Spinel ZnCr$_2$O$_4$ nanorods synthesized by facile sol-gel auto combustion method with biomedical properties

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Abstract

In this study, spinel zinc chromite nanorods (ZnCr$_2$O$_4$ NRs) were successfully manipulated by a simple sol-gel auto combustion process employing urea as fuel. The sample was only required to sinter at 500 °C for 2 h to obtain the single crystalline phase. The phase formation, crystallinity, and surface topography of synthesized ZnCr$_2$O$_4$ NRs were explored by X-ray diffraction (XRD), UV-Vis reflectance spectroscopy (UVDRS), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FESEM), high-resolution transmission electron microscopy (HRTEM), energy dispersive X-ray (EDX) spectroscopy, and vibrating sample magnetometry (VSM). XRD analysis confirms the formation of spinel ZnCr$_2$O$_4$ NRs. The FTIR spectrum displays the two vibrational peaks of Cr–O, and Zn–O at 489 and 615 cm$^{-1}$, correspondingly. These vibrational bonds were correlated with ZnCr$_2$O$_4$ and revealed the production of cubic spinel ZnCr$_2$O$_4$ NRs. FESEM indicates the presence of hexagonal-rod-shaped particles. EDX spectrum demonstrates the elemental composition of the ZnCr$_2$O$_4$ NRs and confirms the primary peak of Zn, Cr, and O. The obtained ZnCr$_2$O$_4$ NRs exhibit an antiferromagnetic behavior. The bandgap energy of ZnCr$_2$O$_4$ NRs was ascertained and was shown to be 3.45 eV. Furthermore, the antifungal and antibacterial effect of ZnCr$_2$O$_4$ NRs was examined against pathogenic strains by disc diffusion technique. Besides these, the antimalarial activity of ZnCr$_2$O$_4$ NRs was studied against Plasmodium falciparum. Thus, the as-synthesized ZnCr$_2$O$_4$ NRs showed significant antibacterial, antifungal and antimalarial activity and may be helpful for research opening a novel horizon in nanomedicine.
Graphical abstract

Keywords  
ZnCr$_2$O$_4$ NRs • Structural properties • Antibacterial activity • Antifungal activity • Antimalarial activity

Highlights

- Spinel ZnCr$_2$O$_4$ nanorods (NRs) successfully synthesized via sol-gel auto combustion approach.
- UV-Vis-DRS, XRD, FESEM, EDX, HRTEM-SAED, VSM, and FTIR analyses were performed.
- FESEM and HRTEM analysis revealed the hexagonal-shaped morphology for ZnCr$_2$O$_4$ NRs.
- The ZnCr$_2$O$_4$ NRs exhibited excellent antibacterial, antifungal, and antimalarial activity.

1 Introduction

Nanotechnology has emerged as one of the most exciting vanguard sectors in recent years, earning much attention from the scientific community [1–3]. Inorganic nanomaterials with superior chemical, mechanical, electrical, and physical properties can be employed in applications such as electronic devices, medicine, agriculture, sunscreens, catalysts, photovoltaic cells, military applications, biological properties, paints, sensing, and the food industry [4–8].

To date, viral infections remain among the most challenging problems facing global healthcare systems. Things can become complicated if we are not ready to promptly deal with a viral outbreak, as was the case with the recent Coronavirus Disease 2019 (COVID-19) pandemic. This highlights the necessity for quick and inexpensive biosensing platforms and in-depth knowledge of potential antiviral effects and drug-delivery opportunities. The same difficulties have been encountered with non-viral immunogenic diseases. Nanomedicine is therefore viewed as a cutting-edge candidate for successfully resolving these global concerns. Due to its unique and inspiring physicochemical properties, such as its large surface area, efficient thermal, magnetic, and electrical properties, ability to detect single molecules, easy functionalization, and anticancer and antibacterial/antiviral properties, it is one of the adaptable nanomaterials frequently used in biomedical applications [9–11].

