



Cite this: *Nanoscale*, 2025, **17**, 25426

Recent progress in the synthesis of CuCr_2O_4 nanomaterials and their composites for catalytic, energy, and biomedical applications: a state-of-the-art-review

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Copper chromite nanoparticles (CuCr_2O_4 NPs) are in the spotlight of modern nanoscience due to their versatile applications across various scientific and industrial domains. This review addresses the synthesis of CuCr_2O_4 NPs, metal-doped CuCr_2O_4 NPs, and nanocomposites (NCs), emphasizing the key parameters, viz., reaction time, pH, temperature, precursor concentration, and the choice of fuel or surfactant, influencing their morphology, crystallinity, and functional properties. CuCr_2O_4 NPs are fabricated using several chemical methods, namely, sol-gel, co-precipitation, hydrothermal, solution combustion, and ball-milling, with different types of fuels or surfactants serving as complexing agents. These materials exhibit remarkable thermal, electrical, and catalytic characteristics. The discussion extends to their broad spectrum of applications, including biological activities such as antimicrobial, anticancer, and bone regeneration as well as their genotoxic impact. We also explore their role in photocatalysis, heterogeneous catalysis, and electrochemical applications, including supercapacitors and lithium-ion batteries. The emerging utility of CuCr_2O_4 NPs in sensor development is also highlighted. This review covers the recent advancements in the synthesis of CuCr_2O_4 NPs, highlighting their key challenges and future directions. Analyzing the structure–property relationships and the influence of synthesis parameters on their performance provides valuable insights into optimizing the design and functionalization of CuCr_2O_4 NPs for specific applications.

Received 21st July 2025,
Accepted 12th September 2025

DOI: 10.1039/d5nr03084f

rsc.li/nanoscale

1. Introduction

In recent years, nanotechnology has emerged in the limelight through its diverse applicability in various fields, ushering in what can be called a “nano-era”. Richard Feynman’s (1959)

talk “There is plenty of room at the bottom” and the invention of the scanning tunneling microscope (1981) paved the way for the development of nanoscience and nanotechnology. Japanese scientist Norio Taniguchi first used the term “nanotechnology” to describe semiconductor processes at the nanoscale about fifteen years after Feynman’s discussion. They promoted the idea that materials may be processed, separated, consolidated, and deformed by a single atom or molecule as part of nanotechnology.¹

In the present scenario, there has been considerable interest in advancing spinel oxide nanoparticles (NPs) across various disciplines of study. Their beneficial traits, including affordability, enhanced efficiency, effortless recuperation, and superior recyclability, suggest that they are potent catalysts.^{2–5} The remarkable catalytic activity of spinel oxides makes them noteworthy. Spinel oxides are inexpensive, harmless, and extremely stable materials with good acid and alkali resistance and high melting temperatures, and their NPs have a comparatively large surface area. Due to these qualities, they can be utilized as solid heterogeneous catalysts in organic chemical

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reactions.^{6–8} The most common type of spinel chromite is binary transition metal oxides, $M^{2+}B_2^{3+}O_4^{2-}$, where $M = Mg^{2+}$, Mn^{2+} , Co^{2+} , Ni^{2+} , Cu^{2+} , or Zn^{2+} . Spinel nanostructures have garnered attention due to their diverse applications in various fields, including nanomedicine, photocatalysis, catalysis, energy storage, lithium-ion batteries, and sensors.^{2,9–11} $CuCr_2O_4$ is a fascinating chromate spinel due to its cost-effectiveness and non-toxicity. As a tetragonally deformed normal spinel, $CuCr_2O_4$ has an axial ratio (c/a) of less than 1, suggesting that its c -axis is compressed relative to its a -axis.⁷ Cr^{3+} ions occupy the octahedral (B) sites, while Cu^{2+} ions are positioned at the tetrahedral (M) sites, and the perfect crystal is anticipated to be an insulator. At normal and high temperatures, spinel $CuCr_2O_4$ crystallizes into a cubic phase and tetragonal phase, respectively. Its first-order phase transition occurs at approximately 854 K (upon heating).^{12,13} According to the Jahn-Teller phenomenon, spinel oxides containing copper have excellent thermal stability.¹⁴

Numerous approaches have been documented in the literature for preparing $CuCr_2O_4$ NPs, including hydrothermal,¹⁵ coprecipitation,¹⁶ sol-gel,¹⁷ solution combustion,⁸ microwave-assisted,¹⁸ and mechanical ball milling.¹⁹ Various fuels and surfactants, such as ethylene glycol, CTAB, tartaric acid, citric acid, glycine, and glucose, have been utilized as capping agents during the synthesis process. These agents are crucial in the synthesis of NPs, given that they enhance the monodispersity, reduce the particle size, and minimize the aggregation of nanoparticles. Surfactants reduce the surface tension of the solution, lowering the energy required to create a new phase from the precursor ions.²⁰

$CuCr_2O_4$ has been identified as a p-type semiconductor ($E_g = 1.40$ eV) for a range of photocatalytic applications.^{15,21–27} Materials known as catalysts accelerate chemical reactions without being consumed by the reaction; however, they are frequently difficult to separate and recover.^{28–30} A green and cost-effective catalyst should have minimal preparation costs, high activity, selectivity, stability, ease of separation, and recyclability.^{31–33} Surprisingly, $CuCr_2O_4$ NPs function as a heterogeneous catalyst in several reactions, such as the thermal disintegration of ammonium perchlorate (AP),³⁴ oxidation of aniline to azoxybenzene,³⁵ preparation of pyrazine,³⁶ synthesis of triarylamine,¹⁶ oxidation of benzene to phenol,^{15,24} hydrogenation of 2-furfuraldehyde,³⁷ and synthesis of biscoumarin and pyrano[*c*]chromene,³⁸ and this catalyst effectively catalyzes toluene oxidation by interfering with C–H bond activation³⁹ and synthesis of [1]benzopyran azo dyes, *etc.*⁴⁰

Intriguingly, $CuCr_2O_4$ nanomaterials/composites show a broad spectrum of biological activities, including anticancer,⁴¹ antibacterial,^{42,43} bone regeneration,⁴⁴ hemolysis,⁴¹ and genotoxicity tests,⁴⁵ with significant potential. Furthermore, $CuCr_2O_4$ nanomaterials and composites have been studied for energy storage applications. The development of high-energy and high-power batteries,⁴⁶ as well as hybrid supercapacitor electrodes due to their capacitive characteristics, cyclic stability, and high power density,⁴⁷ is crucial for meeting energy

storage needs. Rechargeable metal–air batteries based on oxygen electrocatalysis are a viable alternative, particularly when the energy source is intermittent, such as solar or wind. These batteries use bifunctional oxygen electrocatalysis, which includes both oxygen reduction (ORR) and oxygen evolution processes (OER).⁴⁸ Also, $CuCr_2O_4$ has potential as an electrocatalyst in electrochemical hydrogen storage systems, providing a sustainable solution to renewable energy technology.⁴⁹

This review thoroughly examined $CuCr_2O_4$ NPs. The current literature was gathered from Google Scholar, ResearchGate, PubMed, Scopus, and books using the search terms “synthesis + $CuCr_2O_4$ NPs/doped metal/nanocomposites + energy storage + biological + catalytic/photocatalytic applications”. Fig. 1 shows a pie chart of the data received from these keywords. A significant amount of studies has been done on the synthesis and characterization of $CuCr_2O_4$ NPs that can be applied in a variety of scientific and engineering domains. The current summary will help researchers better understand the opportunities and challenges associated with the production of $CuCr_2O_4$ NPs.

2. Synthesis of $CuCr_2O_4$ NPs

The growth and production processes of $CuCr_2O_4$ NPs determine their structure and shape. To date, $CuCr_2O_4$ NPs have been synthesized using several processes, such as sol-gel, coprecipitation, solution combustion, precipitation, hydrothermal, microwave-assisted procedures, and mechanical ball milling, which are summarized in Table 1.

2.1. Pristine $CuCr_2O_4$ NPs

2.1.1. Sol-gel method. The sol-gel approach effectively produces very uniform NPs without any foreign material or co-

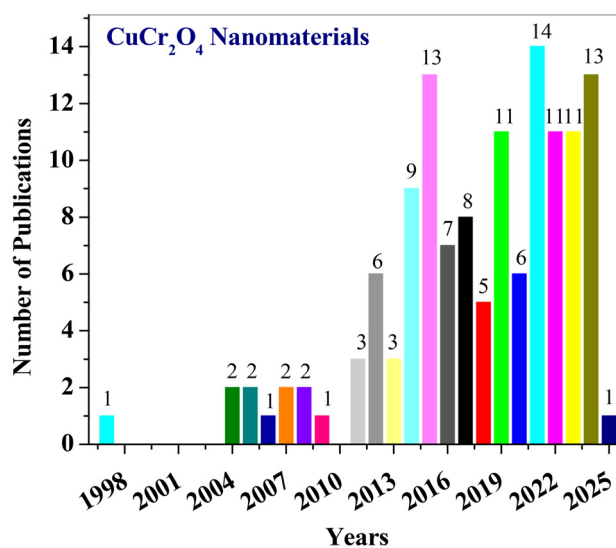


Fig. 1 Data of the number of papers published on “synthesis of $CuCr_2O_4$ nanomaterials and their applications” from Google Scholar, ResearchGate, PubMed, Scopus, and Book database.

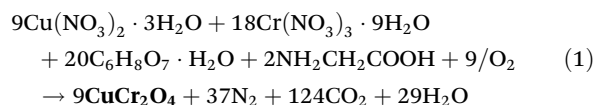
Table 1 Summary of the synthesis methods, characterization techniques, morphology, and applications of CuCr₂O₄ NPs

Synthesis method	Morphology	Particle size (nm)	Characterization techniques	Applications	Ref.
Sol-gel combustion	Laminate, octahedral, and spherical	—	XRD, UV-Vis, FTIR, DSC, TEM, SEM-EDX, ATR	Selective absorber coatings	17
Sol-gel	Spherical	30–55	XRD, FESEM, TEM, TGA-DSC	Catalytic activity	50
Sol-gel	Semi spherical	37	XRD, UV-vis-DRS, FTIR, SEM	Photocatalytic activity	51
Green synthesis	Spherical	54	FT-IR, XRD, BET, FESEM-EDS	Electrochemical hydrogen storage	49
Sol-gel	Quasi-spherical	20–60	XRD, UV-Vis, FTIR, TEM, SEM, RSR, MEM	Photocatalytic activity	52
	Uniform	850 °C < 100	XRD, FTIR, SEM, VSM	Microwave absorber	53
	Poly tetragonal	—	XRD, FTIR, SEM	Antibacterial activity	42
	Spherical	33	UV-Vis-DRS, FESEM, XRD, FTIR	—	55
	Cubic	20–60	XRD, FTIR, SEM, TGA/DTA	Catalytic activity	56
	—	34	XRD, XPS, SEM, HRTEM-SAED	Sensor	60
	—	20–60	XRD, TEM, TG-DTA	—	61
	Octahedral	20–30	XRD, FESEM, EDAX, TG-DTG	—	62
Pechini	—	500 °C-30 700 °C-100	XRD, TG-DTA, TEM-SAED	Catalytic combustion	57
Pechini & modified Pechini	—	650 °C-200	XRD, SEM	—	58
Sol-gel self-combustion	Tetragonal	82.56	XRD, FESEM, EDS, HRTEM-SAED, XPS, GIS	Electrical conductivity	59
Solution combustion self-combustion	Spherical	200 and 225	XRD, TGA-DTA, SEM, BET, XPS	Catalytic activity	8
Combustion	Irregular	25.16	UV-VIS-NIR, XRD, BET, SEM	—	63
	Spherical	CuCr ₂ O ₄ -35–40 NiCuCr ₂ O ₄ -38–46	XRD, FTIR, BET, HRSEM, HRTEM, GC-MS	Polypropylene pyrolysis oil into diesel	66
	Spherical	135.2	XRD, FTIR, TEM, SEM, EDAX, TGA-DTA	Catalytic activity	65
	Spherical	—	XRD, UV-vis-DRS, FTIR, and SEM-EDX	Photocatalytic activity	64
	—	192–300	XRD, FTIR, FESEM, ¹ H, ¹³ C NMR	—	87
	Spherical	20–30	XRD, IR, SEM	Catalytic activity	16
	Spherical	13.1	SEM, TEM, DLLS	Catalytic activity	69
	Spherical	Uniform	XRD, FTIR, SEM, ¹ H, ¹³ C NMR	Catalytic activity	67
	Spherical	—	XRD, SEM, TGA-DTA	Photocatalytic activity	71
	Octahedral	22–28	XRD, FTIR, SEM, ¹ H, ¹³ C NMR	Catalytic activity	68
	Spherical	26–30	XRD, TEM, Raman spectroscopy, X-ray microanalyses	Catalytic activity	92
	Spherical and cubic	—	XRD, SEM, ¹ H and ¹³ C NMR	Catalytic activity	38
	Irregular	20–30	XRD, SEM, ¹ H and ¹³ C NMR	Photocatalytic activity	23
	Quasi-spherical	600 °C-15.1 700 °C-21.7	XRD, TEM, BET	—	72
	Spherical	48	XRD, SEM, UV-vis, TG-DSC	Dye sensitized solar cells	70
	Spherical	520 °C-72	XRD, FTIR, FESEM, TG-DSC	—	15
	Almost uniform	20–45	XRD, XPS, ICP-AES, SEM, TEM, TGA	Catalytic activity	74
	Almost uniform (25–50 nm)	CuCr ₂ O ₄ fresh-35, CuCr ₂ O ₄ spent after five reuses-39	XRD, ICP-AES, XPS, EXAFS, SEM, TEM, TGA	Catalytic activity	75
	Uniform	20–60	XRD, ICP-AES, XPS, FTIR, SEM, TEM, TGA	Catalytic activity	35
	Almost uniform (25–50 nm)	CuCr ₂ O ₄ fresh-35, CuCr ₂ O ₄ spent after five reuses-36	XRD, XPS, SEM, TEM, TGA, ICP-AES	Catalytic activity	15
	Spherical	20–40	XRD, XPS, ICP-AES, SEM, TEM, TGA	Photocatalytic activity	22
	Irregular-500 °C Cubic-600–800 °C	—	XRD, UV-vis-DRS, TEM, TG-DSC, XPS, zeta potential	Supercapacitor	47
	Uniform	20	XRD, FTIR, SEM, TEM	Catalytic activity	79
	Spherical	12	XRD, SEM, TEM	Catalytic activity	76
	Almost uniform	30–60	XRD, FTIR, SEM-EDAX, TEM, XPS	Catalytic activity	40
	Sphere	23	XRD, FTIR, SEM, EDS, ¹ H, ¹³ C NMR	Catalytic activity	77
	—	7.9–23.9	XRD, FTIR, SEM, TEM, TPR, TGA, BET	Catalytic activity	77

Table 1 (Contd.)

Synthesis method	Morphology	Particle size (nm)	Characterization techniques	Applications	Ref.
Uniform	Uniform	20–40	XRD, ICP-AES, XPS, TPR, BET, SEM, TEM, EXAFS	Catalytic activity	39
Uniform	Uniform	CuCr ₂ O ₄ -35 Mn _{0.5} Cu _{0.5} Cr ₂ O ₄ -90	XRD, FTIR, UV-Vis, FESEM, TGA	Photocatalytic activity	80
Uniform	Uniform	20–50	XRD, ICP-AES, FTIR, SEM, TEM, TGA, XPS	Catalytic activity	78
—	—	45	XRD, TEM, SEM, EDAX	Catalytic activity	93
800 °C-spherical	800 °C-spherical	100–300	XRD, SEM-EDX	Solar absorber coatings	14
Irregular and spherical	Irregular and spherical	CuCr ₂ O ₄ -30.17 CuCr ₂ O ₄ -citric acid-2.6.3.8, CuCr ₂ O ₄ -tartaric acid-13.13 nm	XRD, FT-IR, UV-Vis, FE-SEM, HR-TEM, BET	Photocatalytic activity	81
—	—	30	XRD, FTIR, SEM, TEM, BET, TGA/DSC	Catalytic activity	84
Mechanical ball milling	Spherical	—	XRD, FTIR, SEM, EDS, DSC-TG-DTG	Catalytic activity	85
Ultrasonic dispersion method and a mechanical grinding	Flower	21–46	XRD, UV-Vis, IR, FESEM, HRTEM	—	18
Microwave assisted co-precipitation	Spherical	Citric acid-2.3, oleic acid-8.43 Coconut oil-9.62	XRD, FTIR, SEM-EDS	—	82
Microwave	—	Urea-15.48	—	—	—
—	Hexagonal	41–25	UV-vis, XRD, FTIE, SEM, TEM	Dye-sensitized solar cell	83
—	Irregular plate	37–26	XRD, SEM	Antibacterial and electrical conductivity	43
Microwave combustion	—	500 °C-30–40 800 °C-100	XRD, SEM, AFM, TEM	—	89
Sputtering	—	40	XRD, SEM, TEM, DSC/TG	Catalytic activity	90
Wet chemical grinding	Semi-spherical	41.51	XRD, FT-IR, EDX, SEM, zeta potential	Anti-breast cancer activity	41
Co-precipitation, ultrasound, and green chemistry	—	—	—	—	—

crystals. It is known for its simplicity, high yield, and controllability. Pakzad *et al.* synthesized CuCr₂O₄ pigment using a sol-gel combustion-calcination process. In this investigation, they employed Cu and Cr nitrate ions as oxidizers, using a mixture of fuels, such as glycine and citric acid, with low and high fuel ratios, respectively (eqn (1)).¹⁷



Hosseini *et al.* produced CuCr₂O₄ NPs using a temperature-programmed procedure after a sol-gel method in the presence of urea, and the resulting particles were homogeneous, well-separated, and had a sphere-like morphology.⁵⁰ In another study, the researchers used sucrose as a template in the synthesis process and compared the template-assisted and template-free approaches.⁵¹ Lachini *et al.* manufactured CuCr₂O₄ NPs using two different methods, sol-gel synthesis with stearic acid and green synthesis using *Aloe vera*. Researchers successfully investigated the performance of these materials as electrocatalysts in electrochemical hydrogen storage.⁴⁹ Benrighi *et al.* found that CuCr₂O₄ NPs have a spinel structure with an orthorhombic phase, specifically belonging to the *Fddd* space group.⁵²

Peymanfar *et al.* investigated the microwave absorption properties of NPs produced using silicone rubber as a polymeric matrix. In the first study, they investigated a CuCr₂O₄/silicone rubber nanocomposite, which has a microwave absorption of 48.56 decibels (dB) at 10.9 GHz, a thickness of 2.6 mm, and more than 92.99% microwave absorption along the X-band frequency.⁵³ In the second investigation, they created CuCr₂O₄/poly(vinylidene fluoride) (PVDF) nanocomposites. They achieved a maximal reflection loss of –65.57 dB at 14 GHz with a thickness of 2 mm, and absorbed a bandwidth of 7.73 GHz with more than 10 dB absorption. CuCr₂O₄ NPs play a promising function in improving microwave absorption properties in polymeric matrices.⁵⁴ As discussed in a previous report, Akbar Mirzai and team synthesized CuCr₂O₄ NPs using glucose and created antibacterial films using PVDF and silicone rubber.⁴²

Habibi *et al.* employed the citrate sol-gel synthesis method to synthesize CuCr₂O₄ NPs at room temperature using a fuel. The XRD data from the samples indicated the development of the CuCr₂O₄ nanocomposite at a calcination temperature of 700 °C.⁵⁵ Wang *et al.* synthesized nanochromates MCr₂O₄ (M = Co, Ni, Cu, and Zn) using the sol-gel technique. The structural and morphological traits were disclosed employing XRD and SEM.⁵⁶ The Pechini method involves dissolving Cu and Cr salts, and then adding citric acid to produce a homogeneous solution. This mixture was heated at 95 °C for many hours (h) to evaporate the water, yielding dark brown, clear, viscous gels. After that, the gel was dried at 160 °C for 2 h, resulting in frothy dark powders known as the precursor to CuCr₂O₄/CuO nanocomposites.⁵⁷ Additionally, Billman *et al.* manufactured CuCr₂O₄ NPs utilizing two nitrate-citrate combustion syn-

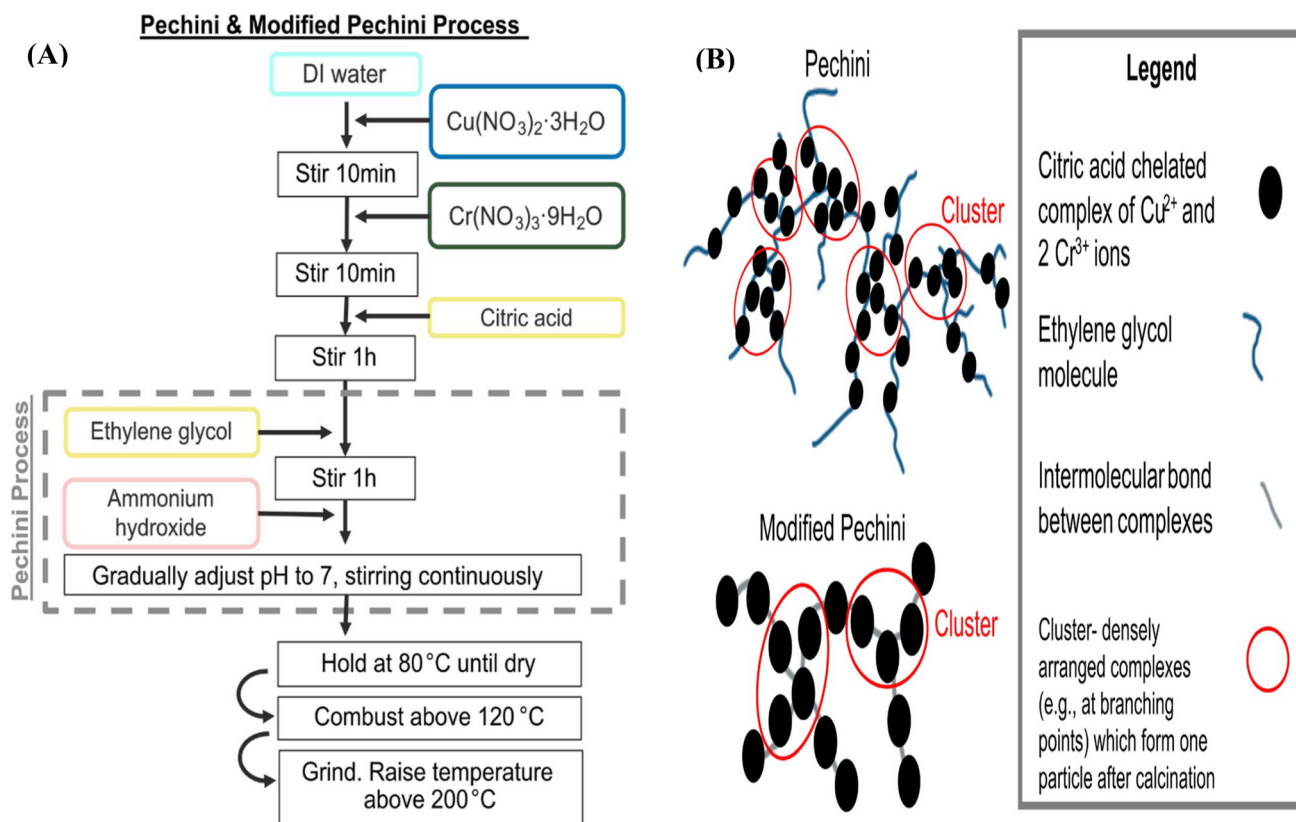


Fig. 2 (A) Diagram of Pechini and modified Pechini method and (B) graphic showing the differences between the two methods for producing gel structures from chelated complexes.⁵⁸

thesis sol-gel methods, as indicated in Fig. 2A, *i.e.*, one with ethylene glycol and neutralized with ammonium hydroxide (Pechini technique (Pechini, 1967)) and one without (modified Pechini method). The Pechini technique yields a broader particle size distribution, characterized by generally smaller and more scattered agglomerates, as shown in Fig. 2B, which exhibits a more branching structure with larger cluster sizes.⁵⁸

Javed *et al.* conducted a detailed analysis of the physical characteristics of CuCr_2O_4 NPs. For the study of their structural attributes, a schematic representation was generated using the VESTA program (Fig. 3). CuCr_2O_4 NPs have a single-phase, tetragonally distorted normal spinel structure with an *I41/amd* space group in the crystal lattice. In the CuO_4 tetrahedral geometry, Cu(II) bonds have shorter lengths and angles compared to Cr(III) bonds in the CrO_6 octahedron. The TEM images revealed *d*-spacing values of 0.27310 nm. XPS analysis revealed the presence of several electrical routes in the CuCr_2O_4 spinel system, including $\text{Cu}^{2+}-\text{O}^{2-}-\text{Cr}^{2+}$, $\text{Cr}^{3+}-\text{O}^{2-}-\text{Cr}^{3+}$, $\text{Cr}^{6+}-\text{O}^{2-}-\text{Cr}^{6+}$, and $\text{Cr}^{3+}-\text{O}^{2-}-\text{Cr}^{6+}$.⁵⁹

Kong *et al.* reported that the CuCr_2O_4 material has a pore structure that allows rapid analyte transport and generates well-dispersed, small-sized NPs with a minimal polyhedral content.⁶⁰ Cui *et al.* created various types of spinel NPs, including CoAl_2O_4 (blue), CoCr_2O_4 (bluish green), ZnFe_2O_4 (brown), and CuCr_2O_4 (black), employing propylene oxide as a

gelation agent *via* the sol-gel technique, which reduces the required calcination temperature and minimizes particle agglomeration.⁶¹ Habibi *et al.* investigated the decomposition mechanism of $\text{Cu(NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and $\text{Cr(NO}_3)_3 \cdot 9\text{H}_2\text{O}$ precursors, as well as the copper chromite xerogel precursor, using thermal analysis. The results revealed that mass loss for copper(II) nitrate and chromium(III) nitrate occurred at 258 °C and 140 °C, respectively. In comparison, the major mass loss for the dried copper chromite gel occurred at 310 °C, confirming the decomposition of nitrates and the phase transformation leading to the complete crystallization of CuCr_2O_4 .⁶²

2.1.2. Solution combustion method. Studies found that the combustion reaction is an excellent chemical approach for synthesizing ceramic oxide spinels (MCr_2O_4). Compared to conventional methods, the solution combustion method enables the synthesis of CuCr_2O_4 NPs with smaller particle sizes and a larger surface area even before calcination.⁸ Barros and colleagues successfully produced chromium spinel MCr_2O_4 ($\text{M} = \text{Zn, Co, Cu, and Ni}$) utilizing urea as fuel.⁶³ Paloma de Jesus Cubas and research team created CuCr_2O_4 NPs employing a self-combustion approach, which is a quick, easy, and effective technique. The synthesized NPs were analyzed, and the findings demonstrate their good purity, homogeneity, and excellent degrading capacity under visible

