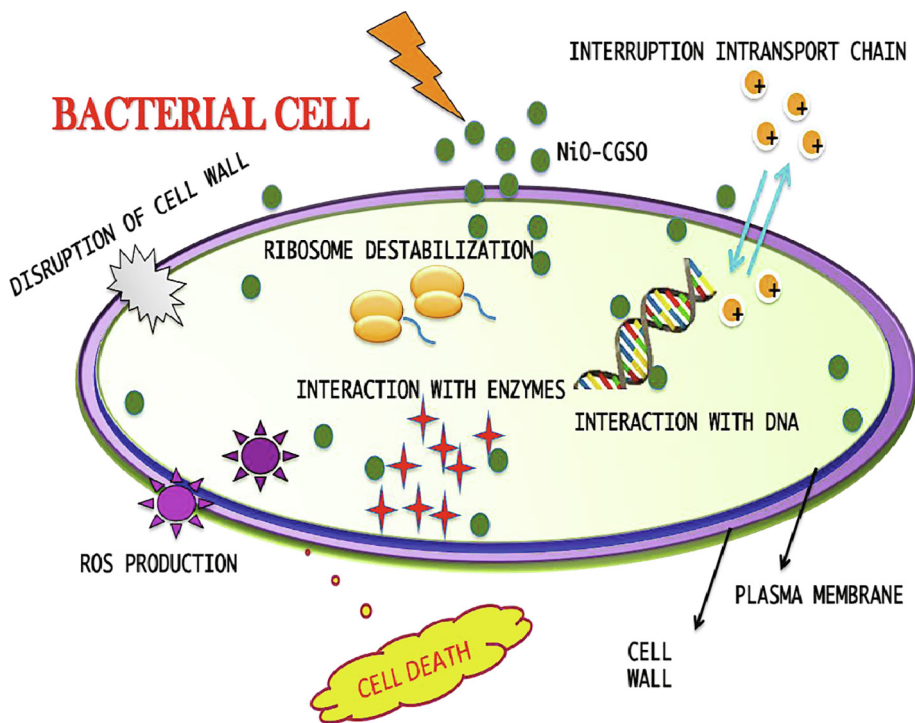


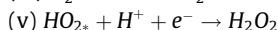
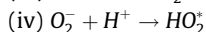
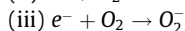
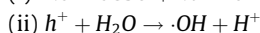
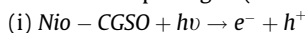
Fig. 11. Antibacterial activity mechanism of NiO-CGSO NC.

Table 9
Antibacterial activity for NiO-CGSO NC with previous reports against *E. coli*.

| Materials | ZOI (mm) | Ref. |
|---|----------|--------------|
| NiO.CeO ₂ .ZnO | 11 | [53] |
| Ag ₂ O.CeO ₂ .ZnO | 12 | [54] |
| CdO–NiO–ZnO | 16 | [17] |
| NiO-CGSO | 22 | Present work |

to cell termination as demonstrated in Fig. 11. Only a few works of literature describe the electrostatic appeal mechanism.

Gd³⁺ and Sm³⁺ are released owing to the communication of NiO-CGSO with the microbial cell membrane. The negatively charged cell wall and positively stimulating Gd³⁺ and Sm³⁺ are mutually fascinated and they root denaturation of proteins, which outcome in thrashing of replica capacity of the DNA thus reasoning the termination of the pathogen (Table 9).



The rough surface texture was responsible for mechanical injure to the cell membranes. It is comprehensible that NiO-CGSO NC has irregular crumples at the exterior surface (SEM and TEM images), which influences the antimicrobial efficacy. The higher concentrations of NiO-CGSO NC are deleterious to both the clients and microbes, but still, concentrations (nano-level) are appropriate for the annihilation of microbes [55,52].

4. Conclusion

In this work, NiO-CGSO NC was effectively extended via a wet chemical route, i.e., co-precipitation route. TGA and XRD patterns exposed the methodology to obtain phase pure materials. The prepared composite structure was validated via XRD and FTIR analysis. Using SEM and EDAX, morphology, and elemental analysis of the sample were examined. The conductivity data of the sample exposed that the sample projected in this paper might be appropriate for anode appliance in SOFC systems. The antibacterial and photocatalytic performance of prepared nanocomposite was investigated and it has shown excellent results, which are further useful to apply in different fields.

CRediT authorship contribution statement

Karthik Kannan: Conceptualization, Methodology, Writing - original draft. **D. Radhika:** Resources, Writing - review & editing, Investigation. **Kishor Kumar Sadasivuni:** Data curation. **Kakarla Raghava Reddy:** Writing - review & editing. **Deepak Kasai:** Methodology. **Anjanapura V. Raghu:** Project administration, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors 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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