Among many promising nanomaterials, spinel and spinel-like nanomaterials have been broadly considered in recent years due to their imperative properties in material science [12–15], resulting in versatile applications such as wastewater treatment [16], energy storage [17], photocatalytic [18, 19], organic transformations [20] and nanomedicine [21, 22]. Together with ternary spinel oxide materials, zinc chromite nanoparticles (ZnCr$_2$O$_4$ NPs) have attracted interest owing to their special applications in the fields of photocatalysis [23], sensing [24], organic transformation [25], and superconductors [26]. Till now, ZnCr$_2$O$_4$ NPs have been explored by several notable methods (as shown in Table 1), such as hydrothermal [23], co-precipitation [27], thermal decomposition [28], reflux condensation and calcination [29], sol-gel auto combustion [30], water-in-oil (W/O) microemulsion [26], thermolysis [31], homogeneous precipitation using urea hydrolysis [32], ultrasonic spray pyrolysis [33], solution combustion [34], solution casting [35], microwave [36], solvothermal methods [37]. In typical, the conventional sol-gel auto-combustion derived spinel ZnCr$_2$O$_4$ NPs, which was proposed by Javed et al., have attracted attention [30]. However, this method requires a long time (>36 h) with multi-steps of sintering at high temperatures (350–600–900 °C) to obtain the single crystalline phase of ZnCr$_2$O$_4$ NPs. Therefore, there remains a needing to find a simple, speedy, effective, and energy-saving method to synthesize ZnCr$_2$O$_4$ NPs.

Herein, we propose a facile and straightforward sol-gel auto combustion method to rapidly fabricated spinel zinc chromite nanorods (ZnCr$_2$O$_4$ NRs) by using urea as a fuel agent. The sample was only required to calcine at 500 °C for
3.9H₂O), and urea. Urea is an organic fuel that serves as a good platform for redox reactions to occur during combustion. Firstly, zinc nitrate, chromium nitrate, and urea were added in 1:1:4 stoichiometric ratios to make a homogeneous paste. Subsequently, the resulting paste was placed on a hot plate for 2 h at about 70–80 °C; thick gel was formed after complete evaporation. This gel was heated to 170–180 °C for 4 h on a hot plate for auto combustion. To obtain nanocrystalline ZnCr₂O₄ powder was calcined for 2 h at 500 °C in a static air box furnace [38, 39]. Finally, the fine pale green-colored shining powder was formed, and this was meticulously collected and stored for further uses.

2.3 Antibacterial activity of ZnCr₂O₄ NRs

Varied concentrations (Table 1) of synthesized ZnCr₂O₄ NRs were evaluated for antimicrobial activity by the disc diffusion method [40, 41]. The bactericidal effect was tested against *Bacillus subtilis*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Salmonella Typhi*, *Klebsiella pneumonia*, *Proteus vulgaris*, *Staphylococcus epidermidis*, *Staphylococcus aureus*, *Streptococcus pneumoniae*, and *Streptococcus pyogenes*. Ampicillin (25 µg/mL) was employed as a reference drug for the antibacterial screening.