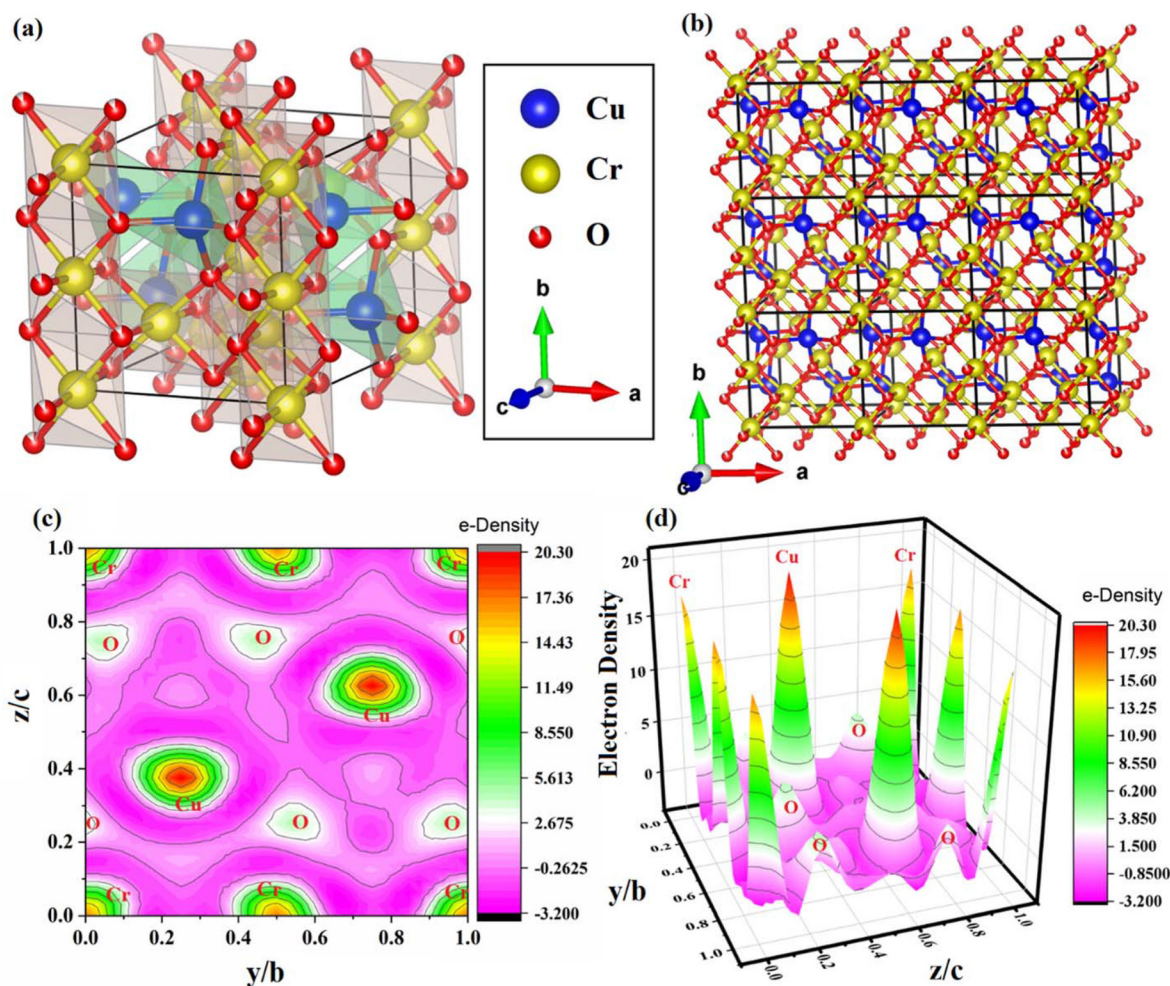


Fig. 3 Schematic of the tetragonally distorted CuCr_2O_4 : (a) unit cell, (b) extended crystal structure, and (c) 2D and (d) 3D electron density maps.⁵⁹

light.⁶⁴ Surprisingly, Viswanath *et al.* synthesized an alginate mixed metal oxide complex. A sodium alginate solution was gradually mixed into a Cu and Cr nitrate solution, forming equal-sized beads. The generated beads were soaked for 24 h. After that, Cu and Cr ions were absorbed into the core of the alginate beads, resulting in a mixture of Cu and Cr alginate. After 24 h, the beads were filtered and vacuum-dried. Finally, the material was calcined at 750–800 °C for 3 h.⁶⁵ Tamizhdurai and research team synthesized undoped and doped Ni- CuCr_2O_4 NPs. They studied the hydro processing of polypropylene pyrolysis oil into diesel fuel.⁶⁶

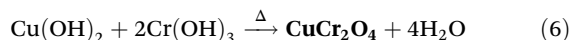
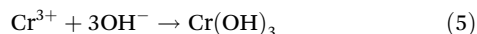
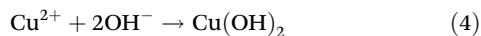
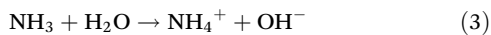
2.1.3. Co-precipitation method. In the co-precipitation approach, authors used sodium acetate (NAOAc) as the precipitating agent and adjusted the pH to 10. After the reaction mixture was maintained at 60 °C, it was heated to 80 °C, at which point precipitation began. Afterward, the sample was calcined, yielding 86% of synthesized CuCr_2O_4 NPs.^{16,38,67–69} The inverse co-precipitation approach was used in the work by Tavakoli and Mamoori to create CuCr_2O_4 NPs at different calcination temperatures of 400 °C, 520 °C, and 800 °C. According to the XRD data, the sample calcined at 400 °C

exhibited amorphous character. After being calcined for 2 h at 520 °C, pure-phase CuCr_2O_4 was produced. The enhanced crystallinity of the NPs was demonstrated by a gradual rise in the intensity of all the diffraction peaks and the narrowing of the peak widths at 800 °C.⁷⁰

Pan *et al.* described the synthesis process of CuCr_2O_4 NPs. They used the appropriate mixed amount of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$, along with $(\text{CH}_2)_6\text{N}_4$ and NH_4HCO_3 , and then agitated for 1 h. After aging the mixture for 12 h, the samples were dried in a vacuum at 105 °C for 6 h, and the dry precursor was obtained. After that, the precursor was calcined at 400 °C, 450 °C, 500 °C, and 600 °C for 3 h each.⁷¹ A mono-dispersed CuCr_2O_4 nanopowder was effectively generated using an alcohol-aqueous mixture and applied as photoanodes in dye-sensitized solar cells.⁷²

Paul *et al.* manufactured CuCr_2O_4 NPs utilizing a co-precipitation method with a 1 : 2 molar ratio of Cu and Cr hydroxide precursors. Initially, urea decomposed to NH_3 and CO_2 (eqn (2)). NH_3 reacts with water molecules to form NH_4^+ and OH^- (eqn (3)). The free OH^- ions subsequently mix with Cu^{2+} and Cr^{3+} ions to form $\text{Cu}(\text{OH})_2$ and $\text{Cr}(\text{OH})_3$ (eqn (4) and (5)),

respectively. The sample was calcined to produce CuCr_2O_4 NPs (eqn (6)).²³



2.1.4. Hydrothermal method. The hydrothermal technique crystallizes materials by heating water at high pressure to produce high-quality, composition-controlled crystals. To allow the quick passage of metal ions, the phases that dissolve at their melting points are provided with a homogeneous composition and stability.⁷³ Cu–Cr samples were manufactured utilizing a non-hydrothermal technique, although the particles were not homogeneous. According to a study by Acharyya *et al.*, autogenous pressure during hydrothermal conditions was required to form homogeneous CuCr_2O_4 NPs.⁷⁴ The energy required to form a new phase from the precursor ions is reduced when the surfactant lowers the surface tension. Various studies on CuCr_2O_4 NPs by Acharyya and colleagues have successfully fabricated CuCr_2O_4 NPs using cetyltrimethylammonium bromide (CTAB), a cationic surfactant,^{35,39,75–78} and cetyl alcohol.¹⁵ Yuan *et al.* reported that cubic-like

CuCr_2O_4 particles can be achieved using the hydrothermal approach.²²

Ismael *et al.* produced CuCr_2O_4 NPs *via* a hydrothermal method. Fig. 4 shows how colloidal CuCr_2O_4 was re-dispersed in acetone after being recovered from its production medium using an ultrasonic probe. Ammonium perchlorate (AP) particles, followed by dissolution in acetone and colloidal CuCr_2O_4 NPs, were integrated into the AP matrix using the antisolvent method.⁷⁹

Soleimani *et al.* prepared CuCr_2O_4 and $\text{Mn}_{0.5}\text{Cu}_{0.5}\text{Cr}_2\text{O}_4$ and compared their effectiveness in degrading malachite green with CuCr_2O_4 NPs.⁸⁰ Pleasingly, Acharyya *et al.* reported the 3D raspberry-like morphology of CuCr_2O_4 in one of their studies. Crystal-face attraction, electrostatic fields, van der Waals forces, hydrophobic interactions, hydrogen bonds, and the autogenous pressure in the autoclave influence the creation of the raspberry morphology. The “soft template” CTAB and Ostwald ripening also have a substantial contribution. Cr^{6+} precursors, such as $\text{K}_2\text{Cr}_2\text{O}_7$, play a significant role in forming the raspberry-like structure, but Cr^{3+} precursors, including CrCl_3 and $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, do not. Synthesizing chromium(III) oxide with CTAB, $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$, and hydrazine resulted in a characteristic 3D strawberry-like texture, underlining the significance of Cr^{6+} in self-assembly (Fig. 5).⁷⁸

Rubin *et al.* hydrothermally synthesized CuFeMnO_4 and CuCr_2O_4 NPs, whereas large-batch sintering produced $\text{Cu}_{0.5}\text{Cr}_{1.1}\text{Mn}_{1.4}\text{O}_4$. These samples were annealed at 800 °C for

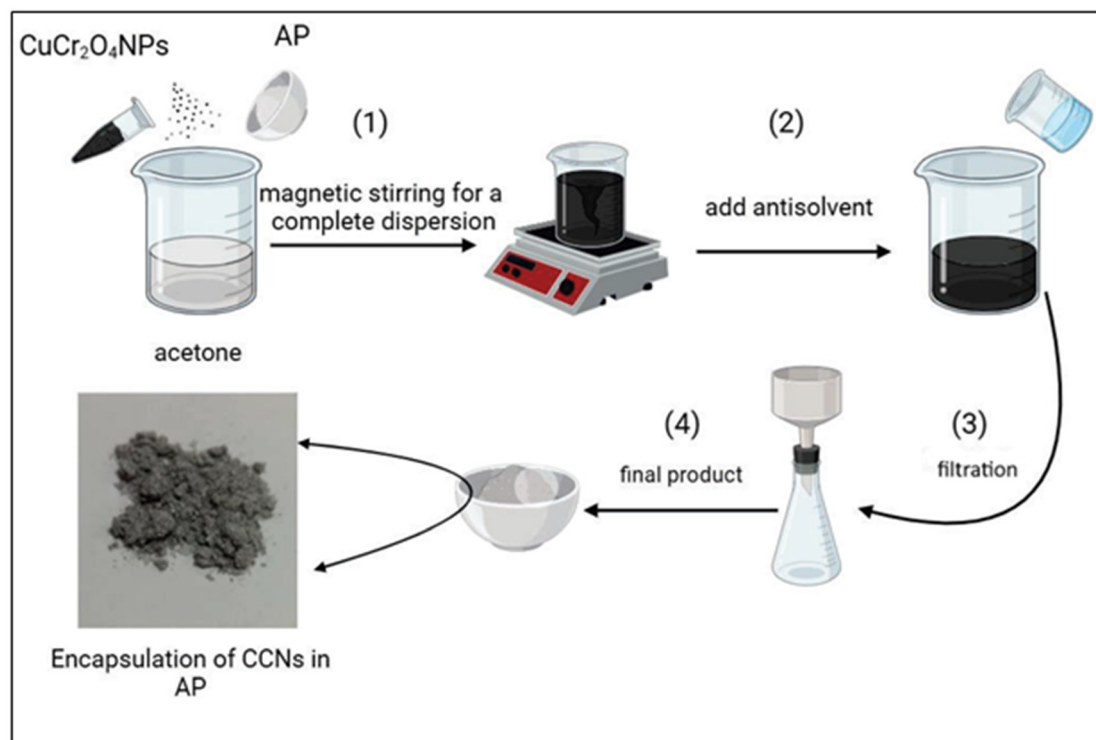


Fig. 4 Integration of CuCr_2O_4 NPs into AP.⁷⁹

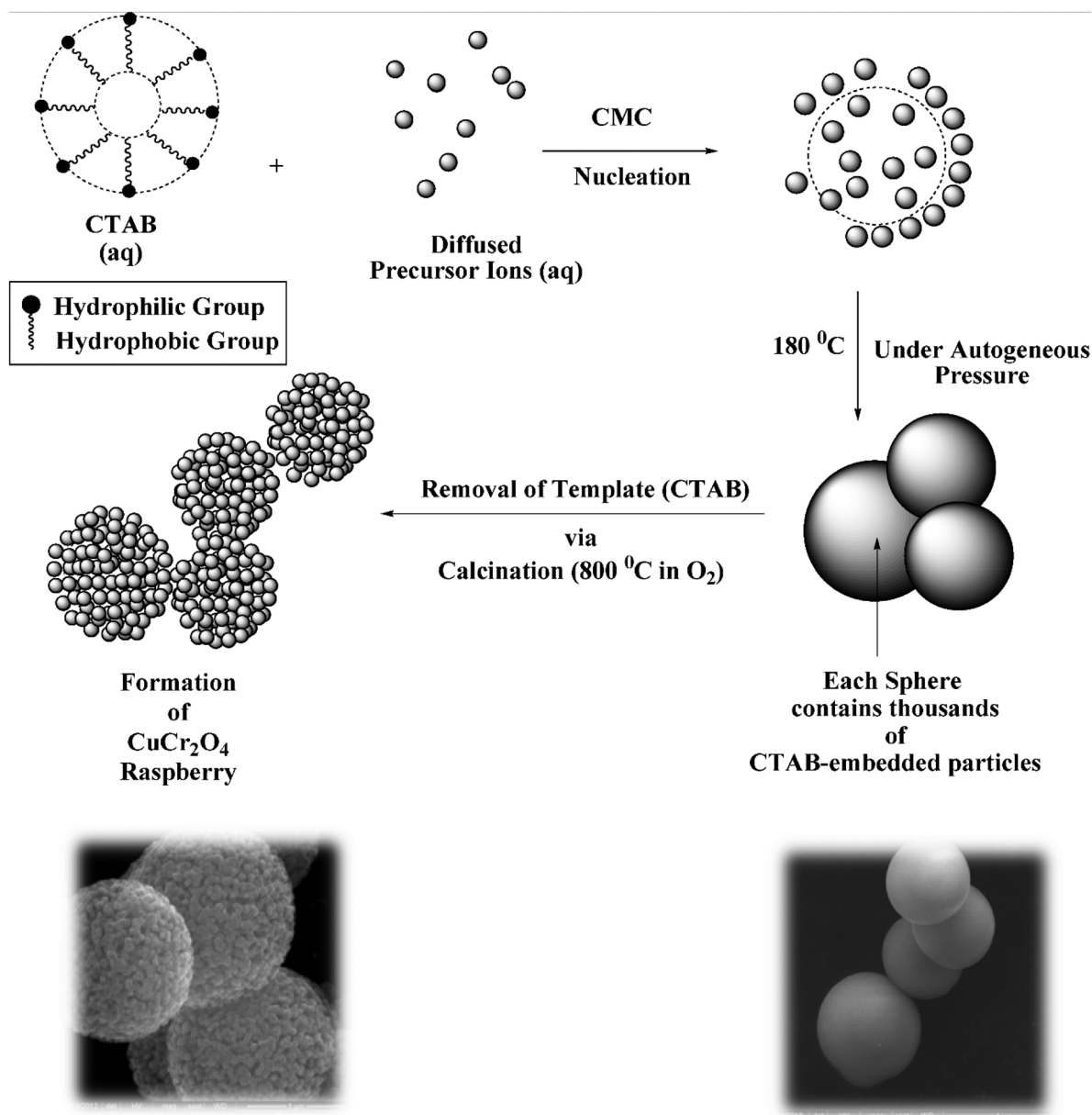


Fig. 5 Schematic of the production of CuCr_2O_4 spinel catalyst with a 3D raspberry-like structure.⁷⁸

various durations, including 100, 300, 1000, and 2000 h. After 2000 h, XRD data verified that the relevant peaks are indeed that of CuCr_2O_4 , and there is no reason to assume a phase transition from CuCr_2O_4 to CuCrO_2 (delafossite).¹⁴ Al-Wasidi *et al.* demonstrated the facile synthesis of CuCr_2O_4 NPs using citric acid and tartaric acid templates, as well as in their absence.⁸¹

2.1.5. Microwave-assisted method. Innovation has made preparation easier using microwave radiation for homogeneous heating. Monika *et al.* manufactured nanocomposites of Cr_2O_3 incorporated with CuO using microwave-assisted coprecipitation. They used NaOH (precipitating agent) and annealed it at $600\text{ }^{\circ}\text{C}$ before pouring 10% chromium oxide and igniting at $200\text{ }^{\circ}\text{C}$, $400\text{ }^{\circ}\text{C}$, and $600\text{ }^{\circ}\text{C}$.¹⁸ The SEM images

of the samples synthesized by microwave energy in a 1 min on-off cycle at 600 W with different capping agents revealed a pyramidal morphology that transformed into a spherical or pseudo-spherical shape as a result of clustering, with inadequate homogeneity due to the large variety of particle sizes.⁸² Rajeswari *et al.* produced pristine CuCr_2O_4 (0.00) and La-doped CuCr_2O_4 (0.01–0.03) using a microwave oven at 2450 MHz.⁸³ Additionally, pristine CuCr_2O_4 and La-doped CuCr_2O_4 were synthesized by Sankudevan *et al.*⁴³

2.1.6. Mechanical ball-milling method. Gou *et al.* synthesized raw CuCr_2O_4 and nano- CuCr_2O_4 using mechanical grinding.¹⁹ The CuCr_2O_4 suspension was prepared using a ball milling process with a mixture of water and ethanol. The suspension was then ground in a 01-HDDM machine at 1200

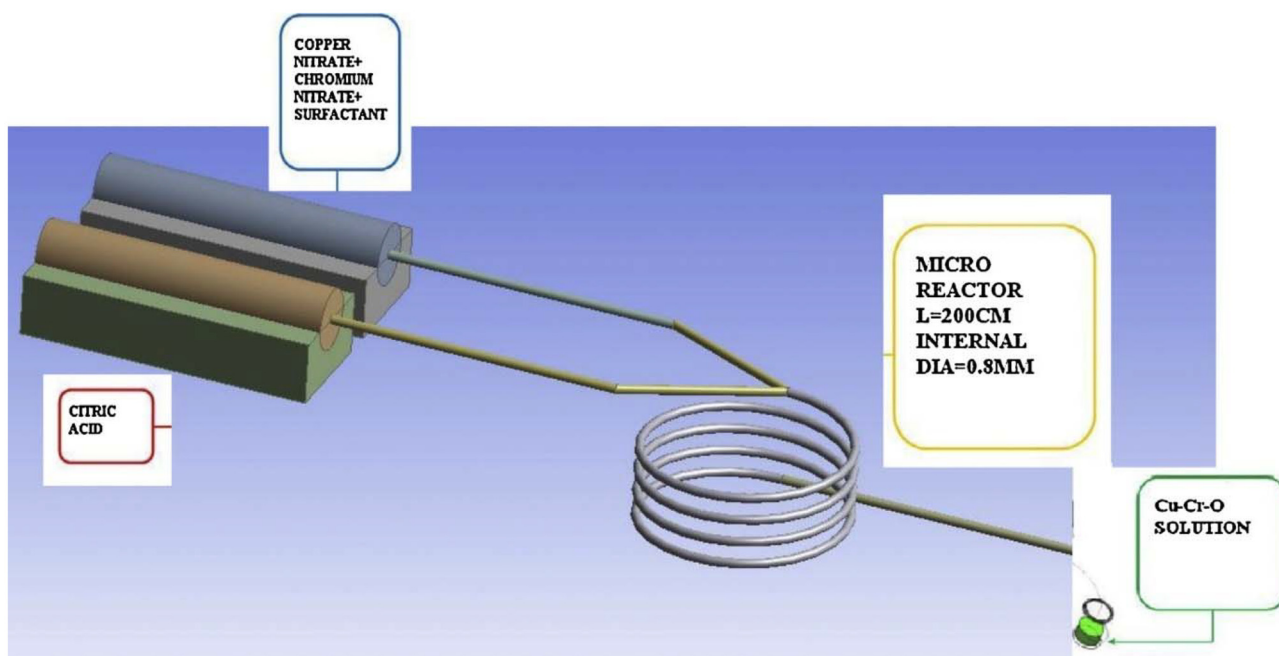


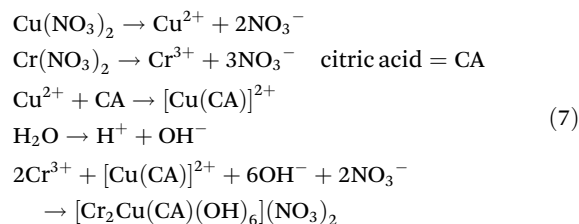
Fig. 6 Illustration of the fabrication of CuCr_2O_4 NPs in a continuous flow microchannel reactor.⁸⁷

rpm for 3 h, maintaining the temperature between 20–30 °C. Finally, the slurry was transferred to a vacuum dryer to obtain nano- CuCr_2O_4 .⁸⁴ Nano- CuCr_2O_4 /ultrafine AP composites were prepared using an ultrasonic dispersion and mechanical ball milling approach by Zhang *et al.*, and varying ball milling periods (30 min, 1, 3, 6, 12, 24, and 48 h) created different dispersions. The researchers examined both methods and determined that nano- CuCr_2O_4 exhibits a better dispersion in ultrafine AP composites made by mechanical ball milling. The dispersion of nano- CuCr_2O_4 on ultrafine AP is improved with longer milling times. The most consistent dispersion of nano- CuCr_2O_4 occurs in ultrafine AP after 6–12 h of milling.⁸⁵

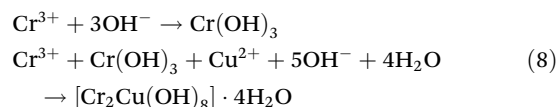
2.1.7. Other methods. Han *et al.* employed the polyacrylamide gel method to synthesize CuAl_2O_4 in the cubic phase, whereas CuCr_2O_4 and CuFe_2O_4 were synthesized in the tetragonal phase. CuAl_2O_4 was produced without impurities, but CuCr_2O_4 and CuFe_2O_4 included CuO impurities. CuAl_2O_4 showed superior photocatalytic activity to the other spinel NPs due to its smaller particle size.⁸⁶

Appalakutti *et al.* fabricated Cu–Cr–O nanocomposites using a continuous flow microreactor. As depicted in Fig. 6, Cu, Cr nitrate, and surfactant (PVP and CTAB) mixtures were filled in a syringe at varying molar ratios. Alternatively, another syringe was filled with a citric acid solution in a 2 : 1 molar ratio relative to the total metal ion concentration. The microreactor was placed in a water bath at 80 °C, with a flow rate ranging from 25 to 150 mL h^{-1} . After that, the nanopowder was generated by calcining at 200 °C for a specific period of 2 h. The hydrolysis and polycondensation reaction (eqn (7)–(10)) mechanisms are provided below.⁸⁷

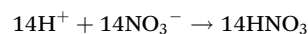
Hydrolysis reaction



(Precursor A)

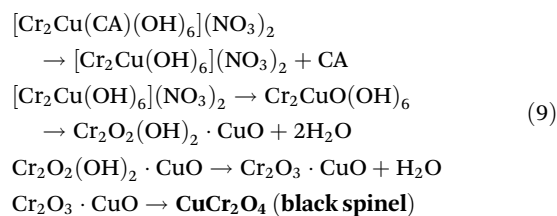


(Precursor B)

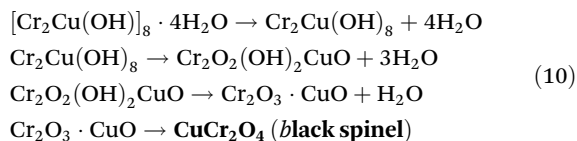


Polycondensation by dehydration

(Precursor A)



(Precursor B)



Interestingly, Chiu and research team used an electro-spinning approach to produce 1-D CuCr_2O_4 fibers.⁸⁸ Tsai and colleagues successfully examined the production of transparent conducting Cu–Cr–O films on fused silica substrates utilizing DC reactive magnetron sputtering with a single equimolar Cu–Cr alloy target and a post-annealing method in a controlled Ar atmosphere at 500–800 °C for 2 h.⁸⁹

The wet mechanical grinding method and the vacuum freeze-drying process present a promising technique for easily producing Cu-based NPs, which have potential applications in the mass preparation of other nanosized catalysts. The Cu-based NPs, including CuO, CuCr_2O_4 , and copper β -resorcyate,

are illustrated in Fig. 7a and b, which detail the synthesis and drying processes, respectively.⁹⁰

Ahmed *et al.* produced CuCr_2O_4 using rosemary (*Rosmarinus officinalis*) leaf extract, employing three techniques including co-precipitation, ultrasonication, and green chemistry. Researchers evaluated its anti-breast cancer efficacy using the MCF-7 cell line, which revealed encouraging results.⁴¹ The CuCr_2O_4 phase develops at a temperature of 750–800 °C for all five mixes. CuO, Cr_2O_3 , and CuCr_2O_4 phases exist in this temperature range; hence, the CuCr_2O_4 phase is produced by reacting the spinel phase with CuO.⁹¹

In conclusion, various physical, chemical, and green synthesis methods have been employed to fabricate CuCr_2O_4 nanomaterials. Among them, the sol-gel method is the most widely used due to its low calcination temperature, excellent chemical homogeneity, and ability to produce monodispersed NPs with a controlled size and morphology. The citric acid-assisted sol-gel technique is particularly effective, given that citric acid functions as a chelating agent, forming homo-