### Table 1: A summary of methods to synthesize zinc chromite nanoparticles (ZnCr₂O₄ NPs)

| No. | Synthesis method                              | Preparation process                                                                 | Morphology                                      | Physical properties                                      | Ref.   |
|-----|----------------------------------------------|--------------------------------------------------------------------------------------|------------------------------------------------|----------------------------------------------------------|--------|
| 1   | Hydrothermal                                 | NaOH was dropped into the mixture of Zn(NO₃)₂•6H₂O and Cr(NO₃)₃•9H₂O. Then, the obtained suspension was autoclaved at 220 °C for 48 h. Following, it was washed and dried at 60 °C for 4 h. | Nanoparticles, irregular in shape               | Particle size: <5 nm; bandgap (Eₚ): 3.46 eV.             | [23]   |
| 2   | Co-precipitation                             | The alkaline solution was dropped into the mixture of Zn(NO₃)₂•6H₂O, capping agent, and CrCl₂•6H₂O. Then, the obtained mixture was heated at 60 °C for 30 min, washed, and dried under a vacuum at 70 °C. Following, the powder was calcined in air at 700 °C for 3 h. | Nanoparticles, coalescent and turn into bulk structures | Particle size: 70 nm; Eₚ: 3.35–3.96 eV.               | [27]   |
| 3   | Thermal decomposition                        | Ammonia solution was added to the mixture of (NH₄)₂CrO₄ and ZnCl₂. Then, the obtained mixture was studied by heating at 200–1000 °C for 2 h. | Nanoparticles                                   | Particle size: 3–55 nm; specific surface areas: 5–60 m²/g. | [28]   |
| 4   | Conventional sol-gel auto combustion         | Ammonia solution was added to the mixture of Zn(CH₃COO)₂•2H₂O, C₆H₈O₇, and Cr(NO₃)₃•9H₂O; then, it was heated at 110 °C for 2.5 h. Following, the resulting aerogel was further heated at about 350 °C. The collected powder was calcined at 600 °C for 8 h. Finally, it was sintered at 900 °C for 24 h. | Pebble-like growth of the grains               | Particle size: 144 ± 4.8 nm; Density: 5.0106–5.3598 g/cm³. | [30]   |
| 5   | Homogeneous precipitation using urea hydrolysis | Solid urea (NH₂CONH₂) was first added to a mixture of Zn(CH₃COO)₂•2H₂O and Cr(NO₃)₃•9H₂O. Then, it was refluxed under magnetic stirring at 90 °C for 8 h. The obtained precipitations were filtered and washed; following, it was dried at 160 °C for 24 h and then calcined at 500 °C for 8 h. | Nanoparticles                                   | Particle size: 13 nm; Surface areas: 47 m²/g; Eₚ: 1.8 eV. | [32]   |
| 6   | Sol-gel auto combustion                      | The paste of Zn(NO₃)₂•6H₂O, urea, and Cr(NO₃)₃•9H₂O was heated at 70–80 °C and 170–180 °C for 2 h and 4 h, respectively. Following, it was calcined in air at 500 °C for 2 h. | Nanorods                                        | Particle size: 45–80 nm; This study Eₚ: 3.45 eV.       |        |

2 h to obtain the single crystalline phase. These synthesized ZnCr₂O₄ NRs were also assessed for antibacterial, antifungal, and antimalarial activities by employing them against chosen pathogenic strains. It was found that efficiently synthesized ZnCr₂O₄ NRs manifested good biomedical application in nanomedicine.

### 2 Experimental

#### 2.1 Materials

Zinc nitrate (Zn(NO₃)₂•6H₂O), chromium nitrate (Cr(NO₃)₃•9H₂O), and urea were used as a precursor for the synthesis of ZnCr₂O₄ NRs. All the required analytical grade chemicals were procured from SRL Chem, India, and used without additional purification.

#### 2.2 Synthesis of ZnCr₂O₄ NRs

ZnCr₂O₄ NRs were successfully fabricated by a simple sol-gel auto combustion technique using the precursors as zinc nitrate (Zn(NO₃)₂•6H₂O), chromium nitrate (Cr(NO₃)₃•9H₂O), and urea. Urea is an organic fuel that serves as a
2.4 In vitro antifungal efficacy of ZnCr$_2$O$_4$ NRs

The antifungal performance for ZnCr$_2$O$_4$ NRs was determined against fungal strains such as *Aspergillus clavatus*, *Aspergillus niger*, *Candida albicans*, *Epidermophyton floccosum*, and *Trichophyton mentogrophytes*. This in vitro antifungal potential was assessed using Pagar et al. and Ghotekar et al. methods [42, 43]. Greseofulvin was applied as a standard drug for in vitro antifungal screening, with the highest dilution indicating at least a 99% growth inhibition zone is considered as MIC.

2.5 In vitro antimalarial efficacy of ZnCr$_2$O$_4$ NRs

According to the modified protocol, the antimalarial effect for ZnCr$_2$O$_4$ NRs was carried out in 96 well microtitre plates [43, 44]. The antimalarial effect was studied against the *Plasmodium falciparum* strain. Quinine and Chloroquine were used as the reference drugs for the study.

2.6 Materials characterization

The crystal structures of the ZnCr$_2$O$_4$ NRs were determined by the XRD pattern (Brukar, D8-Advanced Diffractometer). The nature of the chemical bonding was recorded by FTIR spectrum on JASCO 4100. The surface morphology of ZnCr$_2$O$_4$ NRs was investigated by the FESEM technique (JEOL, JSM-6360). EDX technique was used to study the elemental composition of ZnCr$_2$O$_4$ NRs (Bruker, XFlash 6130). The surface topography and size of the synthesized ZnCr$_2$O$_4$ NRs were explored via the HRTEM studies, and the results are shown in Fig. 3(a, b). The TEM image in Fig. 3(a) showed the hexagonal morphology of spinel ZnCr$_2$O$_4$ NRs with a particle size of about 45–80 nm, consistent with the morphology obtained through FESEM analysis. However, the ZnCr$_2$O$_4$ NRs did not exhibit agglomeration. Figure 3(b) corresponded to the selected area electron diffraction (SAED) pattern of the ZnCr$_2$O$_4$ NRs and showed several bright spots in the SAED pattern through the single-phase, and polycrystalline nature of the spinel ZnCr$_2$O$_4$ NRs was confirmed. SAED patterns were consistent with the results obtained from the XRD study indicating the crystal planes of ZnCr$_2$O$_4$ NRs. Moreover, the elemental composition of the synthesized ZnCr$_2$O$_4$ NRs was revealed EDX spectrum, as shown in Fig. 4. This EDX study represents the existence of zinc (Zn), chromium (Cr), and oxygen (O) in the sample, indicating the formation of ZnCr$_2$O$_4$ NRs.

3 Results and discussion

3.1 Crystallographic analysis

The XRD data were studied to know the formation and crystalline structure of the as-synthesized ZnCr$_2$O$_4$ NRs. Figure 1 revealed characteristic diffraction peaks at 30.34° (2 2 0), 35.74° (3 1 1), 37.4° (2 2 2), 43.44° (4 0 0), 53.92° (4 2 2), 57.46° (5 1 1), 63.08° (4 4 0) and 74.68° (5 3 3). These peaks could be assigned to the spinel ZnCr$_2$O$_4$ with the standard JCPDS Card No. 22-1107. This result is in good agreement with previous studies [45, 46].

![XRD profile of ZnCr$_2$O$_4$ NRs](image)

Fig. 1 XRD profile of ZnCr$_2$O$_4$ NRs

3.2 Morphological and elemental study

FESEM analysis was used to study the morphological characteristics and sizes of fabricated ZnCr$_2$O$_4$ NRs, as shown in Fig. 2. According to FESEM images, the hexagonal-rod shaped morphology and uniform distribution were obtained for as-prepared NRs. Also, morphological analysis of the as-synthesized ZnCr$_2$O$_4$ NRs was performed through the HRTEM studies, and the results are shown in Fig. 3(a, b). The TEM image in Fig. 3(a) showed the hexagonal morphology of spinel ZnCr$_2$O$_4$ NRs with a particle size of about 45–80 nm, consistent with the morphology obtained through FESEM analysis. However, the ZnCr$_2$O$_4$ NRs did not exhibit agglomeration. Figure 3(b) corresponded to the selected area electron diffraction (SAED) pattern of the ZnCr$_2$O$_4$ NRs and showed several bright spots in the SAED pattern through the single-phase, and polycrystalline nature of the spinel ZnCr$_2$O$_4$ NRs was confirmed. SAED patterns were consistent with the results obtained from the XRD study indicating the crystal planes of ZnCr$_2$O$_4$ NRs. Moreover, the elemental composition of the synthesized ZnCr$_2$O$_4$ NRs was revealed EDX spectrum, as shown in Fig. 4. This EDX study represents the existence of zinc (Zn), chromium (Cr), and oxygen (O) in the sample, indicating the formation of ZnCr$_2$O$_4$ NRs.