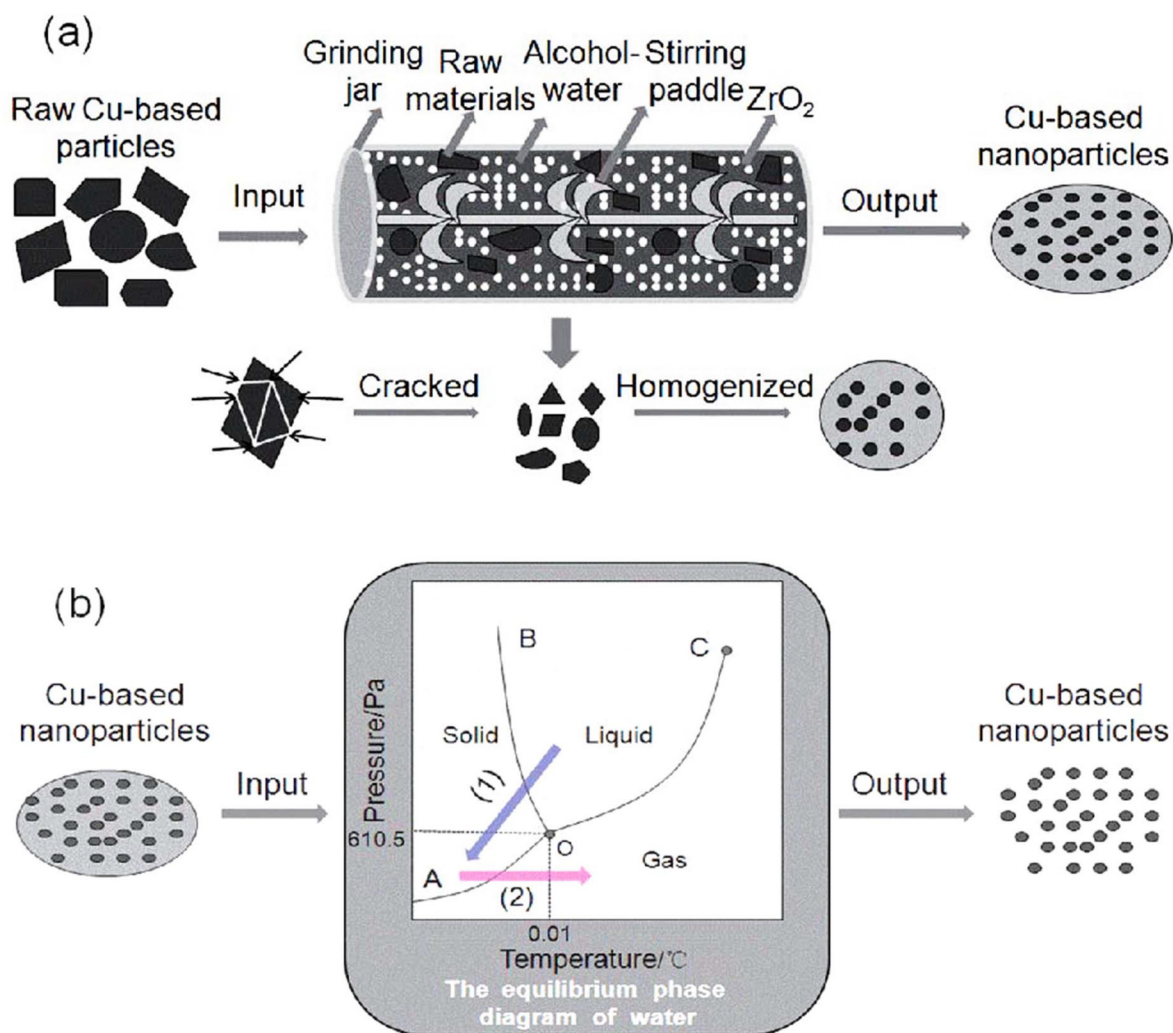


Fig. 7 Diagram of the production of Cu-based NPs: (a) vacuum freeze-drying and (b) wet mechanical grinding.⁹⁰

geneous metal–citrate gels that promote the uniform mixing of metal ions and result in smaller crystallite sizes. According to the literature, this method typically yields nanoparticles with small sizes and spherical morphology, which enhance their surface area and improves their catalytic performance. The improved homogeneity of CuCr_2O_4 synthesized *via* this approach significantly boosts its photocatalytic activity, especially in the degradation of antibiotics, making it more advantageous than conventional synthesis techniques. Additionally, the sol–gel approach facilitates the incorporation of capping agents, such as amino acids, and green alternatives, such as sucrose. These stabilizing agents effectively minimize the agglomeration, enhance the crystallinity, and refine the morphology of the particles. Therefore, the sol–gel method, when combined with suitable chelating and capping agents, provides a versatile, efficient, and environmentally friendly approach for synthesizing high-performance CuCr_2O_4 nanostructures for various applications.

2.2. Synthesis of metal-doped CuCr_2O_4 NPs

Batool *et al.* manufactured Cd- and Nd-doped CuCr_2O_4 NPs ($\text{M}_x\text{Cu}_{1-x}\text{Cr}_2\text{O}_4$) with x values of 0.2, 0.4, 0.6, and 0.8. The band gap of the parent compound was ascertained employing photoluminescence (PL) spectra and found to be 4.96 eV. When elements were doped in the parent compound at varying concentrations, the band gap (E_g) decreased for Nd (2.43 eV) and Cd (2.42 eV). Additionally, microscopic images revealed that the texture of pure CuCr_2O_4 NPs was tetragonal and that doping with other compounds alters the structure according to the characteristics of the NPs.⁹⁴ Anusree *et al.* produced Ba and Co-doped CuCr_2O_4 NPs using a hydrothermal technique and evaluated the catalytic efficacy of the created NPs for the heat breakdown of AP.⁹⁵ These studies, conducted by Acharyya *et al.*, reported the formation of nanoclusters of Cu(II) supported by CuCr_2O_4 NPs. They investigated their catalytic activity in the selective hydroxylation of benzene to phenol,²⁴ the oxyamination of benzene to aniline,⁹⁶ and the selective oxidation of ethanol.⁹⁷

Bai *et al.* reported the synthesis of Fe and Mn-doped CuCr_2O_4 NPs with the formula $\text{CuCr}_{2-x-y}\text{Fe}_x\text{Mn}_y\text{O}_4$. The doped pigments exhibited a higher UV-visible absorbance than CuCr_2O_4 , attributed to electron transfer between $\text{O}^{2-} \rightarrow \text{Fe}^{3+}$, $\text{O}^{2-} \rightarrow \text{Mn}^{3+}$, and d–d intra-atomic transitions of Fe^{3+} and Mn^{3+} . The $\text{CuCr}_{1.93}\text{Fe}_{0.07}\text{O}_4$ black pigment showed a higher NIR solar reflectance ($R^* = 27.7\%$) than the CuCr_2O_4 black pigment ($R^* = 19.0\%$), which was ascribed to the decreased concentration of free carriers.⁹⁸

Yadagiri *et al.* employed the sol–gel method for generating CuCr_2O_4 and Fe-substituted CuCr_2O_4 . XRD analysis of $\text{CuCr}_{2-x}\text{Fe}_x\text{O}_4$ revealed that for $x = 0.0$, the phase is tetragonal, whereas the other three compounds ($x = 0.5, 1.0, \text{ and } 1.5$) have a cubic phase. It was discovered that the average crystalline size increased with Fe substitution.⁹⁹ Ramezanalizadeh *et al.* created La-doped CuCr_2O_4 NPs. The photocatalytic study of doped NPs revealed the outstanding degradation of contaminated dyes.¹⁰⁰ Jagadeesan *et al.* synthesized pure CuCr_2O_4 and

doped $\text{CuCr}_{2-x}\text{Mg}_x\text{O}_4$ ($x = 0.00\text{--}0.1\%, 0.2\%, \text{ and } 0.3\%$) nanocomposites. In short, the Mg-doped sample has a higher specific surface area than the pure CuCr_2O_4 sample.¹⁰¹ Interestingly, Zn-doped CuCr_2O_4 was synthesized at different concentrations and calcined at various temperatures.¹⁰² Interestingly, scientists utilized CuCr_2O_4 black oxide pigment doped with various metals, including Mn, Ni, Co, Al, Zn, and Sn, to create solar-absorbing spinel structures. Among them, the Mn– CuCr_2O_4 pigment exhibited much higher levels of solar absorption than the other metal dopants.¹⁰³

Using a microscopic model and Green's function theory, Apostolov *et al.* theoretically investigated the magnetic, electric, and optical characteristics of both pure and ion-doped CuCr_2O_4 in bulk and nanoparticle forms. Their results showed that the magnetization and band gap energy (E_g) decrease with an increase in the nanoparticle size. Magnetization and polarization (P) peaked when doped with Co ions, while both qualities decreased as the Pr ion concentration increased. Therefore, compared to that substituted with rare-earth elements, Co-doped CuCr_2O_4 NPs are more appropriate for spintronic applications. Alternatively, Pr ion doping raises the E_g , which makes it more suitable for solar cell technology.¹⁰⁴

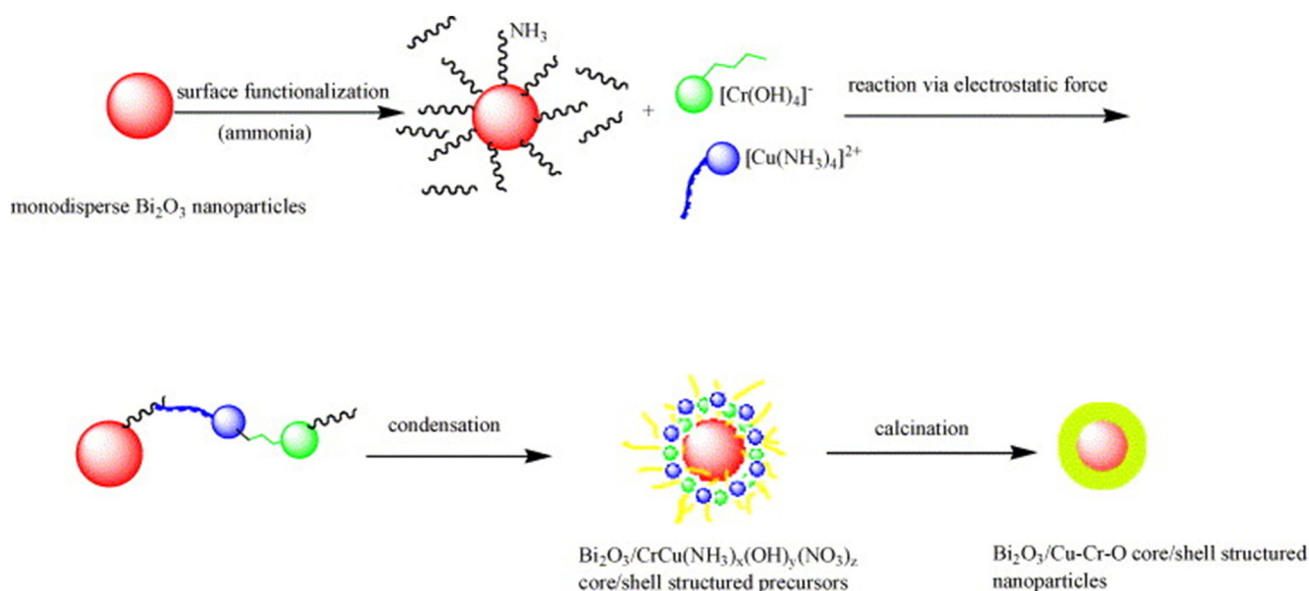
Table 2 summarizes the diverse synthetic approaches, characterization techniques, and applications of metal-doped CuCr_2O_4 NPs.

2.3. Synthesis of CuCr_2O_4 nanocomposites

The interaction of NPs with the matrix and particle–particle interactions are important variables in determining the properties of nanocomposites. Strong interactions between particles and epoxy can enhance the mechanical properties of nanocomposites.^{106–108} Lao *et al.* created CuCr_2O_4 NPs using the sol–gel technique. Then, a suspension was created by diluting the CuCr_2O_4 sample with glucose. $\text{Ag}(\text{NH}_3)_2\text{OH}$ was created by titrating a 0.01 mol L^{-1} AgNO_3 solution from a diluted $\text{NH}_3\cdot\text{H}_2\text{O}$ solution. They combined the suspension with the Ag solution. Then, the mixture was centrifuged and dried, yielding a final weight ratio of CuCr_2O_4 : Ag of 9 : 1. The SEM investigation revealed that the surfaces of the NPs were shiny and smooth. Following electroless plating with Ag, nano-sized particles were found to be equally dispersed on the surface of CuCr_2O_4 , indicating that Ag was efficiently coated onto the CuCr_2O_4 surface.⁴⁶ Kien and colleagues used the sol–gel and impregnation methods to produce $\text{CuCr}_2\text{O}_4\text{-Pd}/\gamma\text{-Al}_2\text{O}_3$.¹⁰⁹ As previously described in their studies, they first synthesised Bi_2O_3 .¹¹⁰ Subsequently, the complexing-coprecipitation method was employed to synthesise $\text{CuCr}_2\text{O}_4\text{-CuO}$. As depicted in Fig. 8, electrostatic interaction between NH_4^+ and Bi_2O_3 NPs resulted in the coating of NH_4^+ on Bi_2O_3 surfaces because of their higher specific surface area and plentiful OH^- . Cu^{2+} and Cr^{3+} salts were then deposited on the Bi_2O_3 surfaces. NH_4^+ on Bi_2O_3 particles exhibits a strong complexing interaction with Cu^{2+} , and it may be absorbed on the surface of Bi_2O_3 , forming $[\text{Cu}(\text{NH}_3)_4]^{2+}$. Similarly, Cr^{3+} can be coated on the surface of Bi_2O_3 particles through electrostatic interaction between $[\text{Cu}(\text{NH}_3)_4]^{2+}$ and $[\text{Cr}(\text{OH})_4]^-$. After calcination,

Table 2 Summary of the synthesis method, characterization techniques, morphology, and applications of metal-doped CuCr_2O_4 NPs

Doped nanoparticles	Synthesis method	Morphology	Particle size (nm)	Characterization techniques	Applications	Ref.
CuCr_2O_4	Sol-gel	Spherical	>100	XRD, EDS, SEM, PL, Raman spectroscopy	—	94
$\text{Cu}_{1-x}\text{Cd}_x\text{Cr}_2\text{O}_4$ and $\text{Cu}_{1-x}\text{Nd}_x\text{Cr}_2\text{O}_4$	—	—	25–55	—	—	—
Ba and Co doped CuCr_2O_4	Hydrothermal	Irregular	CuCr_2O_4 -42 $\text{CoCuCr}_2\text{O}_4$ -52 $\text{BaCuCr}_2\text{O}_4$ -32	XRD, SEM, TEM, BET	Catalytic activity	95
Cu(II) oxide CuCr_2O_4 NPs	Hydrothermal	—	2–8	XRD, SEM, TEM, XPS, FTIR, TGA, TPR, BET, XANES, ICP-AES	Catalytic activity	24
Cu(II) supported CuCr_2O_4	—	—	~55	XRD, XPS, SEM, TEM, TPR, TGA	Catalytic activity	96
Cu(II) -supported CuCr_2O_4	Hydrothermal	—	30–60	XRD, BET, ICP-AES, SEM, TEM, TGA	Catalytic activity	97
Fe/Mn co-doped CuCr_2O_4	Co-precipitation	Octahedron	—	XRD, UV-vis-NIR, XPS, SEM, Raman spectroscopy	—	98
CuCr_2O_4 and Fe-substituted CuCr_2O_4	Sol-gel	Rectangular	—	XRD, UV-vis, Mossbauer, Raman spectroscopy, FESEM	—	99
La- CuCr_2O_4	Sol-gel Pechini	Spherical	—	XRD, SEM, FTIR, DRS, VSM	Photocatalytic activity	100
CuCr_2O_4	Combustion	Irregular	49–53	XRD, FTIR, FESEM, TEM, BET	Catalytic activity	101
Mg (0.1%) CuCr_2O_4	—	—	43–39	—	—	—
Mg (0.2%) CuCr_2O_4	—	—	40–31	—	—	—
Mg (0.3%) CuCr_2O_4	—	—	39–29	—	—	—
CuCr_2O_4	Hydrothermal and co-precipitation	Octahedral	71.84	XRD, UV-vis-NIR, SEM	Solar power	103
CuCrMnO_4	—	Octahedral	38.07	—	—	—
CuCrNiO_4	—	Flake	51.88	—	—	—
CuCrCoO_4	—	Flake	33.71	—	—	—
CuCrAlO_4	—	Irregular rods and polyhedral	31.64	—	—	—
CuCrZnO_4	—	Flake	38.41	—	—	—
CuCrSnO_4	—	Octahedral	86.36	—	—	—
$\text{ZnCuCr}_2\text{O}_4$	Sol-gel	—	—	XRD, SEM-EDS UV, PL, Raman spectroscopy	—	102
$\text{Cu}_{1-x}\text{Mg}_x\text{Cr}_2\text{O}_4$	Sol-gel	Quasi spherical	—	XRD, UV, SEM	—	105

**Fig. 8** Schematic of the spherical $\text{Bi}_2\text{O}_3/\text{CuCr}_2\text{O}_4\text{-CuO}$ core/shell NCs.¹¹¹

monodisperse spherical $\text{Bi}_2\text{O}_3/\text{CuCr}_2\text{O}_4\text{-CuO}$ core/shell nanocomposites were produced.¹¹¹

Balasurya *et al.* synthesized NCs using the co-precipitation method to synthesize CuCr_2O_4 NPs. Using these NPs and Bi salt, they synthesized $\text{CuCr}_2\text{O}_4\text{-BiI}_3\text{O}_9$ nanocomposites *via* a sonochemical method.⁴⁵ Ghorai and co-workers described $\text{CuCr}_2\text{O}_4/\text{BiOBr}$ NC. Sol-gel combustion produces spherical CuCr_2O_4 NPs, which are then decorated on BiOBr plates using precipitation. CTAB was used as the Br source and template (Fig. 9).¹¹²

A $\text{CuCr}_2\text{O}_4@\text{CaFe-LDO}$ hybrid was synthesized by Oladipo and group. They investigated its photocatalytic potential for treating olive mill effluent (OMW). These nanocomposites demonstrated outstanding absorption and photocatalytic activity.^{113,114} The researchers synthesized various nanocomposites, including $\text{CuCr}_2\text{O}_4\text{-CeO}_2$, using the sol-gel and co-precipitation methods, as well as a combination of these techniques.¹¹⁵ Additionally, they employed the impregnation method,¹¹⁶ reflux condensation,²⁶ and ultrasonication-assisted methods¹¹⁷ to synthesize $\text{Cu/CuCr}_2\text{O}_4$, $\text{CuO-CuCr}_2\text{O}_4$, and mesoporous $\text{CuCr}_2\text{O}_4\text{-g-C}_3\text{N}_4$ heterojunctions, respectively.

Hummers' approach was adapted to produce graphene oxide.¹¹⁸ Graphene nanocomposites with electron-donating inclusions increase the electronic significance of the mole-

cules they support.¹¹⁹⁻¹²¹ $\text{g-C}_3\text{N}_4\text{-NS/CuCr}_2\text{O}_4$ using refluxing,¹¹⁹ $\text{CuCr}_2\text{O}_4/\text{GO}$ using co-precipitation,¹²⁰ MXene ($\text{Ti}_3\text{C}_2\text{T}_x$)/ CuCr_2O_4 used wet-chemical sonication-assisted co-precipitation approach,¹²² nanospherical- CuCr_2O_4 nanocomposite decorated with polyaniline (PANI),¹²³ heterogeneous $\text{p-CuCr}_2\text{O}_4/\text{n-SnO}_2$,¹²⁴ and $\text{MCr}_2\text{O}_4/\text{TiO}_2$ (M-Cu, Ni) using the co-precipitation method.¹²⁵

Gao *et al.* employed a high-temperature phase separation technique to synthesize $\text{CuCr}_2\text{O}_4/\text{Cr}_2\text{O}_3$ nanogranular from Cu-doped Cr_2O_3 NPs, as confirmed by XRD tests. Its magnetic susceptibility has two transitions, *i.e.*, CuCr_2O_4 becomes ferrimagnetic at 130 K, and Cr_2O_3 becomes antiferromagnetic at 310 K.¹²⁶

Mechanical milling is a viable solid-state method for creating new $\text{CuCr}_2\text{O}_4/\text{CuCr}_2\text{O}_4$ nanocomposites. This approach affects both the outcome of the reaction product and the phase transitions that occur during the process. This research compares the heat treatment and characteristics of the nanocomposite to that of single-phase delafossite.¹²⁷

As depicted in the reaction pathway in Fig. 10, firstly CuCr_2O_4 NPs were fabricated *via* a sol-gel auto-combustion approach. Then, calcined NPs were dispersed in epoxy resin and polyurethane (PU) using ultrasonication to synthesize EP/PU/ CuCr_2O_4 nanocomposites. By adding CuCr_2O_4 NPs to the

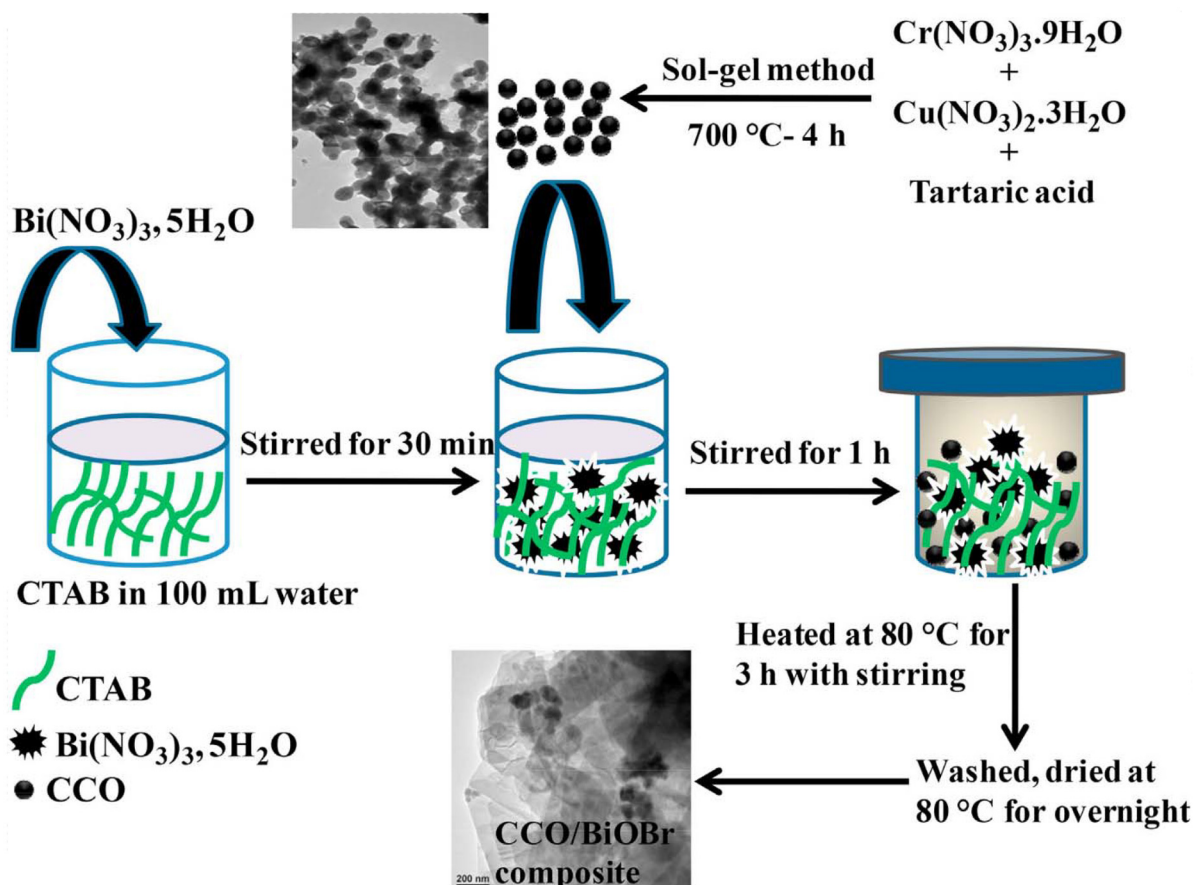


Fig. 9 Fabrication procedure of $\text{CuCr}_2\text{O}_4/\text{BiOBr}$ nanocomposite.¹¹²

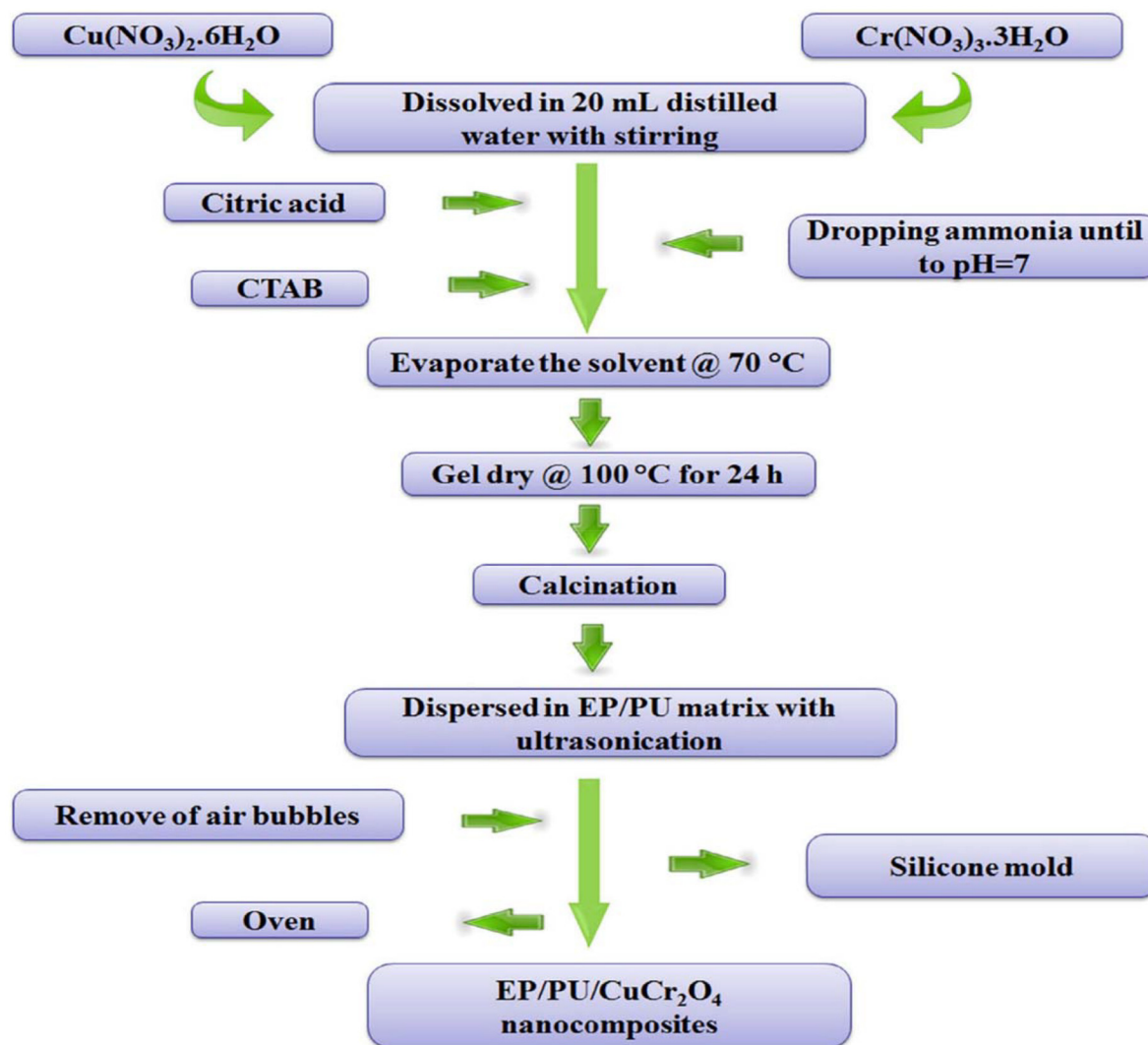


Fig. 10 Schematic layout of the synthesis of EP/PU/CuCr₂O₄ nanocomposites.¹²⁸

EP/PU matrix, the tensile test showed that the mechanical properties were greatly improved. The ideal composition was 0.76 wt% CuCr₂O₄ NPs and 2.6 wt% polyurethane in the epoxy resin. Additionally, molecular dynamics simulations showed how the NPs affected the mechanical properties and interaction energy of the nanocomposite.¹²⁸