3.3 Vibrational properties

The nature of chemical bonding was studied using the FTIR spectrum. The FTIR spectrum of the synthesized spinel ZnCr$_2$O$_4$ NRs is described in Fig. 5. The FTIR spectrum indicates two main strong peaks of bending vibration of Cr–O at 489 cm$^{-1}$ and Zn–O at around 615 cm$^{-1}$, suggesting the successful formation of spinel structure of ZnCr$_2$O$_4$ NR in the cubic phase [39, 47]. Furthermore, the
internal lattice vibrations frequencies of tetrahedral and octahedral coordination compounds in the spinel structure are compatible with these prominent IR vibration bands of Cr–O and Zn–O [48]. This result is consistent with previous finding [47].

3.4 Optical study of ZnCr$_2$O$_4$ NRs

The optical absorbance of the fabricated ZnCr$_2$O$_4$ NRs was studied based on the UV-Vis absorbance data, and the corresponding spectrum is depicted in Fig. 6. Transitions of
octahedral $\text{Cr}^{3+}$ (d$^3$) ions can be ascribed to the signals at 410–450 nm and 580–620 nm [49, 50]. Bandgap absorption of ZnCr$_2$O$_4$ NRs is accountable for the major peak in the UV region. The absorption wavelength for ZnCr$_2$O$_4$ NRs is 292 nm, indicating that they absorb UV light. The bandgap energy of ZnCr$_2$O$_4$ NRs is calculated by using Tauc’s plot [51, 52] (Fig. 7) and was estimated to be 3.45 eV.

### 3.5 Magnetic properties

A VSM was applied to investigate the magnetic behavior of ZnCr$_2$O$_4$ NRs. The hysteresis loop for ZnCr$_2$O$_4$ NRs is depicted in Fig. 8. The applied field ranged from $-10$ kOe to 10 kOe to study magnetic characteristics. The values of $M_s$, $H_c$, and $M_r$ were estimated based on this figure and are shown in Table 2. As a result, synthesized ZnCr$_2$O$_4$ NRs shows an antiferromagnetic behavior. Previously, a similar magnetic behavior of ZnCr$_2$O$_4$ NRs has been reported in the literature [53, 54].
3.6 Antibacterial efficacy of ZnCr$_2$O$_4$ NRs

The bactericidal effect of the prepared ZnCr$_2$O$_4$ NRs with diverse concentrations was assessed against ten various bacterial strains, and the zone of inhibition (ZOI) was recorded. As shown in Table 3, the antibacterial efficacy was observed using ZnCr$_2$O$_4$ NRs and ampicillin. The bactericidal performance depended on the concentration of the ZnCr$_2$O$_4$ NRs and bacterial strains. The higher the ZOI obtained with increasing ZnCr$_2$O$_4$ NRs concentrations employed in antibacterial screenings was also observed. As-prepared ZnCr$_2$O$_4$ NRs displayed potent and good antibacterial performance against *K. pneumonia*, *P. vulgaris*, *S. pyogenus*, *S. aureus*, *B. subtilis*, *S. pneumoniea*, and *S. epidermidis*. However, previously, Taheri et al. [55] revealed the antibacterial performance of zinc chromite-zinc aluminate nanocomposite against *P. aeruginosa* and *E. coli*.

Furthermore, the antibacterial activity of NPs is promising in several fields, especially in medical areas. The size of the NPs plays a fundamental role in their functional training, such as chemical and biological activity. Discovering the mechanism of the antibacterial action of NPs is an attractive aspect of nanobiotechnology. The pathways of the mechanism of antibacterial actions include many steps. First, the physical direct interaction of extremely sharp edges of nanomaterials with cell wall membrane [56]. Second, ROS (reactive oxygen species) could be generated, even in the dark [57, 58]. Third, the bacteria is wrapped within the aggregated nanomaterials [59]. Fourth, oxidative stress [60]. Fifth, interruption in the glycolysis process of the bacteria [61]. Sixth, DNA damaging [62]. Following, Zn ion release [63]. Last, contribution in generation/ explosion of nanobubbles [64]. These mechanistic pathways play a key role for optimization of the drug-delivery system in the medical field.