The synthesis of CuCr₂O₄ NPs *via* the co-precipitation technique, using 6 M KOH as the precipitating agent, is illustrated in Fig. 11a. The TGA examination of the as-dried sample, as shown in Fig. 11b, indicates a weight loss of 25% between 28 °C and 260 °C because of the removal of C₂H₅OH and H₂O and a slight weight loss of 5.5% between 400 °C and 460 °C because of the elimination of HCl. According to the study, annealing should occur at 460 °C, while slower heating rates could lower this temperature. Using XRD analysis, Fig. 11c verifies the generation of CuCr₂O₄ NPs, and Fig. 11d displays the optical absorbance band. The band gap of CuCr₂O₄ NPs is 3.35 eV, according to Fig. 11e.¹²⁹

Susanti *et al.* produced N-doped reduced GO *via* hydrothermal temperature changes. XRD investigation revealed that the *d*-spacing value increased with temperature, indicating the presence of more substituted N atoms. The EDX examination revealed that the sample treated at a hydrothermal temperature of 160 °C had the highest N concentration (14.88%).¹³⁰ Interestingly, CuCr₂O₄ particles were supported on macroscopic polystyrene beads using a low-temperature ultrasonic technique.⁸ According to the SEM pictures, the CuCr₂O₄ particles completely coated the PS surface.¹³¹ TEM examination revealed that CuCr₂O₄ was uniformly distributed on rGO.¹³²

Intriguingly, Shi *et al.* synthesized CuCr₂O₄/SiO₂ composite membranes by impregnating a quartz glass fibrous (QGF) membrane with Cu and Cr precursors, followed by drying at 110 °C and calcining at 700 °C. Flat and frisbee-shaped membranes with varying CuCr₂O₄ loadings were produced with the frisbee-shaped samples assembled with round EPS disks to

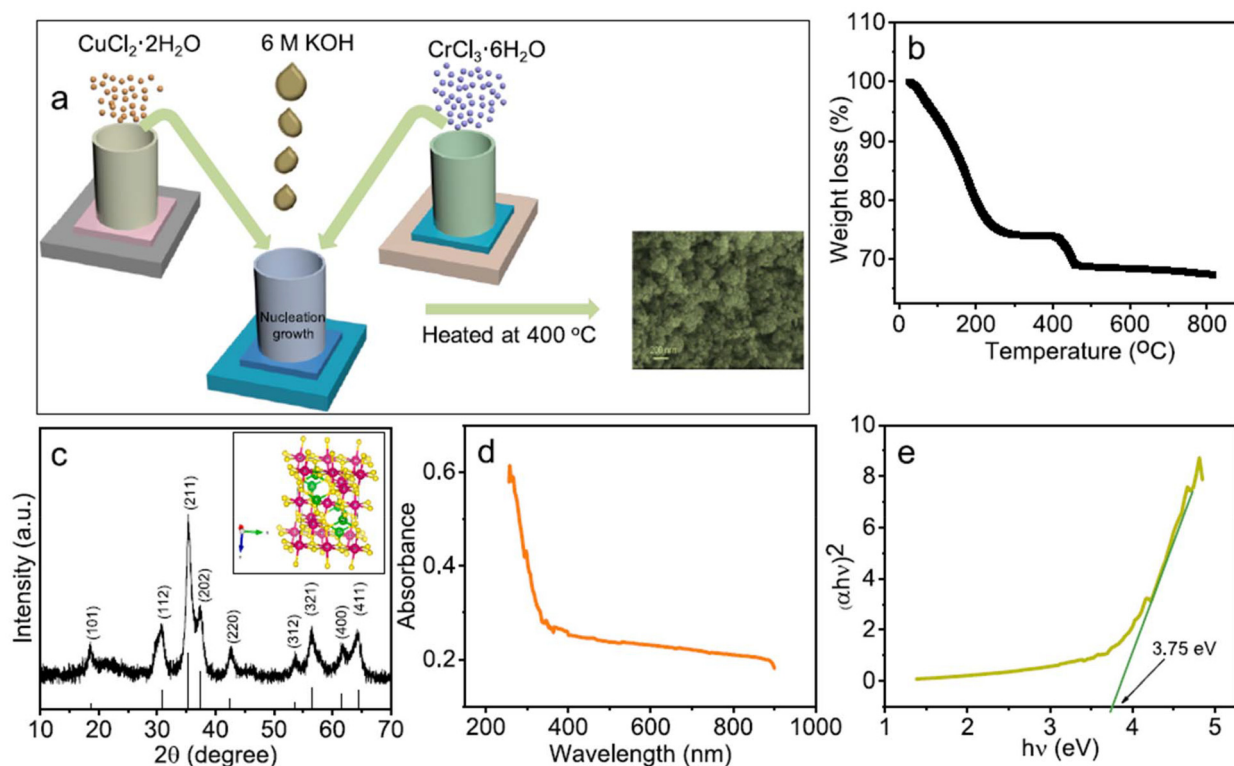


Fig. 11 (a) Diagram of the synthesis of CuCr_2O_4 (with an image of the final product), (b) TGA, (c) XRD spectra of the NPs, with an inset showing the crystal structure, (d) UV-visible spectra, and (e) optical band gap energy of CuCr_2O_4 NPs.¹²⁹

enable floating and provide heat barrier functionality for water distillation performance testing (Fig. 12).¹³³

Holec *et al.* used the sol-gel method to produce $\text{ZnCr}_2\text{O}_4/\text{SiO}_2$, $\text{CuCr}_2\text{O}_4/\text{SiO}_2$, and $\text{CdCr}_2\text{O}_4/\text{SiO}_2$, with HNO_3 as an acid catalyst, formamide (modifier), methanol (solvent), and TEOS (precursor). Furthermore, they studied the magnetic behavior

of the synthesized NPs; among them, the magnetic behavior of CuCr_2O_4 exhibits a ZFC-FC bifurcation at 150 K, hysteresis necking at 2 K, indicating two magnetic phases, and a wide anomaly at 30 K attributed to a surface spin superparamagnetic-like phenomenon.¹³⁴ Ghorai *et al.* and their research team constructed CuCr_2O_4 and TiO_2 by chemically depositing

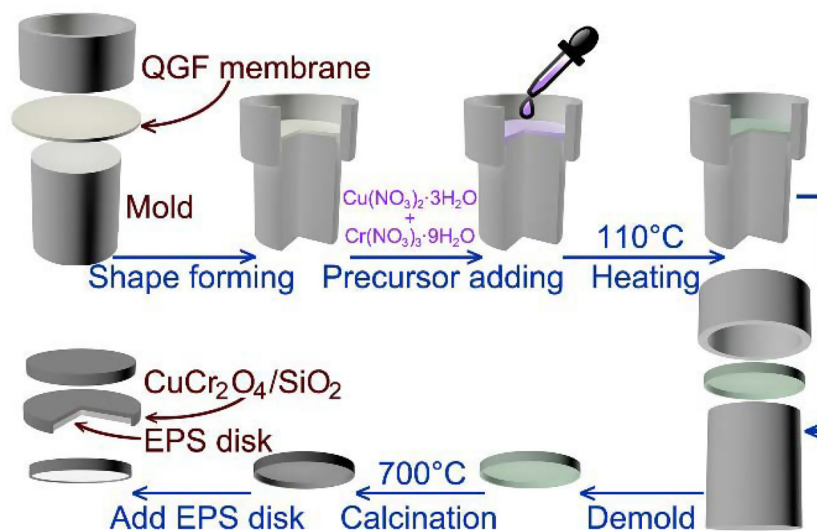


Fig. 12 Frisbee-shaped $\text{CuCr}_2\text{O}_4/\text{SiO}_2$ composite membrane disk synthesis and EPS assembly to generate a composite $\text{CuCr}_2\text{O}_4/\text{SiO}_2$ -EPS tandem-structured disk.¹³³

CuCr_2O_4 on a Ti precursor *via* sonochemical treatment, followed by thermal treatment.⁴⁸ Sivaranjani *et al.* observed that adding CuCr_2O_4 increased the particle size and decreased the full-width at half-maximum of the TiO_2 patterns, indicating improved crystallinity. According to the XRD analysis, CuCr_2O_4 improves the crystal structure of TiO_2 NPs.¹¹⁸

Khan *et al.* found that coupling the $\text{V}_2\text{O}_5/\text{CuCr}_2\text{O}_4$ -MCM-41 nanocomposite interface considerably improves the photocatalytic decomposition of alizarine red (AR) and bromothymol blue (BTB), as well as photoinhibition of *Staphylococcus aureus* and *Escherichia coli* bacteria.¹³⁵ Hosseini *et al.* employed CTAB and the sol-gel method to conduct a qualitative and quantitative study of CuCr_2O_4 NCs with different Cu and Cr molar ratios. Titration procedures were employed to determine the mass percentages of Cr_2O_3 and CuO. For each sample, the total percentage of Cr_2O_3 and CuO was approximately 100%, indicating the absence of impurities and confirming that the samples contained only Cr_2O_3 and CuO.¹³⁶ Vinothkumar *et al.* reported the synthesis of $\text{CuCr}_2\text{O}_4/\text{Cr}_2\text{O}_3$ NCs involving dissolving Cu(II) and Cr(III) precursors in dist. H_2O with stirring to obtain a homogeneous solution. Liquid ammonia is then added as a precipitating agent. The resulting precipitate is dried and calcined to form $\text{CuCr}_2\text{O}_4/\text{Cr}_2\text{O}_3$ NCs. The researchers successfully studied the supercapacitor performance of $\text{CuCr}_2\text{O}_4/\text{Cr}_2\text{O}_3$ composites on nickel foam, as demonstrated in Fig. 13.¹³⁷

Barman *et al.* created nano-copper chromite-loaded H-mordenite by combining equal quantities of copper and chromium salt precursors, followed by the addition of triethanolamine, while continuously stirring. To avoid precipitation, the pH was adjusted using HNO_3 . The solution was heated, and the

resulting fluffy mass was calcined at 923 K. The particle size was found to be 31 nm by TEM examination. The nano-copper chromite was then blended with H-mordenite in different ratios, mixed with 20% bentonite as a binder, extruded, and dried overnight at 373 K.¹³⁸ By thermally breaking down Cu(II) and Cr(III) carboxylate-type precursors that were generated *in situ* inside a silica matrix by a redox reaction with 1,3-propanediol, Ștefănescu *et al.* created $\text{CuCr}_2\text{O}_4/\text{SiO}_2$ and $\text{Cu}_2\text{Cr}_2\text{O}_4/\text{SiO}_2$ NCs that included 50 wt% of the corresponding oxides. Upon heating, the precursors decomposed at around 300 °C into amorphous CuO and $\text{Cr}_2\text{O}_{3-x}$. $\text{Cr}_2\text{O}_{3-x}$ crystallized to $\alpha\text{-Cr}_2\text{O}_3$ at 400 °C, which reacted with CuO to form $\text{Cu}_2\text{Cr}_2\text{O}_4$. Further heating to 800 °C and 1000 °C resulted in the formation of residual CuO and well-crystallized $\text{Cu}_2\text{Cr}_2\text{O}_4$, respectively. In both composites, the oxides were uniformly distributed in the amorphous silica matrix. The samples produced at various annealing temperatures were analyzed using thermal techniques.¹³⁹

Abbasi and colleagues used the sol-gel process to create spherical CuCr_2O_4 NPs with various amino acids as capping agents. They successfully examined how the molar ratio of the stabilizing agent affected the purity, particle size, and shape of the copper chromite. Moreover, a photodeposition method was used to create $\text{CuCr}_2\text{O}_4/\text{Ag}$ nanostructures. They reported that the surface roughness and particle size increase in the $\text{CuCr}_2\text{O}_4/\text{Ag}$ nanostructures compared to pristine CuCr_2O_4 , indicating effective surface modification through Ag coating and potential impact on photocatalytic capabilities.¹⁴⁰

Table 3 summarizes the diverse fabrication strategies, characterization techniques, and applications of CuCr_2O_4 nanocomposites.

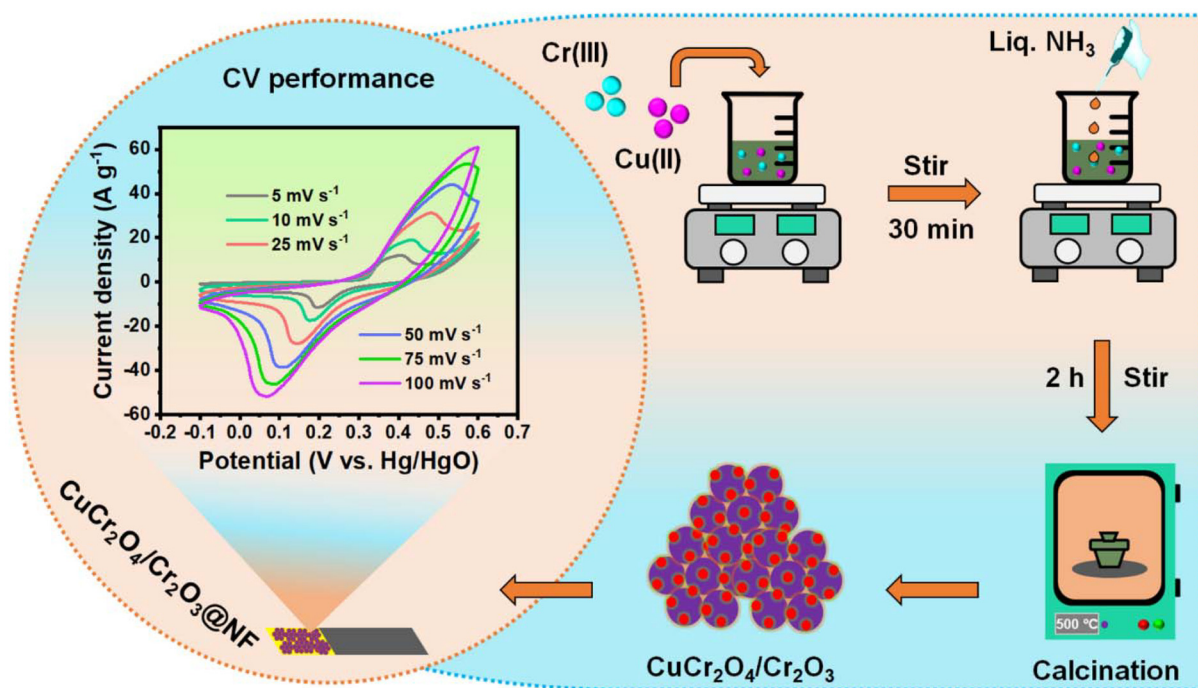


Fig. 13 Schematic layout of the synthesis of $\text{CuCr}_2\text{O}_4/\text{Cr}_2\text{O}_3$ composite on an Ni foam for supercapacitors.¹³⁷

Table 3 Summary of the synthesis method, characterization techniques, morphology, and applications of CuCr₂O₄ nanocomposites

Nanocomposites	Synthesis method	Morphology	Particle size (nm)	Characterization techniques	Applications	Ref.
Ag-coated CuCr ₂ O ₄	Sol-gel	—	—	XRD, SEM-EDS, TEM, TG-DSC	Li-ion batteries	46
CuCr ₂ O ₄ /Pd ₇ /Al ₂ O ₃	Sol-gel and impregnation	Rod	—	XRD, SEM-EDS, BET	Catalytic activity	109
Bi ₂ O ₃ /CuCr ₂ O ₄ -CuO core/shell	Co-precipitation	Spherical	78	XRD, TEM, EDS, FTIR	Catalytic activity	111
CuCr ₂ O ₄	Co-precipitation	Flake	—	XRD, UV-vis-DRS XPS, SEM-EDAX, SAED, BET, EIS, PL	Photocatalytic activity and genotoxic effect	45
CuCr ₂ O ₄ -Bi ₂ O ₃	Sono chemical	CuCr ₂ O ₄ Cube	—	—	—	—
CuCr ₂ O ₄	Sol-gel combustion	Bi ₂ O ₃	—	—	—	—
CuCr ₂ O ₄ /BiOBr	Sol-gel combustion	Spherical	30–80	XRD, UV-Vis, DRS, FESEM, TEM, HRTEM, EDX, XPS, Raman spectroscopy	Photocatalytic and electrochemical activity	112
CuCr ₂ O ₄	Sol-gel	Spherical	65	XRD, UV-vis, FTIR, PL	Photocatalytic activity	141
BiVO ₄	Chemical route	—	—	—	—	—
CuCr ₂ O ₄ @CaFe-LDO	—	—	—	XRD, UV-Vis, DRS XPS, SEM-EDX, TEM, TG-DTA	Photocatalytic activity	114
CuCr ₂ O ₄ @CaFe-LDO	—	—	12.51	—	—	113
CuCr ₂ O ₄ -CeO ₂	Sol-gel combustion	Semi-spherical	15	XRD, UV-Vis DRS, TEM, EDS, XPS, Raman spectroscopy, PL, BET	Photocatalytic activity	115
Cu/CuCr ₂ O ₄	Impregnation	—	50–100	XRD, FTIR, HRTEM, FESEM, XPS, XANES/EXAFS	Catalytic activity	116
CuCrO ₂ /CuCr ₂ O ₄	Ball milling	—	174 ± 5	XRD, UV-vis, FESEM, XPS, DTA, FESEM, EDS, TEM, SAED	Photocatalytic activity	127
CuCr ₂ O ₄ /CuO	Co-precipitation	—	—	SEM/EDAX, ICP/AES, XRD, and XPS	Catalytic activity	142
CuO-CuCr ₂ O ₄	Reflux condensation	Flower	—	XRD, UV-vis-DRS, TEM, PL, FESEM	Photocatalytic activity	26
EP/PU/CuCr ₂ O ₄	Sol-gel auto combustion	Octahedral	—	XRD, UV-vis, FTIR, SEM, XPS, TG-DSC	—	128
CuCr ₂ O ₄ -g-C ₃ N ₄	Ultrasonication-assisted	Irregular	12–16	XRD, FTIR, UV-vis DRS, TEM, PL, XPS, BET, EDX	Photocatalytic activity	117
g-C ₃ N ₄ -NS/CuCr ₂ O ₄	Refluxing	—	12.6	XRD, UV-Vis DRS, FTIR, SEM, EDX, TEM, XPS, PL, BET, TGA	Photocatalytic activity	119
CuCr ₂ O ₄ /GO	Co-precipitation	Quasi-spherical	GO-1.39 CuCr ₂ O ₄ /GO-3.54	XRD, SEM, TEM, EDS, PL, Raman spectroscopy	Energy storage and electrochemical activity	143
CuCr ₂ O ₄ and MWCNT/ CuCr ₂ O ₄	Co-precipitation	Quasi-spherical	—	XRD, UV-Vis, FTIR, XPS, SEM-EDS, BET, HRTEM-SAED	Supercapacitor	129
MXene/CuCr ₂ O ₄	Co-precipitation	—	CuCr ₂ O ₄ -21.2, Ti ₃ C ₂ T _x -25.5 Ti ₃ C ₂ T _x /CuCr ₂ O ₄ - 8.7	UV-vis, SEM-EDX, zeta potential, Raman spectroscopy, PL, FTIR	Energy storage	122
N-doped graphene CuCr ₂ O ₄	Sonication-assisted mechanical mixing	—	—	XRD, FTIR, XPS, SEM-EDS, STEM	Supercapacitor	121
N-doped rGO CuCr ₂ O ₄	Sol-gel	—	—	XRD, FTIR, SEM-EDX	Supercapacitor	130
PANI: CuCr ₂ O ₄	—	Nano spherical	—	XRD, UV-Vis, FTIR, XPS, BET, TEM, and PL	Photocatalytic activity	123
CuCr ₂ O ₄ -PS	Low-temperature ultrasonic	—	—	XRD, FTIR, UV-Vis, SEM-EDS, BET	Photocatalytic activity	131
CuCr ₂ O ₄ /rGO	Hydrothermal	—	50	XRD, FTIR, TEM, TGA, XPS, zeta potential, Raman spectroscopy	Catalytic activity	132
CuCr ₂ O ₄ /SiO ₂	—	Frisbee	—	XRD, UV-vis-NIR, SEM, IR	Solar driven water evaporation	133
CuCr ₂ O ₄ /SiO ₂	Sol-gel	—	—	XRD, TEM	—	134
CuCr ₂ O ₄ /SnO ₂	Sol-gel	—	—	XRD, XPS, FTIR	Photocatalytic activity	124
CuCr ₂ O ₄	Sol-gel	Spherical	25–30	XRD, TEM-SAED, SEM-EDX, BET	Photocatalytic and photoelectrocatalytic activity	48
TiO ₂	Sonication	Semi sphere	6–10	—	—	—
CuCr ₂ O ₄ /TiO ₂	Co-precipitation	—	54	XRD, UV-vis	Dye sensitized solar cell	125
CuCr ₂ O ₄ /TiO ₂	Sol-gel	—	50	XRD, UV-Vis-DRS, TEM	Photocatalytic activity	144

Table 3 (Contd.)

Nanocomposites	Synthesis method	Morphology	Particle size (nm)	Characterization techniques	Applications	Ref.
CuCr ₂ O ₄ -0.7TiO ₂ -rGO	Sol-gel assisted Hummers' method	Spherical	100	XRD, FTIR, SEM-EDX, XPS, TGA-DTA, Raman spectroscopy	Catalytic activity	145
CuCr ₂ O ₄ /TiO ₂	Sol-gel	Spherical	25–40	XRD, SEM, TEM	Catalytic activity	146
CuCr ₂ O ₄ /TiO ₂	Sol-gel	—	66	XRD, DRS-UV, BET, FT-IR	Photocatalytic activity	118
V ₂ O ₅ /CuCr ₂ O ₄ /MCM-41	Hydrothermal	CuCr ₂ O ₄ /MCM-41-hexagonal V ₂ O ₅ -spherical Pseudospherical	32 6 50–500	XRD, UV-Vis, FTIR, BET, HRTEM, SEM-EDX, TGA	Photocatalytic and antibacterial activity	135
CuCr ₂ O ₄ nanocomposites	Sol-gel	—	—	XRD, SEM, EDAX, FTIR	Photocatalytic activity	136
CuCr ₂ O ₄ -Cr ₂ O ₃	Co-precipitation	Spherical	±25	XRD, FTIR, UV-Vis, SEM, EDX TEM, SAED, XPS, EIS, BET	Supercapacitors	137
50 mass% CuCr ₂ O ₄ /SiO ₂	Modified sol-gel	—	43	XRD, FTIR	—	139
50 mass% Cu ₂ Cr ₂ O ₄ /SiO ₂	—	—	50.5	—	—	—
CuCr ₂ O ₄ /Ag-leucine	Sol-gel	Quasi-spherical	30–60	ICP-OES, DRS, XRD, SEM, EDS, TEM	Photocatalytic activity	140

3. The impact of experimental parameters on CuCr₂O₄ NPs physico-chemical properties

3.1. pH

According to Pakzad *et al.*, particle size is significantly affected by pH and the amount of fuel. Doubling the amount of fuel greatly increases the particle size. They observed that low citric acid (4 mmol):glycine (10 mmol) and high citric acid (8 mmol):glycine (20 mmol) ratios resulted in average particle sizes of 0.74 and 5.18 μm , respectively. It was also discovered that as the fuel/oxidizer ratio increased, the particle shape shifted toward octahedral structures, with larger particles observed at pH 9. They concluded that particle size increases with an increase in the fuel/nitrate ratio or solution pH.¹⁷

3.2. Time

Mobini *et al.* investigated the effect of aging time on material structure and found that prolonged aging significantly influenced crystallinity. They reported that the surface area increased from 20.4 to 42.8 $\text{m}^2 \text{g}^{-1}$ as the aging period progressed. At the same time, the overall crystallinity decreased, accompanied by an increase in amorphous content and the development of different crystalline phases.⁷⁷ Chiu *et al.* delved into the impact of time on nanoparticle morphology. The topology of the fibers was altered significantly as the sintering time increased. All the fibers had a coarse morphology, and this roughness increased with sintering time.⁸⁸ Surprisingly, researchers aimed to study the development of 3D raspberry-like structures. They conducted time-dependent experiments at 180 °C utilizing a hydrothermal approach. As seen in Fig. 14, after 18 h of crystallization, the particles formed a raspberry-like shape with homogeneous diameters. However, prolonged crystallization (>18 h) led to the development of polydispersed particles.⁷⁸ Similarly, Yuan *et al.* used a Teflon-lined stainless-steel autoclave heated to 180 °C and maintained for 11 h to synthesise cubic-like CuCr₂O₄ particles.²²

3.3. Concentration

Sankudevan *et al.* found that increasing the Mn doping concentration in CuCr₂O₄ NPs reduced the particle size from 37 to 26 nm, as confirmed by XRD analysis.⁴³ Pure CuO and CuO-CuCr₂O₄ NCs were made with various Cr³⁺ concentrations. The monoclinic structure of pure CuO is reflected in its XRD. CuO-CuCr₂O₄ with a 10% Cr³⁺ concentration exhibits a weak peak at 18.12°, indicating the presence of CuCr₂O₄, which has a tetragonal structure. The CuCr₂O₄ peak intensifies when the Cr³⁺ concentration increases (CuO-CuCr₂O₄: 20% Cr³⁺ concentration and CuO-CuCr₂O₄: 30% Cr³⁺ concentration), whereas the CuO peaks diminish. The crystallite size decreases with an increase in Cr³⁺ content, indicating that CuCr₂O₄ inhibits CuO growth and may diminish the lattice characteristics of CuO.²⁶ Batool *et al.* reported that the grain size reduced when the Cd and Nd doping concentrations increased, which was further