3.7 Antifungal activity of ZnCr$_2$O$_4$ NRs

The fungi *C. albicans*, *A. niger*, *A. clavatus*, *T. mentographye*, and *E. floccosum* were used as fungal strains for the antifungal screening of ZnCr$_2$O$_4$ NRs. The results of antifungal screening of ZnCr$_2$O$_4$ NRs are summarized in Table 4. The simple sol-gel auto combustion approach mediated ZnCr$_2$O$_4$ NRs exhibited moderate activity against *A. clavatus*, *T. mentographye*, and *A. niger* while showing excellent activity against *C. albicans* (250 µg/ml) and *T. mentographyes* (50 µg/ml). The inhibitory effect of fungus by ZnCr$_2$O$_4$ NRs is caused by a direct interaction between NPs and cell surfaces that also alters the permeability of membranes through which NPs enter and cause cell damage in fungus cells, resulting in cell growth inhibition and ultimately cell death (Fig. 9) [65].

### Table 3

ZOI (mm) of as-synthesized ZnCr$_2$O$_4$ NRs against selected pathogenic strains

| Test pathogens | ZOI (mm) of ZnCr$_2$O$_4$ NRs (µg/ml) | Control (ampicillin) |
|----------------|---------------------------------------|----------------------|
|                | 25 50 100 250 500                      |                      |
| *E. coli*      | 12 17 19 22 23                        |                      |
| *K. pneumonia* | 13 17 19 22 23                        |                      |
| *P. aeruginosa*| 13 17 19 22 23                        |                      |
| *P. vulgaris*  | 12 17 19 22 23                        |                      |
| *S. typhi*     | 12 17 19 22 23                        |                      |
| *S. pyogenus*  | 13 17 19 22 23                        |                      |
| *S. aureus*    | 13 17 19 22 23                        |                      |
| *B. subtilis*  | 13 17 19 22 23                        |                      |
| *S. pneumoniae*| 12 17 19 22 23                        |                      |
| *S. epidermidis*| 14 17 20 21 19                       |                      |

### Table 4

MIC of the as-prepared ZnCr$_2$O$_4$ NRs against fungal pathogens

| Fungal test strains | MIC (µg/ml) of ZnCr$_2$O$_4$ NRs | MIC (µg/ml) of Reference drug |
|---------------------|----------------------------------|------------------------------|
| *C. albicans*       | 250 500                          |                              |
| *A. clavatus*       | 100 100                          |                              |
| *A. niger*          | 100 100                          |                              |
| *E. floccosum*      | 100 100                          |                              |
| *T. mentographyes*  | 50 100                           |                              |

Fig. 9 Plausible mechanism of antifungal activity of ZnCr$_2$O$_4$ NRs


3.8 Antimalarial activity of ZnCr₂O₄ NRs

The synthesized ZnCr₂O₄ NRs were assessed for their antimalarial effect against *Plasmodium falciparum* by measuring the MIC (µg/mL), as shown in Table 5. Thus, the synthesized ZnCr₂O₄ NRs showed considerable antimalarial performance and may play a crucial role in the upcoming biomedical field.

| No. | Compound name               | IC₅₀ value (µg/mL) |
|-----|-----------------------------|-------------------|
| 1   | ZnCr₂O₄ NRs                 | 0.68              |
| 2   | Quinine (Reference drug)    | 0.26              |
| 3   | Chloroquine (Reference drug)| 0.02              |

4 Conclusion

In this study, ZnCr₂O₄ NRs were synthesized using the simple sol-gel auto combustion approach. The sample was only required to sinter at 500 °C for 2 h to obtain the crystalline single phase, XRD, FESEM, and HRTEM studies confirmed the spinel and hexagonal-shaped ZnCr₂O₄ NRs. Also, the bandgap energy of ZnCr₂O₄ NRs was estimated by the UVDRS study in the UV-visible region and was determined to be 3.45 eV. Moreover, as-prepared ZnCr₂O₄ NRs exhibited potent antimicrobial activity against bacterial and fungal strains. We also depicted and discussed the plausible antimicrobial mechanisms for ZnCr₂O₄ NRs. Also, ZnCr₂O₄ NRs exhibited considerable antimalarial activity.

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Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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