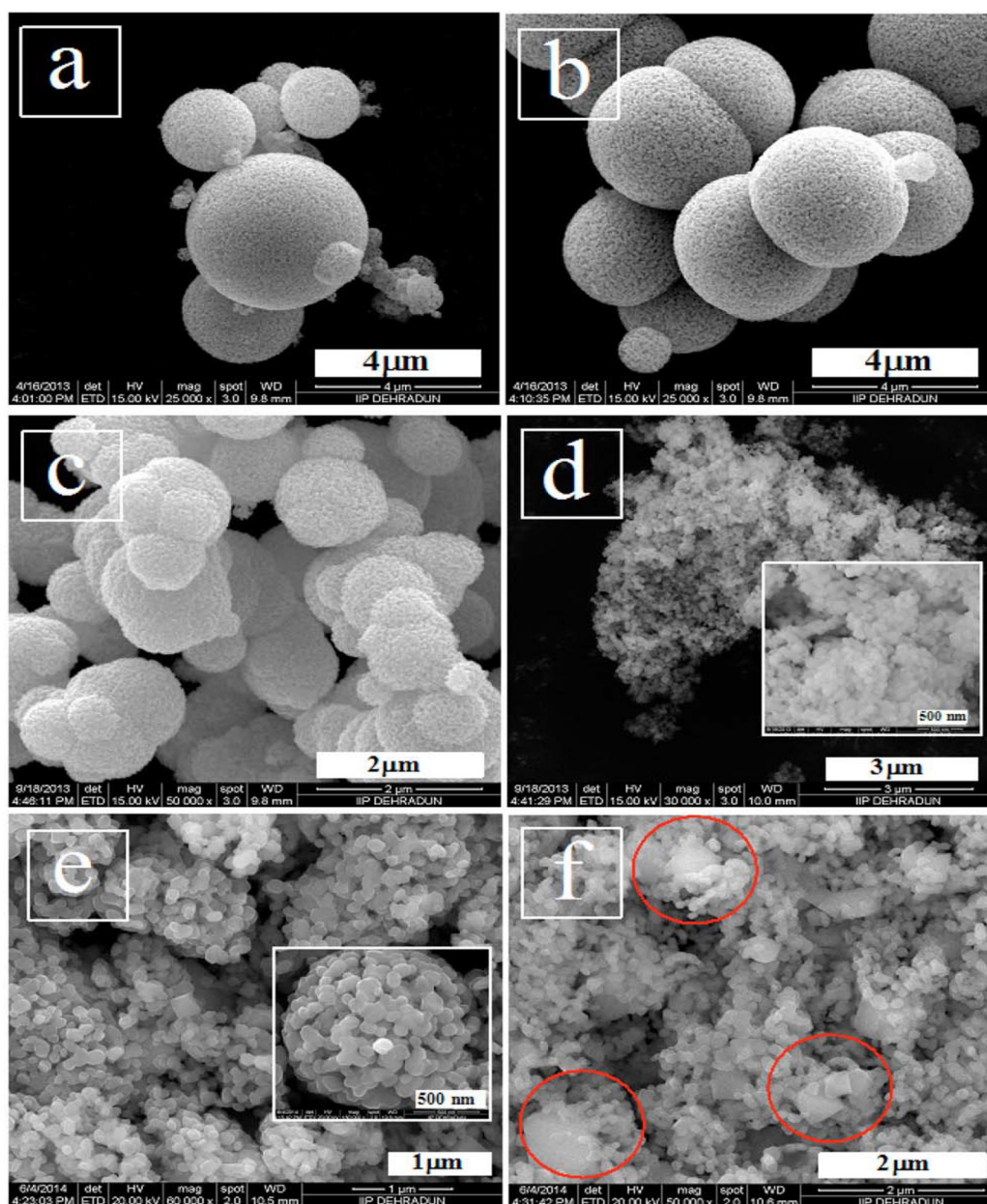


Fig. 14 SEM images of CuCr₂O₄ NPs synthesized using hydrothermal techniques at 180 °C for (a) 6 h, (b) 18 h, (c) 72 h, (d) 7 days, (e) after 7 days of constant stirring, and (f) after 7 days of aging.⁷⁸

confirmed by SEM analysis.¹⁰² Ramezanalizadeh *et al.* investigated the effect of varying the La loading on the morphology, size, and crystallinity of CuCr₂O₄ NPs using FESEM analysis. According to the experimental findings, the shape of the nanostructure changed dramatically with an increase in La concentration, going from plate-like at 0.35 La loading to spherical-like at 0.7, as shown in Fig. 15. Additionally, a marked change in particle size was observed. These structural and morphological changes, as supported by XRD data, indicate that the addition of La to the CuCr₂O₄ lattice has a significant impact on the crystalline phases and physical properties of the material.¹⁰⁰

3.4. Temperature

Hosseini *et al.* investigated several factors, including the Cu/Cr ratio and calcination temperature in the range of 500–750 °C, to examine the formation of diverse phases, including CuCr₂O₄, CuO, and CuCrO₂. It was established that the phase composition of the resulting CuCr₂O₄ NPs is determined by the molar ratio of Cu–Cr in the initial reactants. When the reaction temperature is set to 600 °C and the Cu/Cr molar ratio is 0.5, pure spinel CuCr₂O₄ NPs are obtained.⁵⁰

Tavakoli *et al.* demonstrated that the phase structure of the CuCr₂O₄ compound depends on the initial reactant tempera-

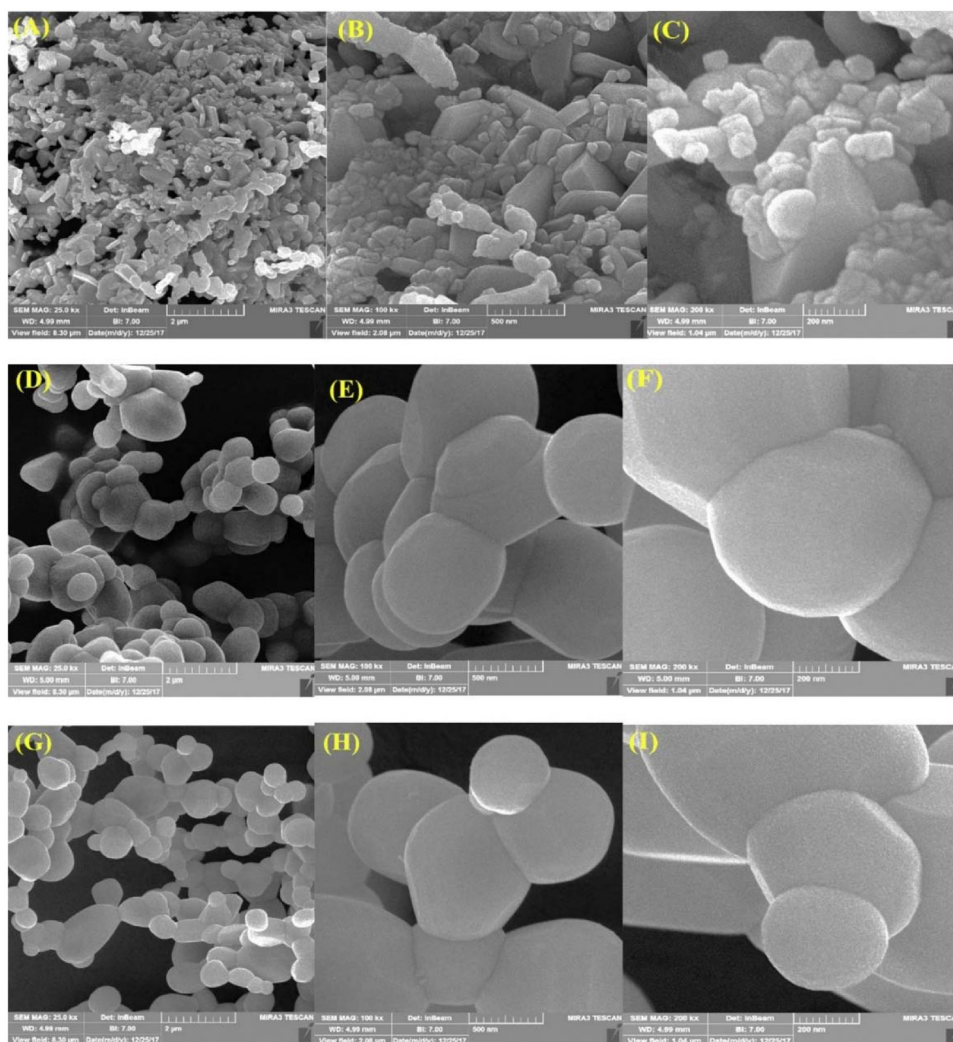


Fig. 15 FESEM images of $\text{CuCr}_{1.65}\text{La}_{0.35}\text{O}_4$ (A–C), $\text{CuCr}_{1.3}\text{La}_{0.7}\text{O}_4$ (D–F), and CuCrLaO_4 (G–I).¹⁰⁰

ture and the molar ratio of Cu:Cr.⁷⁰ Pal *et al.* observed that CuCr_2O_4 samples synthesized at 400–500 °C formed NPs with slight agglomeration, while calcination at 550 °C led to significant particle growth and the formation of predominantly octahedral-shaped structures. The particle size increased in tandem with the calcination temperature, primarily due to the aggregation of smaller particles at higher temperatures.⁷¹ Chiu *et al.* tested a sample sintered at temperatures in the range of 500–800 °C for varying periods of time. The fiber sintered at 500 °C included a combination of Cr_2O_3 and CuCr_2O_4 , but sintering at 600–800 °C produced single-phase CuCr_2O_4 . The mean fiber diameter reached 500 nm. Secondary-phase Cr_2O_3 was discovered when the fiber was sintered at 700 °C for 5 and 10 min, while single-phase CuCr_2O_4 occurred when the sintering time was raised to 15–30 min.⁸⁸ Mobinia's study suggested that the thermal sintering of particles at high temperatures causes the BET surface area to decrease, given that sintering reduces the porosity and crystal size.⁷⁷

Billman *et al.* suggested that the modified Pechini method produces smaller particle sizes and increased agglomeration.

The measured particle growth kinetics are consistent with a diffusion-limited, inhibited grain growth model.⁵⁸ Similarly, Tsai *et al.* revealed that higher temperatures increase the particle size by boosting atomic diffusion, and also enlarge the voids by inducing atoms to cluster around the void margins due to capillary forces. They further reported that annealing the nanostructure at 500 °C transformed the film structure from amorphous to a mixture of monoclinic CuO and spinel CuCr_2O_4 . Delafossite CuCrO_2 was formed by further annealing at 600 °C, which depleted the CuCr_2O_4 phase. Increasing the annealing temperature also altered the microstructure, evolving from compact nanograins to larger agglomerated grains with voids, resulting in a rougher surface.⁸⁹ Similarly, Batool *et al.* also observed the effect of temperature on the morphology of CuCr_2O_4 NPs doped with Mg. The researchers observed that at lower temperatures, the particles exhibited quasi-spherical shapes with low homogeneity. As the temperature increased, the particle size increased, and the shapes became less uniform. The particles showed higher aggregation, less spherical appearance, and more disorder at 750 °C.¹⁰⁵

3.5. Fuel

Capping agents play a crucial role in the synthesis of nanoparticles by increasing the monodispersity, reducing the particle size, and preventing the aggregation of NPs. Sathiskumar and colleagues studied Cu–Cr–O NC oxide catalysts manufactured using various Cu–Cr mole ratios to better understand the impact of the mole ratio on the final product, using fuels such as glycine and citric acid.⁸ Bisht *et al.* utilized capping agents, namely, urea, coconut oil, oleic acid, and citric acid, to generate CuCr₂O₄ NPs. It was observed that improved surface capping occurred in the presence of oleic acid.⁸² Paymenfar *et al.* studied the influence of a capping agent on CuCr₂O₄ NPs using FESEM analysis. According to the analysis, they concluded that the uncapped particles exhibited a semi-spherical morphology with noticeable aggregation and low monodispersity. In contrast, the use of sucrose as a green capping agent reduced the particle size, minimized the agglomeration, and greatly enhanced the monodispersity. The findings demonstrate the importance of the capping agent in regulating the shape and crystallinity of nanoparticles during sol–gel production.⁵¹ To adjust morphology and particle size, Kumar *et al.* employed a variety of surfactants, including PEG, CTAB, and PVP. Among them, the CuCr₂O₄ material made with CTAB surfactant exhibits the greatest specific capacitance due to its larger surface area, rougher surface, and enhanced ion adsorption active sites resulting from its spherical shape.⁷³ Al-Wasidi *et al.* created CuCr₂O₄ NPs with and without citric acid and tartaric acid as templates. The presence of organic acids caused lower crystallite sizes. Organic templates had a considerable influence on crystallite size and morphology; the nanoparticles produced without templates had irregular forms, but those with templates were spherical.⁸¹ The impact of glycine, alanine, valine, and leucine as capping agents in the synthesis of CuCr₂O₄ NPs was examined by Ali Abbasi *et al.* Glycine, the smallest amino acid, has a weak steric barrier due to its tiny molecular size, resulting in the development of agglomerated particles with an unknown shape. In contrast, the NPs synthesised with leucine as both a capping and reducing agent have a well-defined, sphere-like shape. Among the amino acids investigated, leucine exhibited the most pronounced steric hindrance effect, resulting in more uniform and smaller particles. Based on the structural properties of the amino acids, it can be concluded that larger amino acids provide more steric hindrance, which is advantageous for the controlled synthesis of subminiature NPs with enhanced shape and reduced agglomeration.¹⁴⁰

Table 4 describes the diverse parameter-dependent fabrication of CuCr₂O₄ nanomaterials.

4. Proposed mechanism for plant extract-mediated green synthesis of CuCr₂O₄ NPs

This eco-friendly process produces nanoparticles that are more stable, less prone to deformation and aggregation, and

promote the adsorption of phytochemicals on their surface, thereby accelerating the reaction times.¹⁴⁷ Lachini *et al.* employed an aqueous extract of *Aloe vera* leaves as a reducing agent for the synthesis of CuCr₂O₄ NPs.⁴⁹ Fig. 16 presents the probable mechanistic process for the green synthesis of CuCr₂O₄ NPs. The proposed reaction mechanism demonstrates the stabilising and reducing effects of active phytochemicals. Numerous active phytochemicals, including anthraquinones, polysaccharides, flavonoids, vitamins, enzymes, saponins, tannins, and sterols, are found in *Aloe vera* leaves.^{148–150} To suggest the possible reaction mechanism, a flavonoid has been chosen as a model biomolecule.¹⁵¹ The electrostatic interaction between the hydroxyl groups of flavonoids and the cations of metal precursors causes aromatic hydroxyl groups to bind to copper and chromium ions, and stable complexes are formed between the metal ions and flavonoids. After calcination, the complex breaks down, yielding CuCr₂O₄ NPs.

Furthermore, the CuCr₂O₄ NPs were created by reducing Cu²⁺ and Cr³⁺ ions using bioactive phytochemicals, such as polyphenols, flavonoids, and terpenoids, which are present in rosemary leaf extract and serve as natural stabilizing and reducing agents. The reduction was then finished by adding NaBH₄, and the crystalline spinel structure was achieved by calcining the mixture at 650 °C.⁴¹

5. Applications of CuCr₂O₄ nanomaterials

5.1. CuCr₂O₄ nanomaterials as catalysts

In an aqueous medium, ethyl acetoacetate, hydrazine hydrate, and aldehydes underwent a pseudo-five-component reaction to produce 4,4'-(arylmethanediyl)bis(3-methyl-1H-pyrazol-5-ols), according to Safaei-Ghomi *et al.* (Scheme 1).⁶⁷

Safaei-Ghomi *et al.* utilized CuCr₂O₄ NPs as a catalyst in the Ullman reaction. C–N cross-coupling reactions between anilines and iodobenzenes were performed in toluene under reflux conditions with a nitrogen atmosphere, using KOH as the base and a CuCr₂O₄ nanocatalyst under ligand-free conditions. This nanocatalyst enhanced the pace and efficiency of triarylamine synthesis (Scheme 2).¹⁶

Shahbazi-Alavi *et al.* described a one-pot pseudo-five-component condensation reaction utilizing a nano-CuCr₂O₄ catalyst to produce bis-thiazolidinones (Scheme 3). The creation of primary imine intermediates, followed by the attack of the sulphur atoms of thioglycolic acid on the activated imine groups, intramolecular cyclization, and the removal of water, results in the cyclized product bis-thiazolidines, according to the suggested mechanism. By activating the C=O, C=N, and S–H groups, the nano-CuCr₂O₄ surface atoms improve the interaction with nucleophiles. These surface atoms are hubs for catalytically stimulating chemical processes.⁶⁸

The study by Shahbazi-Alavi *et al.* employed a similar process, but they incorporated ammonium acetate as a key component to alter the synthesis of arylated dipyrazolodihydropyridine derivatives. When ammonium acetate, EAA, 4-nitrobenzal-

Table 4 Overview of the parameters influencing the development of CuCr₂O₄ NPs

Synthesis method	Parameters for the synthesis of CuCr ₂ O ₄ NPs				Temperature			Ref.
	Concentration of Cu salt or weight	Concentration of Cr salt or weight	Molar ratio Cr ³⁺ : Cu ²⁺	pH	Reaction (°C)	Calcination (°C)	Time (h)	
Sol-gel	0.01 mol	0.02 mol	—	—	95	600	3	50
	—	—	—	—	50	700	6	52
	0.01	0.02	—	—	90	750 and 850	4	53
	—	—	1 : 2	8.5	90	300 and 850	6 and 4	42
	2 mmol	4 mmol	—	—	70	—	—	55
	—	—	—	10–11	80	820	4	56
	0.799 g	1.833 g	—	—	180	600	4	60
	—	—	1 : 2	7	120	900	24	46
	—	—	0.5	3	—	550	5	109
	0.005 mol	0.01 mol	—	—	80	800	1.5	141
	—	—	—	—	95	750	4	143
	0.1 mol	0.2 mol	—	—	50–65	500	2	121
	—	—	—	—	40	800, 900, 1000, 1100	2	134
	0.005 mol	0.01 mol	—	—	80	300, 500, 700	1.5	144
	5 mmol	10 mmol	—	—	80	600	3	146
	5 mM	10 mM	—	—	95	600	3	118
	0.01	0.02	—	~7	90	750	4	102
	0.01 mol	0.02 mol	—	—	95	600	3	99
	2.0 g	10 g	—	8	180	750	6	27
	0.01296	0.02592	1 : 1	—	90	800	3	139
0.5 g (2 mmol)	1.658 g (4 mmol)	1 : 2	7	70	1000	2	62	
1 mmol	2 mmol	1 : 2	—	70	650	3	140	
Sol-gel auto combustion	7.5 mmol	15 mmol	1 : 2	8–10	90	950	1	17
	—	—	—	—	120	900	10	59
	0.9053 g	3 g	—	—	80	700	4	112
	2.152 mmol	4.298 mmol	—	7	70	700	3	128
Sol-gel	0.9057 g	3 g	1 : 2	—	80	700	4	48
	1 mmol	2 mmol	—	—	80	650	3	49
Green synthesis	1 mmol	2 mmol	—	—	100	650	3	—
Pechini	0.07 mol	0.1 mol	—	—	95	600	3	57
Pechini-and modified Pechini	—	—	1 : 2	7	80	650, 750, and 850	1 and 24 h	58
Combustion	36.23 g	60 g	—	—	—	750–800	2	65
	0.5 M	1 M	—	—	—	400	5	101
Self-combustion	10 mmol	20 mmol	—	—	90	600	3	64
Co-precipitation	1.4 g	4.0 g	—	10	80	600	5	16, 38 and 67–69
	0.020 mol	0.040 mol	—	—	—	400, 450, 500 and 600	3	71
	0.35 M	0.35 M	—	—	—	300	6	92
	—	—	—	—	—	Reduced with H ₂ 300	8	—
	1.208 g	4.0015 g	—	—	110	600 and 700	4	23
	0.5 M	0.5 M	—	8	90	800	5	111
	0.1 M	0.2 M	—	11	80	700	4	45
	0.005 mol	0.01 mol	—	8–9	—	1000	2	72
	1.70 g	5.32 g	—	—	85	400	4	129
	—	—	1 : 2	—	—	700	6	131
	0.05 mol L ⁻¹	—	8–9	—	—	900	1	98
	0.005 mol	0.01 mol	1 : 2	9	—	400, 520, and 800	2	70

Table 4 (Contd.)

Synthesis method	Parameters for the synthesis of CuCr ₂ O ₄ NPs								
	Concentration of Cu salt or weight	Concentration of Cr salt or weight	Molar ratio Cr ³⁺ : Cu ²⁺	pH	Temperature			Ref.	
					Reaction (°C)	Calcination (°C)	Time (h)		
Hydrothermal	1.5 g	5.0 g	1 : 2	8	180	750	6	74 and 75	
	3.5 g	11 g	1 : 2	8	200	700	-	35	
	5 mmol	10 mmol	1 : 2	10–10.3	70	500–800	4	22	
	2.25 g	7.5 g	1 : 2	8	180	750	6	24	
	5.54 g	15.4 g	—	7	—	500	5	47	
	2.3 g	7.5 g	1 : 2	8	180	750	6	76	
	3.8 g	12.3 g	1 : 2	9	180	650	6	39	
	0.1 g	0.27 g	—	—	120	400	3	40	
	—	—	1 : 2	9, 10, 11, 12	160, 180 and 200 (11 h)	400, 500, 600 and 700	4	77	
	1 mmol	2 mmol	—	14	180	700	5	80	
	2.6 g	2.7	1 : 2	—	180	800	6	78	
	1 mmol	2 mmol	—	—	—	—	—	93	
	1 M	2 M	—	10–11.5	200	550	5	14	
	Equal	Equal	—	7	200	750	5	95 and 103	
	5.5 g	16 g	—	8	200	750	—	97	
	2.30 g	7.50 g	—	10	180	700	6	81	
	Microwave	1 mmol	2 mmol	—	—	—	500–800	3	82
	Ultrasonication assisted	5.5 g	15.4 g	—	—	40	550	3	117
Refluxing	0.03 g	0.19 g	—	10	96	520	4	119	
—	5.54 g	15.4 g	—	7	60	500	5	123	
—	0.03 g	0.07 g	—	10	60	500	4	135	
Co-precipitation, ultrasound, and green chemistry	1.7 g	0.5 M	—	7	—	650	4	41	

dehyde, and hydrazine hydrate were reacted in ethanol at room temperature, using nano-CuCr₂O₄ as a catalyst, significant yields of tetrahydrodipyrzopyridines were produced (Scheme 4). Furthermore, the recyclability of the catalyst was demonstrated up to five cycles in the model reaction.¹⁵²

Wołosiak-Hnat *et al.* produced a CuCr₂O₄ catalyst with a more crystalline structure and a higher copper concentration than the commercial catalyst, enabling greater selectivity for 1,2-propanediol. At 210 °C, 80 wt% aqueous glycerol and 8 wt% catalyst resulted in the maximum glycerol conversion of 76.9% and 90.7% selectivity to 1,2-propanediol compared to the inserted glycerol, respectively. The studies were conducted under an initial hydrogen pressure of 3.0 MPa, stirring speed of 100 rpm, reaction time of 24 h, and pressure of 5.6 MPa throughout the process. Under the same conditions, the maximum selectivity for 1,3-propanediol was just 2.5% at a glycerol conversion rate of 30.9%. This study suggested that the catalyst must be present for the C–O bond in alcohols to be activated for hydrogenolysis. The hydrogenolysis of glycerol to 1,2- and 1,3-propanediol is generally considered a two-step procedure. Glycerol is dehydrated to produce acetol or 3-hydroxypropanal in the first stage. Hydrogenating these intermediates to 1,2- and 1,3-propanediol, respectively, is the second step (Scheme 5).⁹²

Karimi-Jaberi *et al.* described the formation of benzylidene bis(4-hydroxycoumarin) and dihydropyrano[c]chromene

analogs in H₂O at RT with nano-CuCr₂O₄ as a catalyst. This process is eco-friendly, consumes less time, produces high yields, and the catalyst can be reused, making it simple, practical, and sustainable (Scheme 6a and b).³⁸

Acharyya and research team have published multiple reports on the synthesis of CuCr₂O₄ NPs and their catalytic activity. In this report, a nano-catalyst with improved thermal stability and enhanced catalytic performance in the hydroxylation of benzene to phenol, oxidation of ethylbenzene to acetophenone, and oxidation of aniline to azoxybenzene was generated, utilizing H₂O₂ as the oxidizing agent. The study found that at 80 °C, the benzene conversion was 72.5% with 94% phenol selectivity (entry 1, Table 5).⁷⁴ In comparison, at 70 °C, the ethylbenzene conversion was 68.5%, yielding 78% acetophenone (entry 2, Table 5),⁷⁵ aniline conversion was 78%, with 92% selectivity to azoxybenzene (entry 3, Table 5),³⁵ at 75 °C the benzene conversion of 67% with 94% phenol selectivity (entry 4, Table 5),¹⁵ and at 50 °C, 70% cyclohexane conversion and 85% selectivity towards cyclohexanone (entry 5, Table 5),⁷⁶ and 57.5% toluene conversion with 84.4% selectivity of benzaldehyde. CuCr₂O₄ NPs can be reused several times without losing effectiveness (entry 6, Table 5).³⁹ Similarly, the effect of Cu(II) nanoclusters supported by CuCrO₄ spinel NPs on the selective oxidation of ethanol was investigated. Without a solvent, this process yielded 65% con-

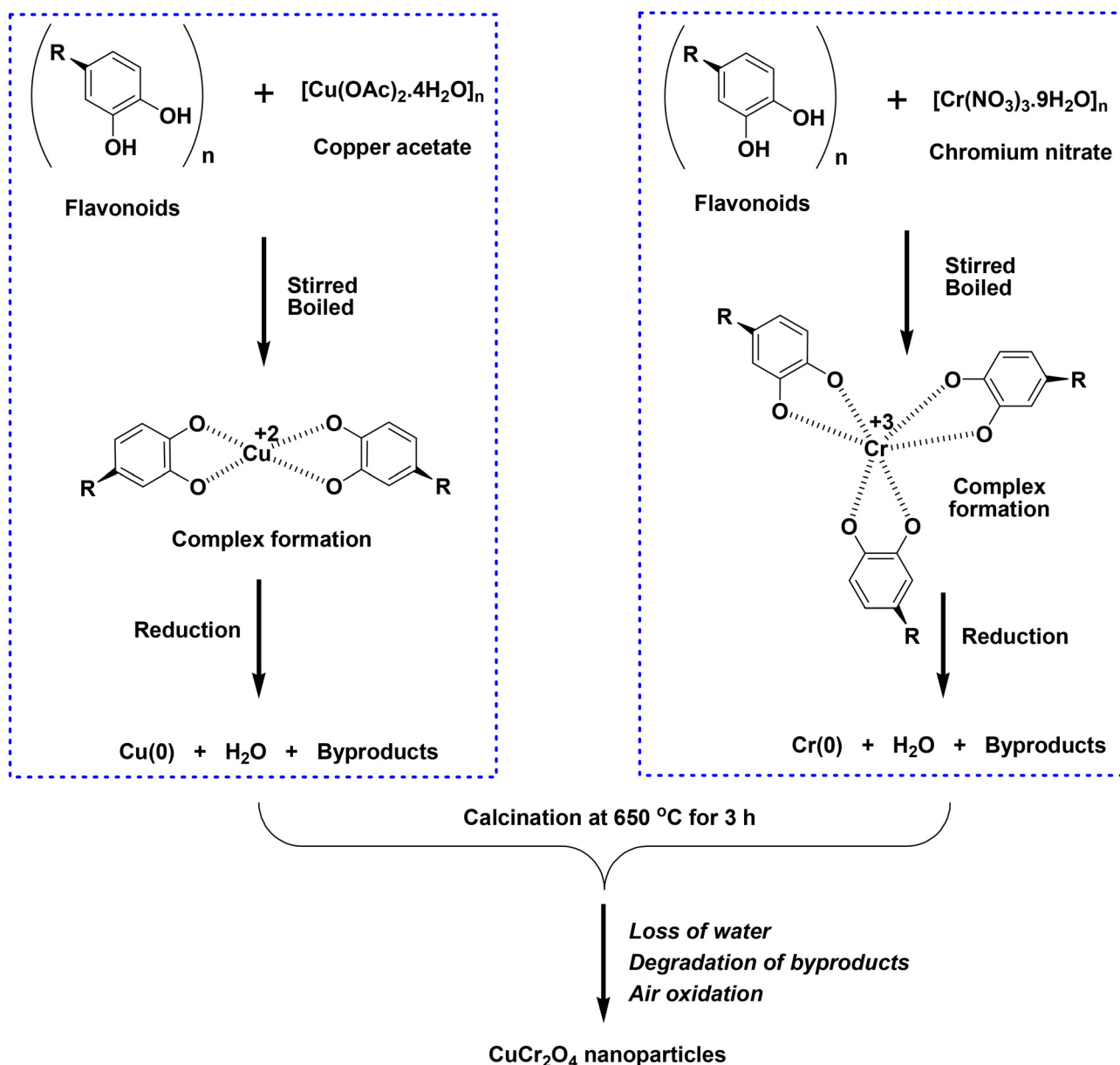
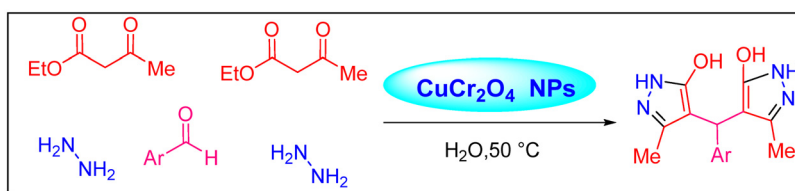


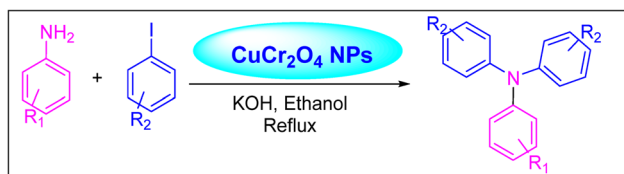
Fig. 16 Possible reaction mechanism behind the green synthesis of CuCr_2O_4 NPs using plant extract.



Scheme 1 Production of 4,4'-(arylmethanediyl)bis(3-methyl-1H-pyrazol-5-ol) analogues using catalytic amounts of CuCr_2O_4 NPs.⁶⁷

version of ethanol to acetic acid with 97% selectivity at 75 °C, utilizing H_2O_2 as the liquid-phase oxidant.⁹⁷ However, Table 5 describes the diverse organic transformations catalyzed by CuCr_2O_4 NPs.

In another study, Cu(II) nanoclusters supported on CuCr_2O_4 NPs were used to explore their catalytic activity in the sustained oxyamination conversion of benzene to aniline *via* a direct procedure employing NH_3 and H_2O_2 . The technique was



Scheme 2 Triarylamine synthesis using CuCr_2O_4 NPs.¹⁶

efficient in terms of activity, selectivity, mild reaction conditions, and catalyst. The Cu(II) component was found to significantly affect both the conversion rate and product selectivity.⁹⁶

Scheme 7 illustrates that ultra-small Cu(II) oxide supported on CuCr_2O_4 spinel NPs participates in converting benzene to phenol when air is used as an oxidant. The textural promoter Cr(III) prevents the sintering of Cu(II) oxide, thereby preserving the surface area of the catalyst during the reaction. In this process, the supported Cu(II) species is reduced by benzene to Cu(I) species **A**, which subsequently transitions into its canonical form **B**. Simultaneously, benzene generates a peroxide radical (HOO^\cdot) and a benzene radical cation ($\text{C}_6\text{H}_6^{+\cdot}$). The benzene radical cation then reacts with species **B** to form complex **C**, which subsequently converts into its canonical form **D**. After that, a proton is removed by complex **D**, restoring aromaticity and creating intermediate **E**. Phenol and Cu(II) oxide species **F** are produced when intermediate **E** dissociates. The interaction between species **F** and the concurrently generated hydroxyl radical ($^\cdot\text{OH}$) results in the production of intermediate **G**. The catalytic cycle is finished when a benzene molecule reduces **G** back to Cu(I) species, creating a benzene radical cation that rejoins the cycle.²⁴

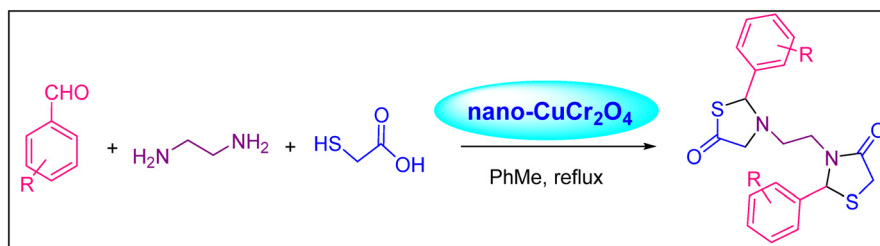
Chen *et al.* found that CuCr_2O_4 was the most effective catalyst, achieving a cyclohexane conversion rate of up to 95.8% and a cyclohexanone selectivity of 78.1%. The catalyst also showed good cycle stability after five cycles.¹³²

Abdolmohammadi's research shows that CuCr_2O_4 NPs have a larger surface area due to their acidic coordinated sites. CuCr_2O_4 NPs interact with the carbonyl oxygen group in (*E*)-1,2-diphenyl-1-diazene to generate an intermediate. This intermediate then undergoes a Knoevenagel reaction with 4-hydroxycoumarin, resulting in the formation of an alkene. Following that, a Michael-type addition occurs, in which another mole of 4-hydroxycoumarin interacts with the alkene. After cyclization and dehydration procedures, 7-(4-hydroxy-2-oxo-2*H*-[1]benzopyran-3-yl)-9-[(*E*)-2-(phenyl)-1-diazenyl]-6*H*,7*H*-[1]benzopyrano [4,3-*b*][1]benzopyran-6-one is produced (Scheme 8).⁴⁰

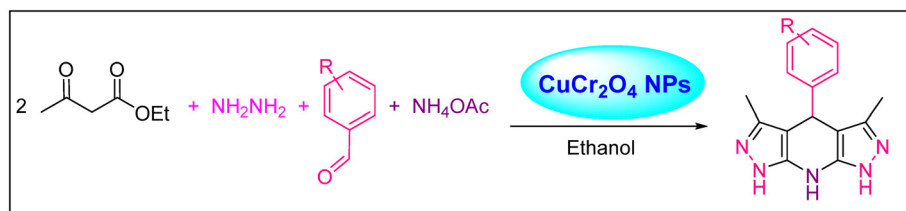
The long-term stability test results show that the catalyst exhibited excellent stability, even at 400 °C in the presence of CO_2 and water vapor. The calcination temperature is a key factor in improving the catalytic performance in the oxidation of CO .⁷⁷

Interestingly, a zinc oxide-doped, strongly acidic zeolite catalyst increased the formation of a toluene intermediate during the catalytic pyrolysis of cellulose. As shown in Fig. 17, $\text{CuCr}_2\text{O}_4@\text{MCM-41}$ exhibited strong catalytic activity. H_2O_2 , as an oxidizing agent, under optimal conditions of 60 °C and 4 h, resulted in 87.5% cresol selectivity and 83.0% conversion.¹⁵³

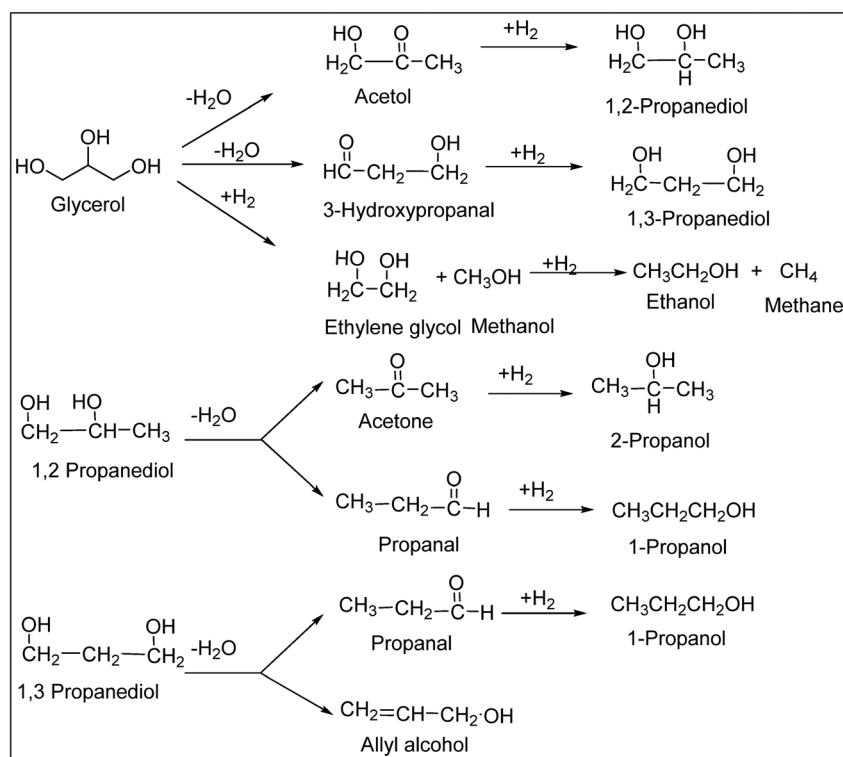
Scheme 9 depicts a multicomponent reaction involving 3,4-methylenedioxyphenol, aromatic aldehydes, and C-H-activated acidic compounds, such as dimedone. This method confirmed the universality of the route by producing high yields of xanthenones and chromenopyrimidinediones. Water was used as a green, non-volatile solvent, and ultrasonic irradiation was



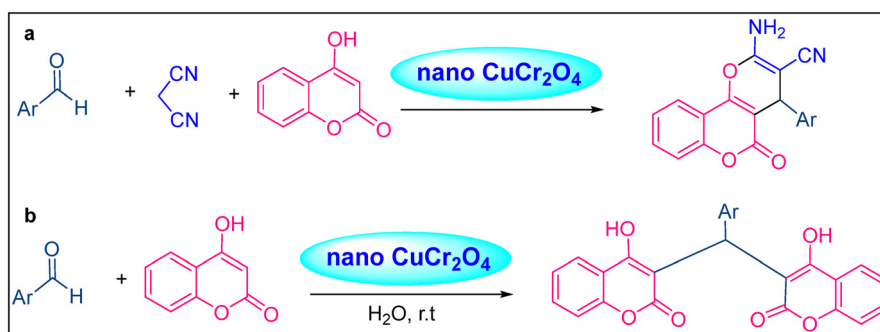
Scheme 3 Bis-thiazolidine synthesis using nano- CuCr_2O_4 catalyst.⁶⁸



Scheme 4 Preparation of tetrahydropyrazolopyridines using nano- CuCr_2O_4 .¹⁵²



Scheme 5 Possible pathways for the catalytic hydrogenolysis of glycerol.⁹²



Scheme 6 (a) Synthesis of dihydropyrano[c]chromene and (b) benzylidene bis(4-hydroxycoumarin).³⁸

employed as a safe energy source, enabling the quick synthesis of products.¹⁵⁴

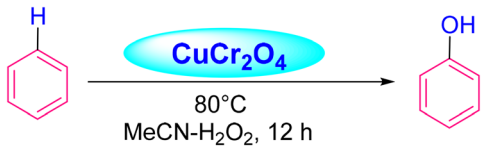
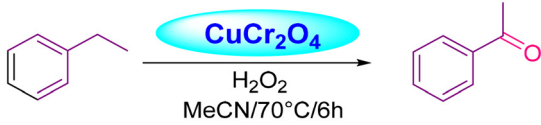
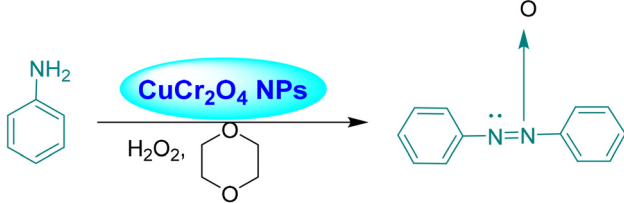
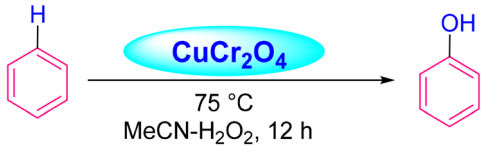
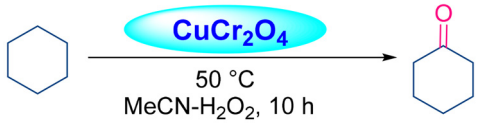
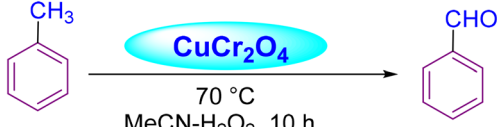
Kien and colleagues examined the fact that Pd may enhance the catalytic efficacy of spinel CuCr₂O₄ and Pd catalytic processes for the oxidation of carbon monoxide.¹⁰⁹ Using 0.100 g of catalyst, a temperature range of 30–450 °C, and a gas flow of 15 L h⁻¹, the study employed the temperature programmed surface reaction (TPSR) method to investigate a mixture of 1% CO, 20% O₂, and 79% N₂. The overall catalytic activity was considerably enhanced by distributing Pd NPs on CuCr₂O₄ NPs.¹⁰⁹ The core/shell NPs demonstrated promising catalytic activity for CO oxidation.¹¹¹ According to the researchers, HCOOH is more difficult to create than CH₃OH due to its more complex chemical structure. The conversion of CO₂ into

CH₃OH is most likely the predominant reaction. Furthermore, the selectivity of HCOOH and CH₃OH at 110–180 °C with calcined Cu/CuCr₂O₄ catalysts demonstrates alterations in the CO₂ conversion pathway.¹¹⁶

Shojaei *et al.* investigated the catalytic activity of CuCr₂O₄ and CuCr₂O₄/TiO₂ with an optimal chromite-to-TiO₂ molar ratio, catalyst amount, oxidant type and amount, and solvents. CuCr₂O₄-TiO₂ heterojunctions significantly enhance the catalytic activity in converting benzylic alcohols to aldehydes, particularly under microwave irradiation with *t*-BHP and H₂O₂ as oxidants.¹⁴⁶

As seen in Scheme 10, homolytic cleavage of the H₂O₂ molecules gives two ·OH radicals. The resulting radicals combine with Cr³⁺ and Cu²⁺ ions on the surface of CuCr₂O₄, producing

Table 5 Diverse organic transformations catalyzed by CuCr₂O₄ NPs

Entry	Reaction	Ref.
1		15, 74 and 78
2		75
3		35
4		15
5		76
6		39

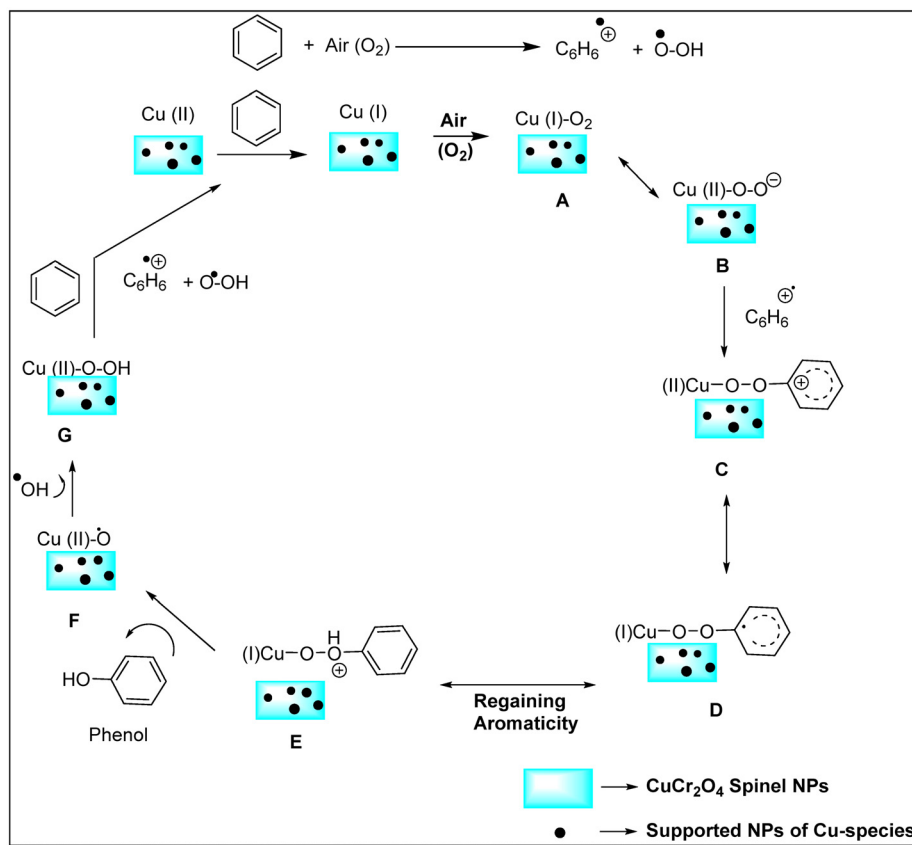
Cu²⁺ and Cu⁺ ions, respectively, as well as hydroperoxyl. The hydroperoxy radicals also further react with hydroxyl radicals on the surface, producing water and oxygen. Veratryl alcohol (VA) molecules are absorbed into the surface of CuCr₂O₄, generating metal-alkoxide intermediates and adsorbing hydrogen through O–H bond breakage. The surface O–H group is generated when the adsorbed hydrogen bonds with the surface lattice oxygen and coordinates with the carbon atom of the methyl group in the VA molecule. After activating the C–H bond in the β site, veratraldehyde and water are formed.¹⁰¹

Ghasemi *et al.* utilized CuCr₂O₄ NPs, along with a basic ionic liquid, to fabricate cyclopropanecarboxylic acids. They fabricated (\pm)-*trans*-2-phenylcyclopropanecarboxylic acid using *trans*-cinnamic acid as the starting material, along with the NPs. They utilized various solvent forms, but THF gave the optimal results (Scheme 11).¹⁵⁵

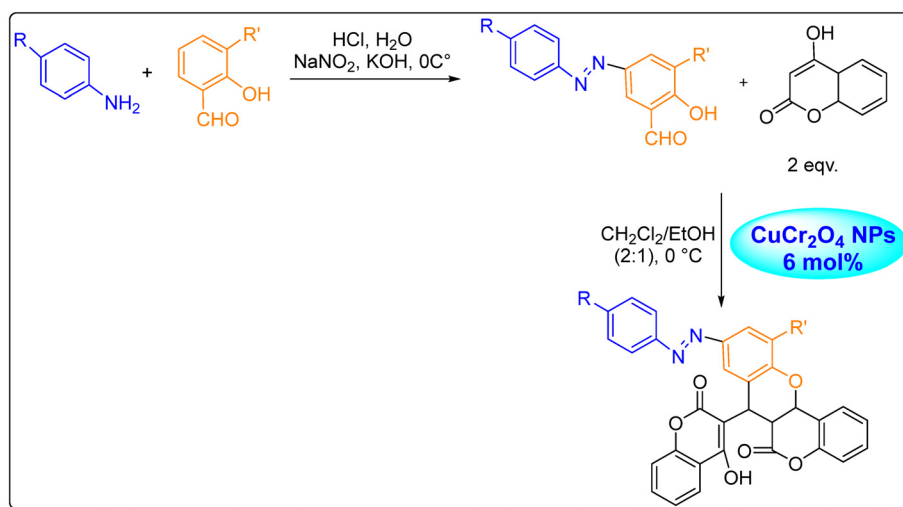
Barman *et al.* presented a method for hydrogenating acetone using a catalyst. Firstly, chromite reversibly reacts with hydrogen, resulting in the formation of a decreased spinel and the release of copper as surface-bound particles. Acetone adsorbs onto Cu⁰ atoms as π -complexes, getting electrons from

the metal and a proton from chromite, resulting in negative charges. The oxidised copper returns to the spinel. A second proton is injected, and a proton from the acidic medium combines with the OH groups to produce a carbonium ion and water. This ion conducts electrophilic substitution with benzene to form cumene (Scheme 12).¹³⁸ As depicted in Fig. 18, the catalytic performance of CuCr₂O₄ NPs for various organic transformations was demonstrated.

Patil and colleagues studied the thermal decomposition of p-type nano-CuO and CuCr₂O₄ using AP. The DSC analysis concluded that the electrochemical technique produced NPs with better catalytic effects on HTD and AP. Nano-CuCr₂O₄ outperformed nano-CuO in reducing high-temperature decomposition by 118 °C at a 2 wt% concentration. Nano-CuO and CuCr₂O₄ produced significant heat emissions of 5.430 and 3.921 kJ g⁻¹, respectively.¹⁵⁶ A similar investigation was conducted by Shukri Ismael *et al.* They proposed that the transition metal Cr³⁺ in CuCr₂O₄ exhibits hole conductivity due to its partially filled outermost d-orbitals. It rapidly takes electrons released by ClO₄⁻, generating Cr²⁺, which then migrates to the surface of the catalyst and reacts with NH₄⁺ to generate



Scheme 7 Possible process for phenol production over ultra-small Cu(II) based on CuCr_2O_4 NPs.²⁴



Scheme 8 Formation of [1]benzopyran azo dyes catalyzed by CuCr_2O_4 NPs.⁴⁰

ammonia and hydrogen atoms. As a result, the AP ion and its intermediate products can absorb electrons, accelerating the thermal deterioration of AP (Fig. 19).⁷⁹

Ashraf *et al.* reported that copper chromite was efficiently incorporated into an ammonium nitrate (AN) matrix. The possible effect of copper chromite on the thermal behavior and

decomposition enthalpy of AN was examined using TGA and DSC. Copper chromite had a considerable impact on the primary breakdown process of AN. AN decomposition was entirely transformed from a strong endothermic reaction ($+1554 \text{ J g}^{-1}$) to a strong exothermic reaction (-1625 J g^{-1}).⁹³ Nano- CuCr_2O_4 noticeably enhances the degradation tempera-

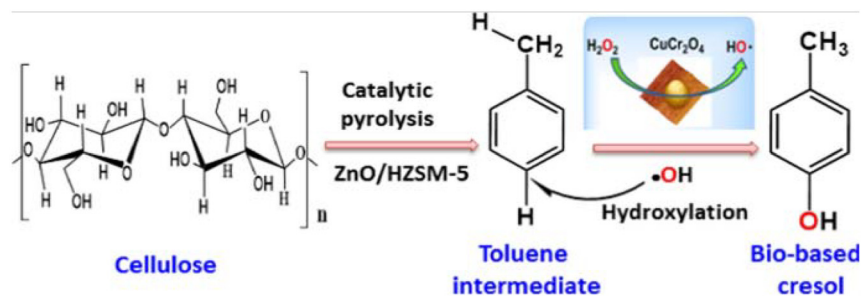
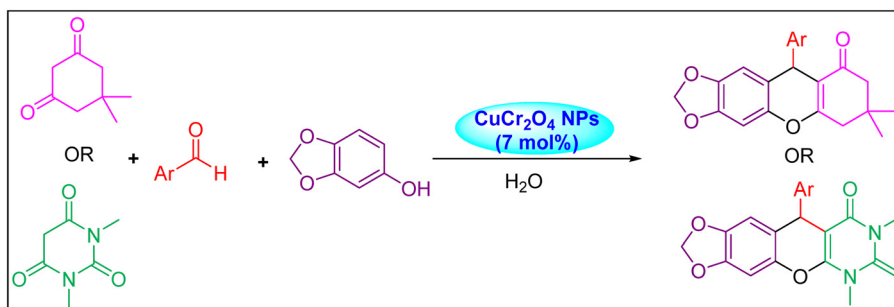
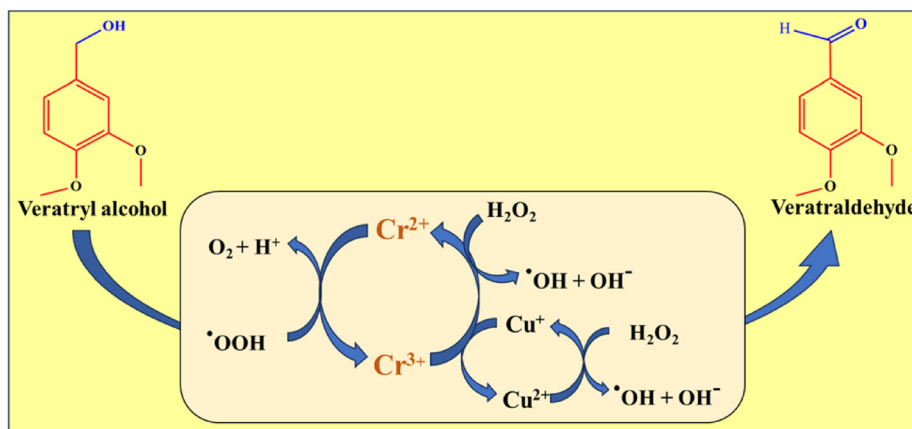


Fig. 17 CuCr_2O_4 @MCM-41 catalyst used for the efficient catalytic pyrolysis of cellulose and catalytic oxidation.¹⁵³



Scheme 9 Ultrasound-based, green, and sustainable method for xanthenone and chromenopyrimidinedione derivatives.¹⁵⁴

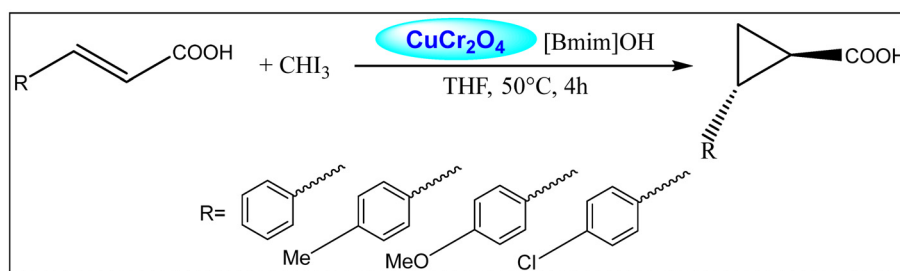


Scheme 10 Oxidation of VA to veratraldehyde.¹⁰¹

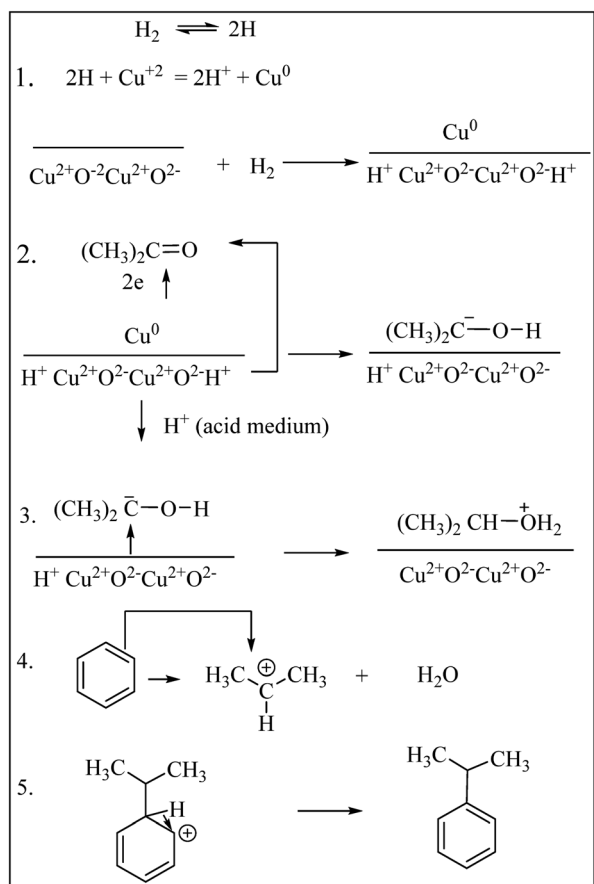
ture of AP/HTPB (ammonium perchlorate/hydroxy-terminated polybutadiene propellant) and AP/CMDB (ammonium perchlorate/composite modified double-base propellant), hence increasing the apparent heat of degradation. Furthermore, the burning rates of nano- CuCr_2O_4 -AP/HTPB and nano- CuCr_2O_4 -AP/CMDB propellants increased to 1.60 mm s^{-1} and 3.23 mm s^{-1} , representing an increase of 28.3% and 26.3%, respectively.¹⁹

The TGA/DSC study verified that nano CuCr_2O_4 had a higher catalytic effect on ammonium dinitramide (AND) than nano CuO . Fuyao Chen and team believed that this could be because the catalytic activity of the two NPs differed for the

preliminary decomposition product (AN) of ADN. They also conducted an ignition test on raw ADN and several copper-based catalyst/ADN composites. Raw ADN produced only a small amount of smoke at 200 ms, with the most intense decomposition time at 514 ms. The entire decomposition process of four distinct catalysts/ADN composites took less than 500 ms, indicating that copper-based catalysts can greatly facilitate ADN decomposition, and nano CuCr_2O_4 had a stronger catalytic effect on ADN decomposition than nano CuO . Moreover, researchers explored the adsorption capacity for NH_3 on the selected typical crystal face of nano CuCr_2O_4 (211) and the typical crystal faces of nano CuO (002) and (200). The



Scheme 11 Reaction of functionalized *trans*-cinnamic acids *via* cyclopropanation.¹⁵⁵



Scheme 12 Mechanism of reductive isopropylation of benzene with acetone.¹³⁸

(211) crystal face of nano CuCr_2O_4 showed the greatest negative value of E_{ads} among the three models, implying that nano CuCr_2O_4 has stronger adsorption ability to the NH_3 molecule than that of nano CuO , which may be the primary explanation for the excellent catalytic performance of nano CuCr_2O_4 for AN (Fig. 20).⁸⁴

Li *et al.* proposed that the efficient phase interaction between spinel CuCr_2O_4 and delafossite CuCrO_2 is present in Cu-Cr-O NCs. The catalyst with Cu/Cr of 0.7 shows excellent catalytic performance, a low-pressure exponent (0.43), and a high burning rate at all pressures.⁵⁷ Hosseini *et al.* found that

adding CuCr_2O_4 NPs to pure AP decreases its decomposition temperature by 56 °C to 84 °C and increases the apparent heat released by 516 to 608 J g^{-1} .⁵⁰ Several studies have shown that AP decomposes at high temperatures.^{85,90,142} CuCr_2O_4 NPs demonstrated remarkable catalytic activity on ultrafine AP, as revealed by the lowest high-temperature decomposition temperature and Gibbs free energy of the nano- CuCr_2O_4 /ultrafine AP composite generated *via* ball milling for 6–12 h.⁸⁵

Wang *et al.* investigated the catalytic effects of chromate NPs on the thermal decomposition of FAP and CL-20. The inclusion of chromate nanoparticles significantly decreased the decomposition temperature of FAP. Specifically, the produced CuCr_2O_4 NPs reduced the decomposition temperature of FAP by 57.7 °C, demonstrating that CuCr_2O_4 has significantly greater catalytic effectiveness than other chromates.⁵⁶ Viswanath *et al.* studied nano CuCr_2O_4 as a ballistic modifier in a typical solid rocket propellant (SRP) composition. According to the report, 17.5% CuO was required to achieve the desired phase for its application as a potential ballistic modifier. This excess CuO in CuCr_2O_4 enhances the burn rate of the typical SRP.⁶⁵ The addition of 5 wt% Cu-Cr-O-0.7TiO_2 -rGO catalyst to AP considerably improved the thermal decomposition. Compared to the industrial catalyst (activated CuCr_2O_4), it showed a 133.61% greater solid composite propellant (SCP) burn rate. With 0.6% catalyst in AP, the burn rate increased from 4.866 mm s^{-1} (pure AP) to 8.531 mm s^{-1} , exceeding that of the industrial catalyst of 6.385 mm s^{-1} at 33 bar and displaying good ballistic performance.¹⁴⁵ The superior catalytic performance of metal-doped CuCr_2O_4 can be attributed to the presence of mixed active sites, including Co^{2+} and Ba^{2+} sites, as well as Cu^{2+} and Cr^{2+} sites. The mechanism indicates that the interaction between the catalyst and AP, which benefits from more active metal sites in the doped catalyst, demonstrates significant potential for AP decomposition. Furthermore, factors such as pore volume and the presence of active sites, as presented by doping, are also critical in influencing the catalytic performance.⁹⁵

5.2. CuCr_2O_4 nanomaterials as photocatalysts

Environmental issues, notably water pollution, have received much attention in the last few years.¹⁵⁷ One of the main types of contaminants in wastewater is dyes.^{158,159} Wastewater containing dyes has a significant impact on humans, animals,

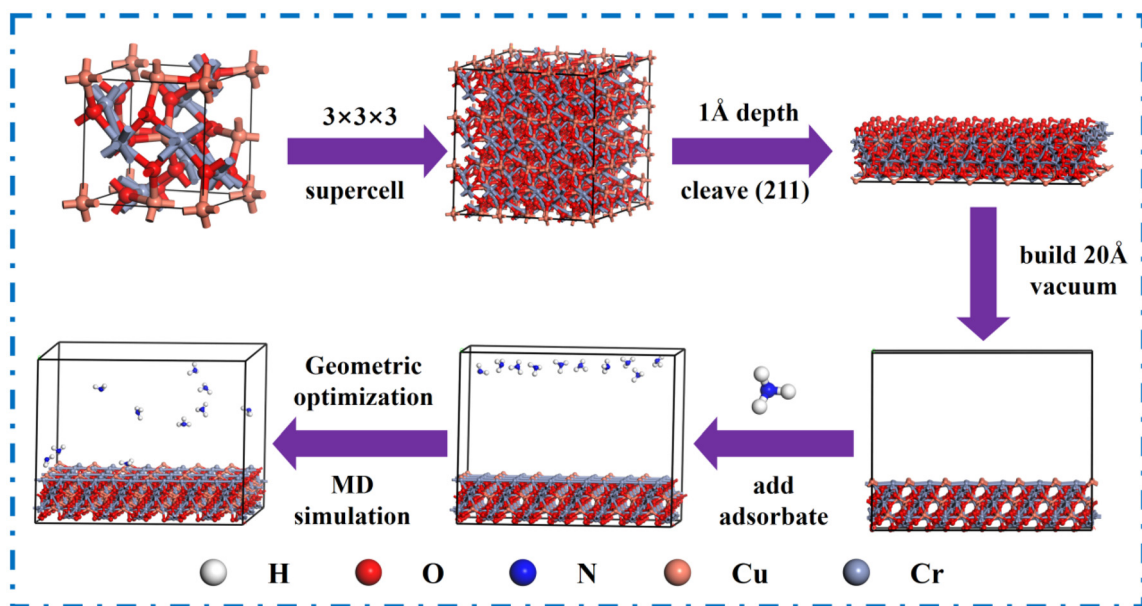


Fig. 20 Schematic of the calculation and modeling process of adsorption heat between nano CuCr_2O_4 and NH_3 .⁸⁴

for photocatalytic activity and significantly accelerates the degradation rate.¹⁶² Paul *et al.* proposed that CuCr_2O_4 NPs act as photocatalysts by absorbing solar energy, exciting electrons from the valence band (VB) to the conduction band (CB) and leaving holes in the VB. Dye is oxidized into innocuous chemicals by these holes, and hydroxyl radicals (OH^\bullet) are produced through the reduction of H_2O_2 . The high bandgap energy and vast surface area of CuCr_2O_4 NPs promote the formation of electron–hole pairs, while inhibiting recombination, significantly increasing the photocatalytic efficiency.²³

Pan *et al.* suggested that a pH of 9 plays a crucial role in the catalytic process of the samples. At greater concentrations, *p*-nitrophenol was successfully destroyed by 0.3 g L^{-1} of CuCr_2O_4 calcined at 400°C , with excellent reproducibility.⁷¹ Yuan *et al.* observed photocatalytic activity using Rhodamine B (RhB), methyl orange (MO), and methylene blue. The absorption peaks of RhB (554 nm) and MB (664 nm) decreased significantly with an increase in the irradiation time and almost disappeared by 60 min. In contrast, the characteristic absorption peak of MO at 464 nm only slightly decreases with an increase in exposure time.²²

Soleimani and team investigated the degradation of malachite green (MG) using CuCr_2O_4 and $\text{Mn}_{0.5}\text{Cu}_{0.5}\text{Cr}_2\text{O}_4$. The results show that $\text{Mn}_{0.5}\text{Cu}_{0.5}\text{Cr}_2\text{O}_4$ is more effective than CuCr_2O_4 . In the photodegradation process, morphology and surface area significantly influence the efficiency. CuCr_2O_4 exhibits an agglomerated shape and a lower surface area, which results in reduced photodegradation capability. The $\text{Mn}_{0.5}\text{Cu}_{0.5}\text{Cr}_2\text{O}_4$ sample exhibits lower overall electronegativity than CuCr_2O_4 , which affects its band gap and facilitates easier electron transitions between the conduction and valence bands.⁸⁰

Fig. 21 illustrates the predicted photocatalytic mechanism for the decomposition of tartrazine azo dye. Under visible light

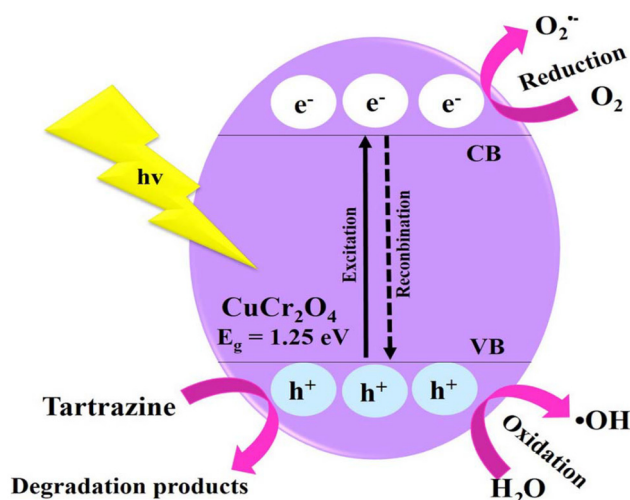
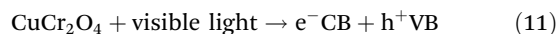
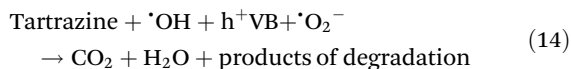


Fig. 21 Proposed mechanism for photocatalytic degradation of a dye.⁶⁴

($\geq 1.25 \text{ eV}$), CuCr_2O_4 generates electron–hole pairs (eqn (11)). Electrons react with oxygen to form O_2^\bullet (eqn (12)). At the same time, holes in the valence band interact with H_2O to produce OH^\bullet radicals (eqn (13)). These reactive species, especially OH^\bullet and h^+ , break down the azo dye into CO_2 , H_2O , phenols, aromatic amines, and acids such as oxalic, tartaric, and malic (eqn (14)).⁶⁴





Peymanfar *et al.* proposed that photocatalytic degradation under both visible and dark light conditions performed better than expected, suggesting that the capped CuCr_2O_4 structures and the produced CuCr_2O_4 function as photocatalytic materials rather than adsorbents.⁵¹

The impacts of pH, catalyst dosage, and initial concentration on photocatalytic decomposition were examined by Benrighi *et al.* They found that dye degradation was significantly influenced by the pH of the solution, with the best degradation taking place at pH values close to 4.0. Dye degradation decreases significantly as the pH increases from 4.0 to 10. This reduction can be related to the negative charge that anionic dyes acquire at higher pH values. It might impede degradation by repelling them from the surface of the photocatalyst due to charge repulsion.⁵²

Han *et al.* examined the photocatalytic degradation of Congo red dye and antibiotics, including TCH, metformin hydrochloride (MFH), oxycline hydrochloride, and naproxen sodium (NPS). CuAl_2O_4 oxides reveal high photocatalytic performance in TCH and OCH, while MFH and NPS exhibit poor photocatalytic activity.⁸⁶ The electronic band structure (Fig. 22a and c) and density of states (DOS) of nanomaterials were computed using the DFT function. The Fermi values of CuCr_2O_4 and BiI_3O_9 were 4.32 and 1.41 eV, according to Fig. 22b and d, respectively. The DOS analysis of pure CuCr_2O_4 and BiI_3O_9 revealed electron excitation at different orbitals. $\text{CuCr}_2\text{O}_4\text{-BiI}_3\text{O}_9\text{-10\%}$ NCs outperformed other concentrations

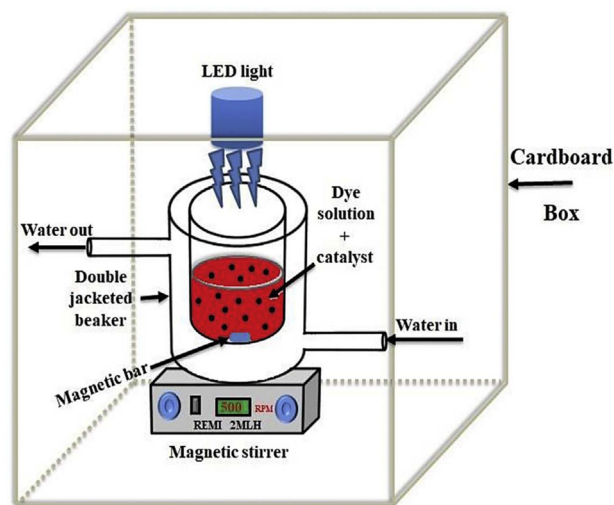


Fig. 23 Layout of the dye degradation setup.¹¹²

and pure CuCr_2O_4 and BiI_3O_9 NPs in terms of photocatalytic activity. The efficient photodegradation is attributed to the effective generation of heterojunctions, improved charge carrier separation and migration, and a wide surface area. OH^\cdot significantly contributed to cefixime (CEF) deterioration. The proposed Z-scheme is effective in photodegrading CEF.⁴⁵

Ghorai *et al.* studied the photocatalytic performance of a $\text{CuCr}_2\text{O}_4/\text{BiOBr}$ nanocomposite for degrading RhB and tetracycline hydrochloride (TC-HCl) under household LED light, as shown in Fig. 23.¹¹²

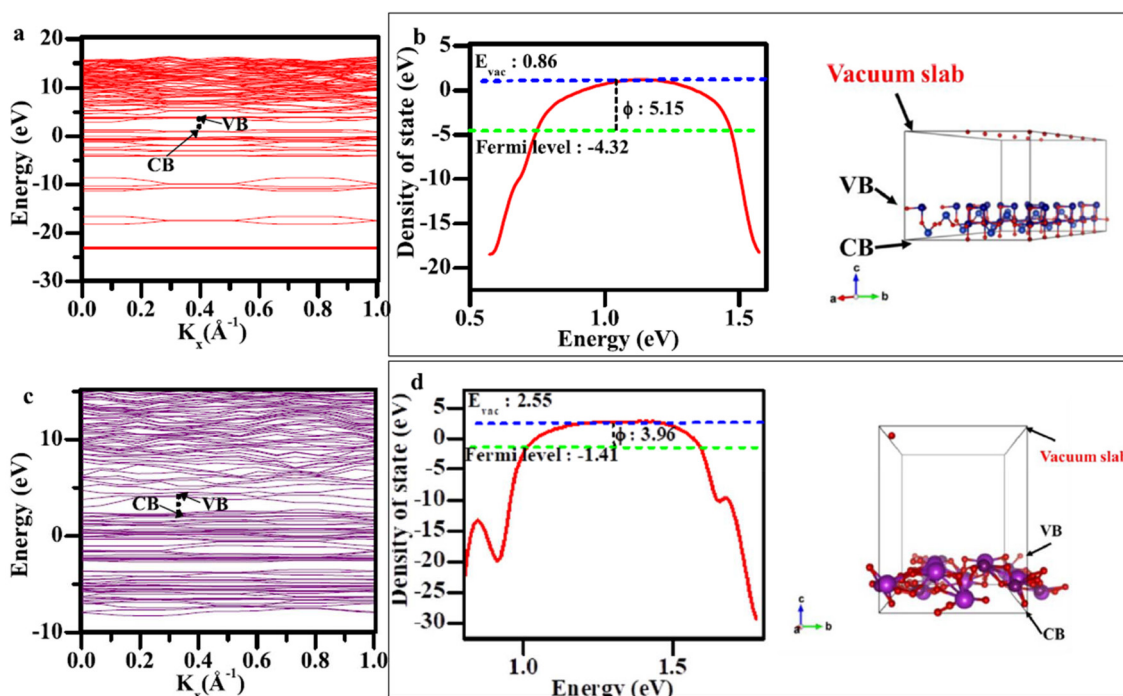


Fig. 22 Electronic band structure, work function, and atomic arrangement of CuCr_2O_4 (a and b) and BiI_3O_9 NPs (c and d).⁴⁵

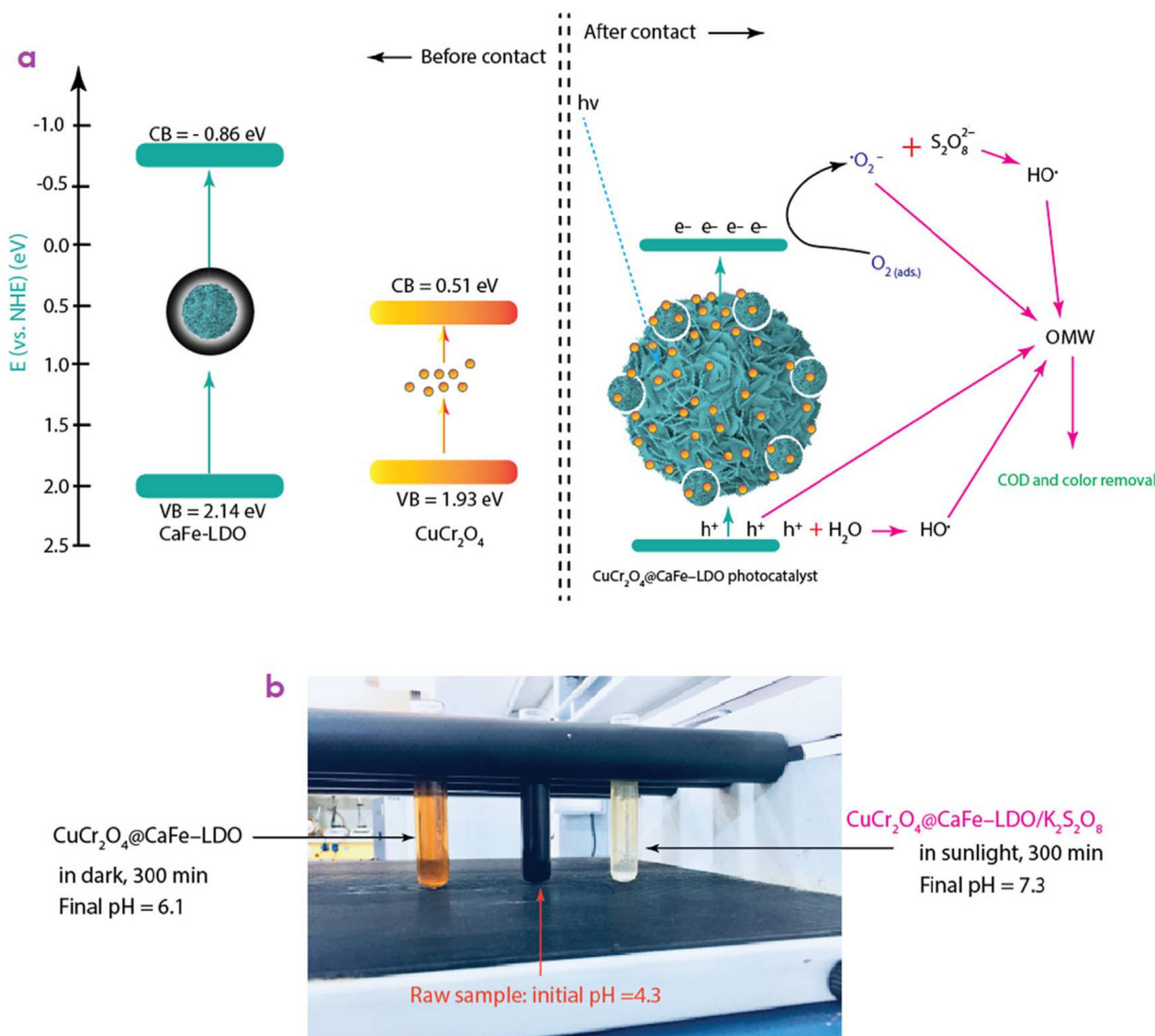


Fig. 24 Probable reaction pathway of the $\text{CuCr}_2\text{O}_4@/\text{CaFe-LDO}/\text{K}_2\text{S}_2\text{O}_8/\text{sunlight}$ system. (b) Illustration of the catalyst performance under both dark and light conditions.¹¹³

The $\text{BiVO}_4\text{-CuCr}_2\text{O}_4$ (1 : 0.25) nanocomposite exhibits $\sim 90\%$ photocatalytic efficiency for MB dye degradation under visible light, which increases with PANI addition. This enhancement is attributed to the improved charge separation of electron-hole pairs, further strengthened by the incorporation of PANI.¹⁴¹

According to Oladipo *et al.*, 1.0 g $\text{CuCr}_2\text{O}_4@/\text{CaFe-LDO}$ removed 66% COD without an oxidant after 300 min in the dark. However, after 180 min of reaction, the $\text{CuCr}_2\text{O}_4@/\text{CaFe-LDO}/\text{K}_2\text{S}_2\text{O}_8/\text{sunlight}$ system removed $\sim 99\%$ and 98.3% COD and colour, respectively. The crude OMW contained seven phenolic compounds, the most abundant of which were hydroxy tyrosol (76.84%) and tyrosol (15.14%) [Fig. 24].¹¹³ Without an oxidant, $\text{CuCr}_2\text{O}_4@/\text{CaFe-LDO}$ degraded phenol by 54.8% after 4 h in the dark. Within 40 min, the deterioration reached 90% when exposed to sunshine and 0.05 M $\text{K}_2\text{S}_2\text{O}_8$. After 120 min, the $\text{CuCr}_2\text{O}_4@/\text{CaFe-LDO}/\text{K}_2\text{S}_2\text{O}_8$ system, with UV assistance,

achieved 99% phenolic degradation, 95.1% COD removal, and 84.6% BOD removal.¹¹⁴

It was discovered that the 10% $\text{CuCr}_2\text{O}_4\text{-CeO}_2$ nanocomposite sample exhibited a remarkable photo-Fenton performance. Tests using coumarin fluorescence probes and radical trapping verified that $\cdot\text{OH}$ is the main factor causing RhB degradation. Visible LED light promotes the production of $\cdot\text{OH}$ during the Fenton-like reaction more efficiently than under dark conditions. The favorable redox reaction between $\text{Ce}^{4+}/\text{Ce}^{3+}$ and $\text{Cu}^{2+}/\text{Cu}^+$ couples triggers a dark Fenton-like reaction. However, the low-molecular-weight compounds produced during degradation accumulate on the active sites of the catalyst, reducing its reusability. Fig. 25 illustrates the photo-Fenton reaction mechanisms.¹¹⁵

The degradation of MO and MB dyes followed a pseudo-first-order kinetic model.¹²⁷ Mageshwari *et al.* analyzed the time-

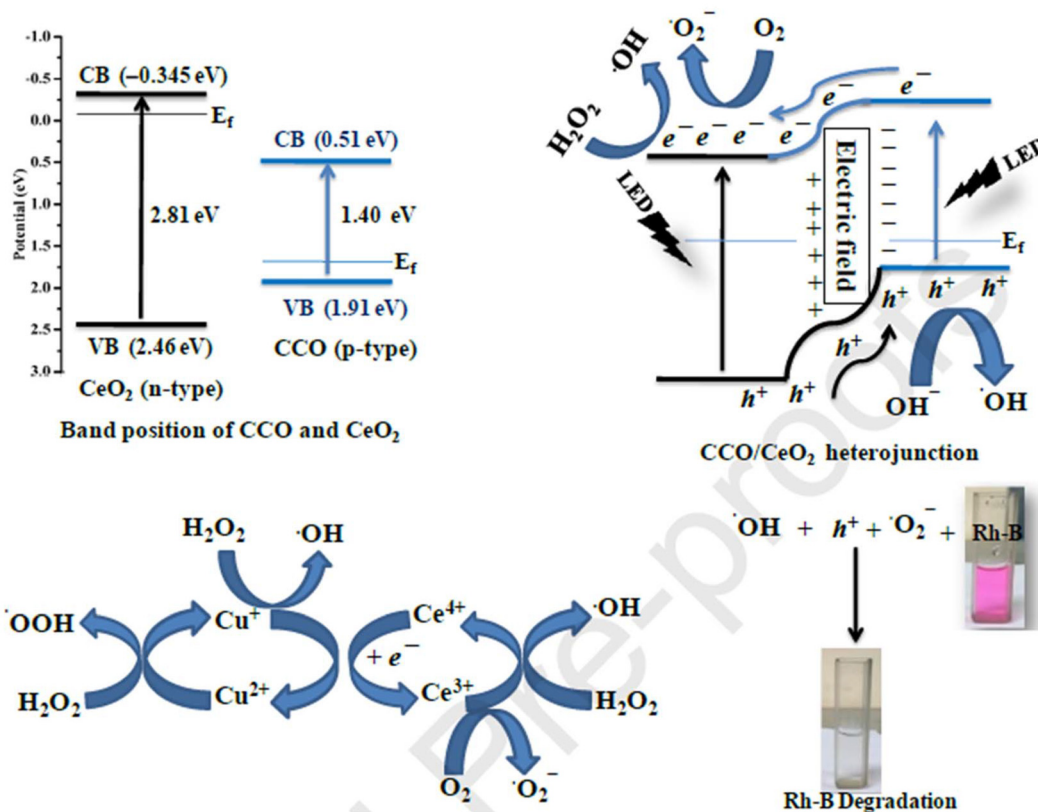


Fig. 25 Photo Fenton-like reaction process of $\text{CuCr}_2\text{O}_4\text{-CeO}_2$ NCs.¹¹⁵

dependent UV-vis absorption spectra of MO and MB in the presence of CuO and $\text{CuO-CuCr}_2\text{O}_4$ (20% Cr^{3+}). After 270 min with CuO and 145 min with $\text{CuO-CuCr}_2\text{O}_4$, the MO absorption peak at 464 nm, which was associated with the azo ($-\text{N}=\text{N}-$) bond, disappeared. Likewise, after 360 min with CuO and 180 min with $\text{CuO-CuCr}_2\text{O}_4$, the MB peak at 665 nm disappeared. The time-dependent UV-vis absorption spectra in Fig. 26 demonstrate the effective degradation of MO and MB dyes by the CuO and $\text{CuO-CuCr}_2\text{O}_4$ samples. The dyes gradually lost color, indicating their complete degradation. In the case of MO, the peak at 464 nm disappeared after 270 min with CuO and 145 min with $\text{CuO-CuCr}_2\text{O}_4$. In the case of MB, the 665 nm peak vanished after 360 min with CuO and 180 min with $\text{CuO-CuCr}_2\text{O}_4$, indicating that the $\text{CuO-CuCr}_2\text{O}_4$ sample exhibited superior photocatalytic activity compared to pure CuO.²⁶ The photocatalytic efficacy of $g\text{-C}_3\text{N}_4\text{-NS/CuCr}_2\text{O}_4$ NCs was evaluated by degrading MB and RhB dyes and phenol under visible light. The nanocomposite performed best with a 10 wt% CuCr_2O_4 loading.¹¹⁹

Mohamed *et al.* investigated $\text{CuCr}_2\text{O}_4\text{-g-C}_3\text{N}_4$ photocatalysts for photocatalytic hydrogen generation utilizing glycerol under visible light illumination (Fig. 27). Their findings revealed that the CuCr_2O_4 content in the heterojunction had a substantial impact on photocatalytic performance, with a 3 wt% composition being the optimal. At this ideal level, the 3 wt% $\text{CuCr}_2\text{O}_4\text{-g-C}_3\text{N}_4$ heterojunctions produced thirty-times more hydrogen from glycerol than pure $g\text{-C}_3\text{N}_4$.¹¹⁷

According to the results, when exposed to simulated sunlight, pure CuCr_2O_4 or TiO_2 showed a weak photocatalytic performance for generating hydrogen from an aqueous oxalic acid solution. However, $\text{CuCr}_2\text{O}_4/\text{TiO}_2$ heterojunction coupling showed noticeably higher photocatalytic activity. A mass concentration of 0.8 g L^{-1} exhibited promising hydrogen evolution capability when calcined at 500°C .¹⁴⁴ Baoum *et al.* investigated the bandgap energy of CuCr_2O_4 and $\text{PANI@CuCr}_2\text{O}_4$ composites using UV-vis spectroscopy. The bandgap values for pure CuCr_2O_4 and 0.02%, 0.04%, 0.06%, and 0.08% $\text{PANI@CuCr}_2\text{O}_4$ were 2.12, 2.00, 1.92, 1.73, and 1.72 eV, respectively. The outcomes indicate that the incorporation of PANI reduces the bandgap, thereby enhancing the photocatalytic activity of the nanocomposites.¹²³ Daimalah *et al.* demonstrated the significance of pH and dose in photocatalytic activity. An increase in catalyst dosage resulted in a higher abatement rate. Furthermore, as the pH decreased from 9 to 3.5, the cefixime (CFX) removal efficiency improved from 30% to 94%.¹³¹

Ghorai *et al.* investigated the photocatalytic activity of dyes and antibiotics, yielding promising results. As demonstrated in Fig. 28, radical trapping experiments revealed that in the PFL reaction, dye abatement decreased significantly with IPA and AgNO_3 , confirming that $\cdot\text{OH}$ radical and excited electrons are the main reactive species, respectively. Na_2EDTA and BQ had minimal effects, indicating the limited roles of

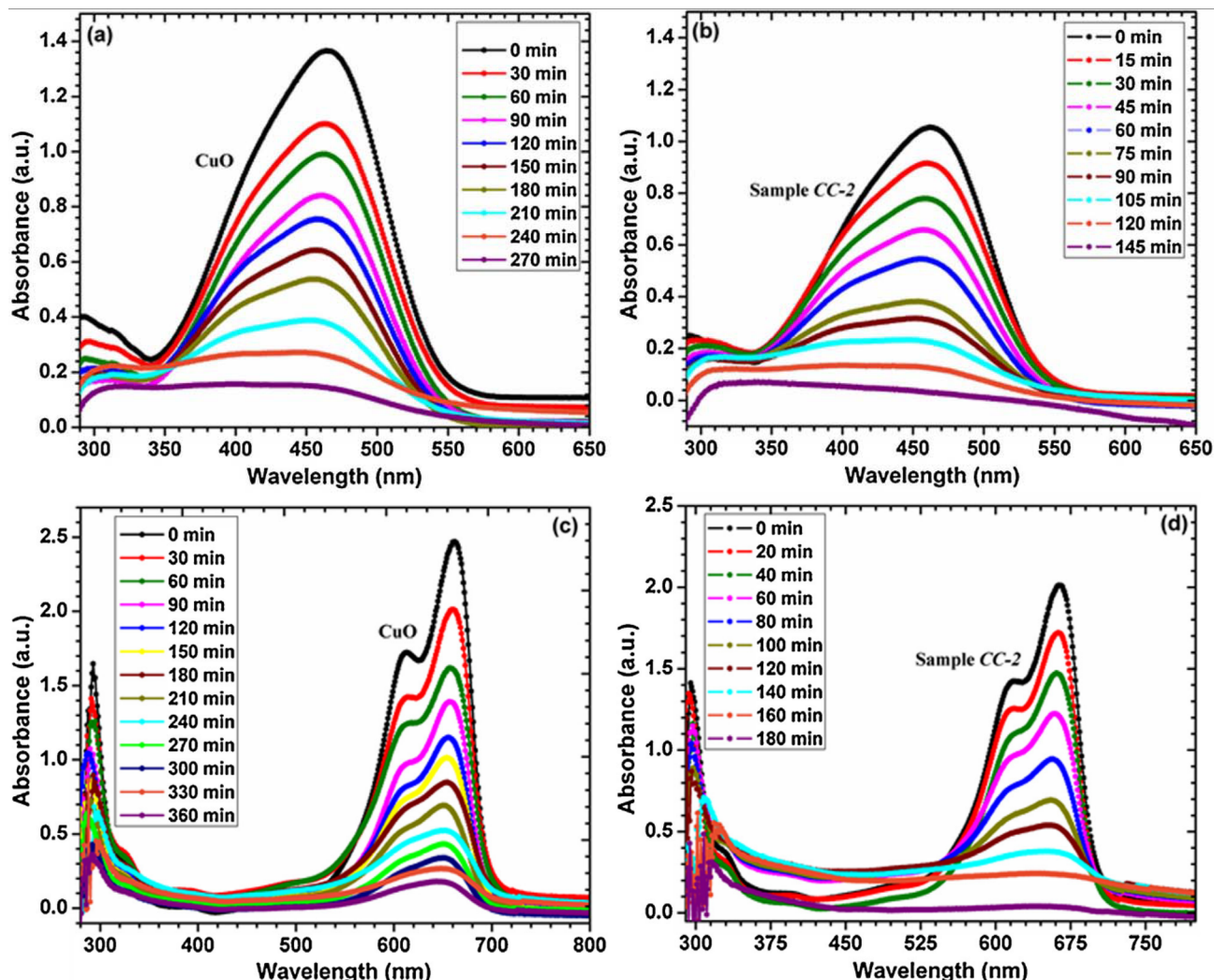


Fig. 26 Time-dependent UV-vis absorption spectra of (a and b) MO and (c and d) MB dye solutions in the presence of CuO and CuO-CuCr₂O₄ (20% Cr³⁺ concentration) samples under UV irradiation.²⁶

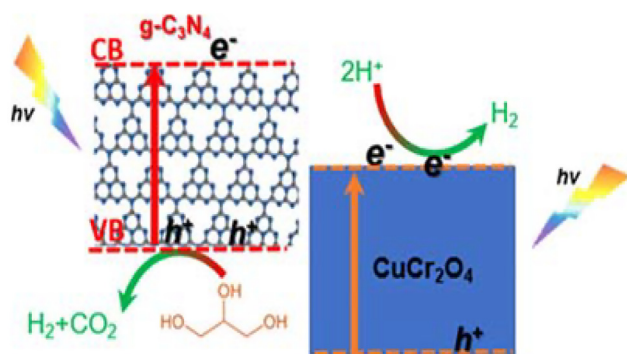


Fig. 27 Proposed mechanism for H₂ gas generation using the CuCr₂O₄-g-C₃N₄ heterojunction.¹¹⁷

holes and superoxide species, respectively. In the DFL reaction, the degradation activity decreased significantly in the presence of IPA.⁴⁸

Sivaranjani *et al.* stated that increasing the number of photons per unit area with an increase in light intensity enhances the deterioration. Furthermore, the photocatalytic process exhibits a positive relationship with pH, with the highest degradation occurring at a pH of 7.0. Degradation is reported to diminish above pH 7.0 because of the reaction between the $\cdot\text{OH}$ and h^+ generated by the semiconductor. Compared to other anions such as Cl^- , HCO_3^- , CH_3^- , and NO_3^- , the presence of SO_4^{2-} anions was considered to cause the most deterioration. The bonding behavior of the complexes and the contribution of molecular orbitals (HOMO and LUMO) were predicted by DOS plots.¹¹⁸

As shown in Fig. 29, the alizarine red dye molecule absorbs light energy, exciting electrons in V₂O₅/CuCr₂O₄-MCM-41, which generates ROS, such as $\cdot\text{OH}$. These $\cdot\text{OH}$ groups interact with alizarine red molecules, breaking the dye into smaller, non-toxic components such as CO₂, SO₄²⁻, and H₂O.¹³⁵

Ramezanalizadeha *et al.* highlighted the synergistic involvement of La in improving the photocatalytic activity. Notably, CuCrLaO₄ demonstrated remarkable degradation of both RhB

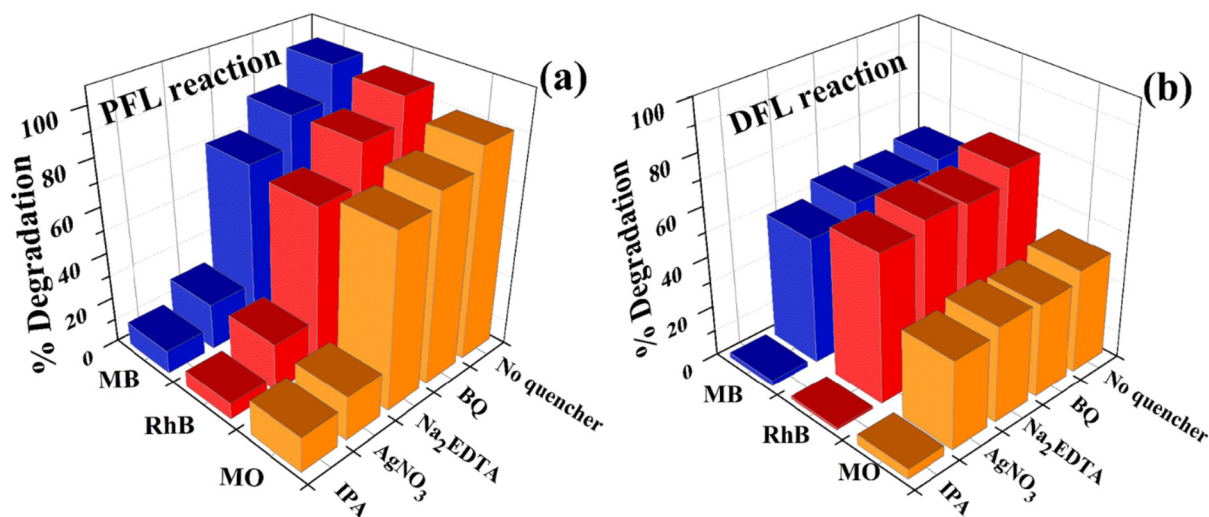


Fig. 28 Impact of various radical quenchers on the PFL and DFL activity of the 100% $\text{CuCr}_2\text{O}_4/\text{TiO}_2$ nanocomposite for MB, RhB, and MO degradation.⁴⁸

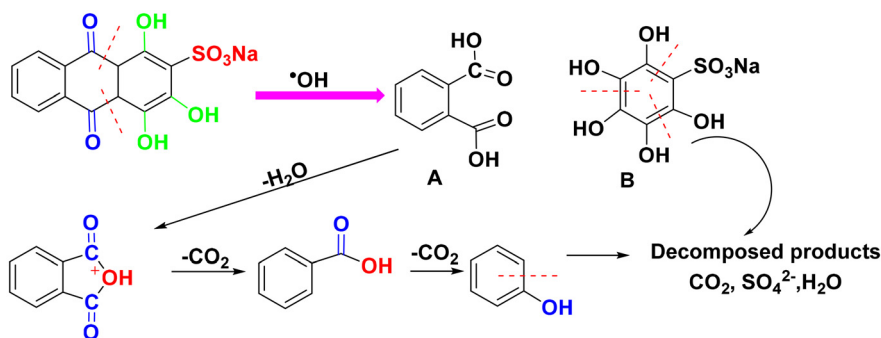


Fig. 29 Plausible mechanism for the decomposition of Alizarin Red dye.¹³⁵

and MO dye.¹⁰⁰ Hosseini *et al.* investigated the photocatalytic activity of CuCr_2O_4 against *p*-nitrophenol contamination. The CuO content of the samples considerably influenced their photocatalytic performance. The findings demonstrate that the Cu/Cr molar ratio has a significant impact on the photocatalytic efficiency of the samples.¹³⁶

Al-Wasidi *et al.* studied the degradation of Acid Orange 7 dye using a CuCr_2O_4 photocatalyst (Fig. 30). Under visible light, the copper chromite photocatalyst absorbs energy equivalent to or greater than its band gap, resulting in electron-hole (e^-/h^+) pairs on its surface. The photogenerated electrons combine with adsorbed oxygen molecules to form superoxide radicals ($\text{O}_2^{\cdot-}$), and holes react with water molecules to produce hydroxyl radicals ($\cdot\text{OH}$). These reactive species, particularly hydroxyl radicals and holes, effectively breakdown Acid Orange 7 dye into CO_2 , H_2O , or other byproducts.⁸¹

Abbasi *et al.* found that CuCr_2O_4 and $\text{CuCr}_2\text{O}_4/\text{Ag}$ nanostructures effectively degraded MO, MB, and RhB dyes. The researchers found that using leucine-capped $\text{CuCr}_2\text{O}_4/\text{Ag}$ (3%) nanostructures instead of CuCr_2O_4 NPs improved the RhB dye degradation efficiency from 76% to 91%. Their study also high-

lighted the role of pH in the breakdown process. As shown in Fig. 31, the surface of the particles under acidic conditions exhibits a significant positive charge, which inhibits cationic dye adsorption due to electrostatic repulsion. The best degrading efficiency for RhB was reached under neutral circumstances. The catalytic efficiency of leucine-capped $\text{CuCr}_2\text{O}_4/\text{Ag}$ (3%) for RhB degradation was 91% at pH 7 compared to 73% at pH 5.¹⁴⁰

Furthermore, Fig. 32 and Table 6 describe the decomposition of diverse dyes and antibiotics in detail using CuCr_2O_4 NPs.

5.3. Energy storage application of CuCr_2O_4 nanomaterials

5.3.1. Supercapacitor. Supercapacitors are among the best electrical energy storage technologies due to their extended lifespan, sustainability, high-temperature performance, fast charging capability, and long cycle life. The three primary types of supercapacitors are electrochemical double-layer capacitors (EDLCs), pseudocapacitors, and hybrid supercapacitors (HSC).¹⁶³ A hybrid supercapacitor is an energy storage device that combines the features of EDLC and pseudo-

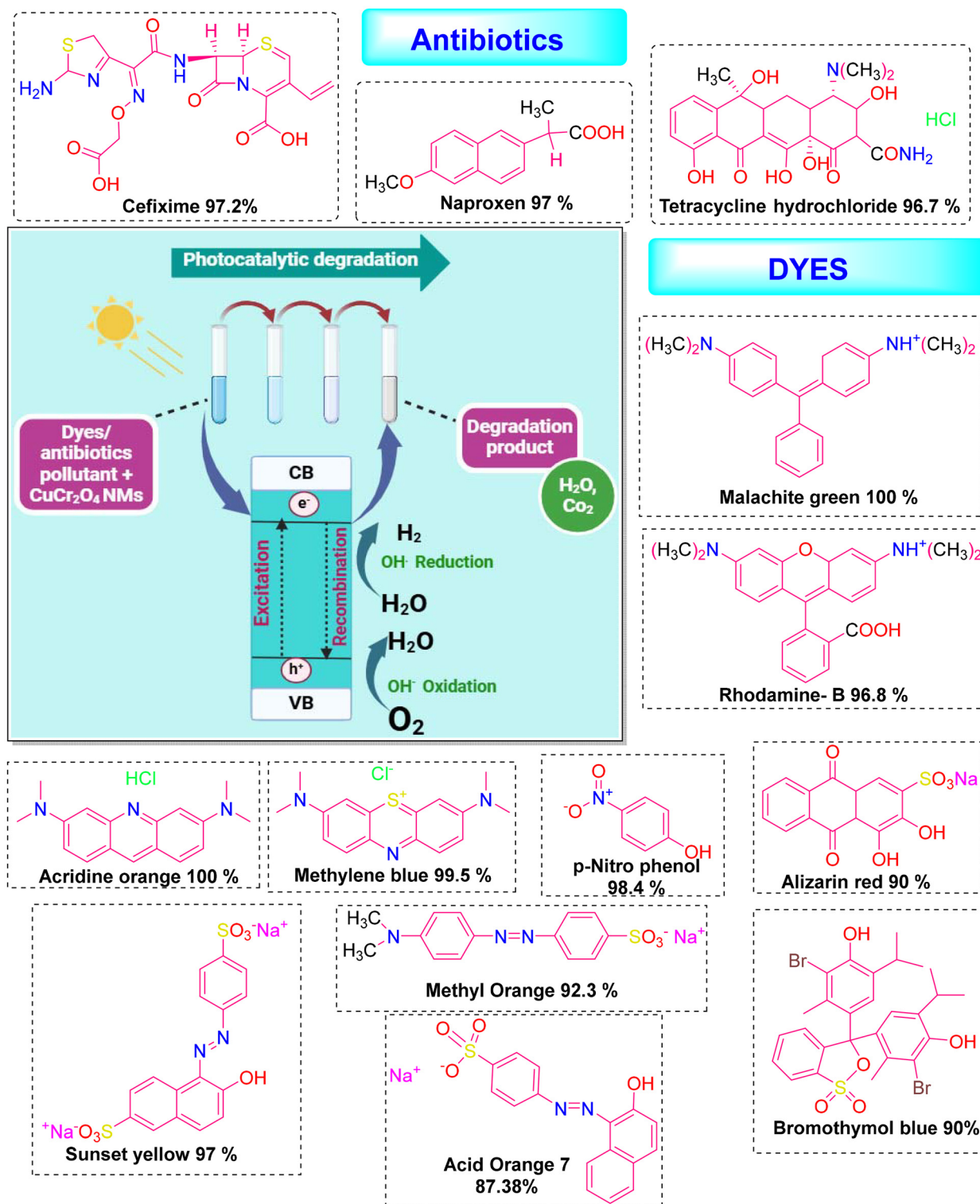


Fig. 32 Illustration of the photocatalytic decomposition of various dyes and antibiotics employing pristine CuCr_2O_4 , metal-doped CuCr_2O_4 , and their nanocomposites.

capacitors. Shayeh *et al.* found that incorporating CuCr_2O_4 NPs into polypyrrole (Ppy) films increases their active surface area and charge transfer sites, leading to improved polymeric electrode stability and capacitance after 1200 cycles (Fig. 33).⁴⁷

Rajeswari *et al.* examined the photovoltaic efficiency of different materials. Among them, CuCr_2O_4 had the lowest photovoltaic efficiency, lowest fill factor, and highest short-circuit photocurrent density. In contrast, La-doped oxides had

Table 6 Overview of the application of CuCr₂O₄ NPs in the decomposition of various dyes/antibiotics

Dye/antibiotic	Dye conc.	CuCr ₂ O ₄ NPs loaded	Band gap	Light source	Degradation (%)	Degradation time (h)	Cycles	Rate constant for degradation (min ⁻¹)	Ref.
<i>p</i> -Nitrophenol	20 mg l ⁻¹	100 mg	—	Visible light	400 °C-98.4 450 °C-95.7 500 °C-86.8 550 °C-85.12 600 °C-77.2	180	10	—	71
MB	10 mg L ⁻¹	10 mg	1.4	Solar light	80.6	18	5	0.132	23
MO	—	—	—	—	92.3	18	—	0.147	—
RhB	—	—	—	—	93.6	12	—	0.2095	—
RhB	—	20 mg (600 °C)	—	Visible light	96.8	60	5	—	22
MB	—	—	—	—	99.5	—	—	—	—
MO	10 ppm	40 mg	1.37	Solar light	14	40	3	—	80
MG (Mn _{0.5} Cu _{0.5} Cr ₂ O ₄)	—	—	—	—	100	—	—	—	—
Tartrazine azo	10 mg L ⁻¹	0.5 g	1.25	Visible light	99.6	120	4	0.04578	64
MB CuCr ₂ O ₄	10 mg L ⁻¹	0.03 g	2.4	Visible light	98	90	5	—	51
MO capped-CuCr ₂ O ₄	0.8 g L ⁻¹	10 ppm	0.9	Visible light	87 capped-CuCr ₂ O ₄	100	5	0.01685	52
Sunset yellow	—	—	—	Visible light	97	4 h	4	0.0128	45
CEF	—	—	—	Visible light	97.2	200	6	—	—
RhB	10 mg L ⁻¹	25 mg	1.83 CuCr ₂ O ₄ 2.31 BiI ₃ O ₆	Led light	95.97	15	4	0.22427	112
TC-HCl	10 ppm	25 mg	1.48 CuCr ₂ O ₄	Visible light	96.7	300	—	—	—
RhB	10 ppm	50 mg	2.62 BiOBr	Visible LED light	99	15	—	—	115
MB	2 mg L ⁻¹	0.2 g	2.81 CuCr ₂ O ₄ 1.40 CeO ₂	Visible light	85	—	—	—	—
MO	15 mg L ⁻¹	50 mg	2.64	Visible light	60	180	3	0.0052	127
MO	15 mg L ⁻¹	—	—	—	84	—	—	0.0025	—
MO	2.50 × 10 ⁻⁵ M	—	—	—	57	—	—	0.01803	26
Acridine orange dye	10 ppm	1 mg mL ⁻¹	1.40 CuCr ₂ O ₄ 1.63 CuO	UV light	97.16	145	—	0.02211	—
CFX	10 ppm	1 g L ⁻¹	1.4 CuCr ₂ O ₄ 2.7 g-C ₃ N ₄	Visible light	98.16	180	5	—	119
Crystal violet	15 mg L ⁻¹	1 mg mL ⁻¹	1.73 0.93	Visible light	98.8	210	—	—	—
MB	10 ppm	50 mg	1.39-CuCr ₂ O ₄ 3.07-SnO ₂	Visible light	100	40	5	—	123
RhB	10 ppm	50 mg	1.4-CuCr ₂ O ₄ 3.1-TiO ₂	Visible light	CuCr ₂ O ₄ -PS-94 CuCr ₂ O ₄ -60 (pH=3.5)	180	4	—	131
MO	10 ppm	10 ppm	—	Sun light	—	90	0.012	—	124
TC-HCl	10 ppm	10 ppm	—	LED light	99.2	15	—	0.30794 0.24358 0.14943	48
Norfloxacin (NORF)	—	—	—	—	89.6	—	—	—	—
MG	30 mg	30 mg	6.48	Visible light	99 (PFL)	—	—	0.9855	118
AR	40 mg	40 mg	1.3	Visible light	58 (DFL)	60	—	—	135
BTB	20 mg	20 mg	2.5, 2.6 and 2.65	LED visible light	97 (PFL)	60	6	—	100
RhB	10 mg l ⁻¹ (pH = 7)	20 mg	—	—	74 (DFL)	90	—	—	—
MO	20 ppm	25 mg	1.4	UV light	86	60	—	—	—
4-NP	100 mg L ⁻¹	0.10 g	CuCr ₂ O ₄ -1.25	Visible light	90	100	—	0.0330	136
Acid orange 7	25 mg L ⁻¹	25 mg	CuCr ₂ O ₄ -citric acid-1.88	Visible light	97.2	40 min	—	0.053	81
RhB	10 ppm	0.04 g	CuCr ₂ O ₄ -tartaric acid-1.92	UV light	87.38	—	—	0.083	—
			3.05	—	96.52	—	—	0.115	—
			—	—	98.81	—	—	—	—
			—	—	Leucine-CuCr ₂ O ₄ /Ag (1%)-81	60 min	—	—	140
			—	—	Leucine-CuCr ₂ O ₄ /Ag (3%)-87	—	—	—	—
			—	—	Leucine-CuCr ₂ O ₄ /Ag (5%)-91%	—	—	—	—

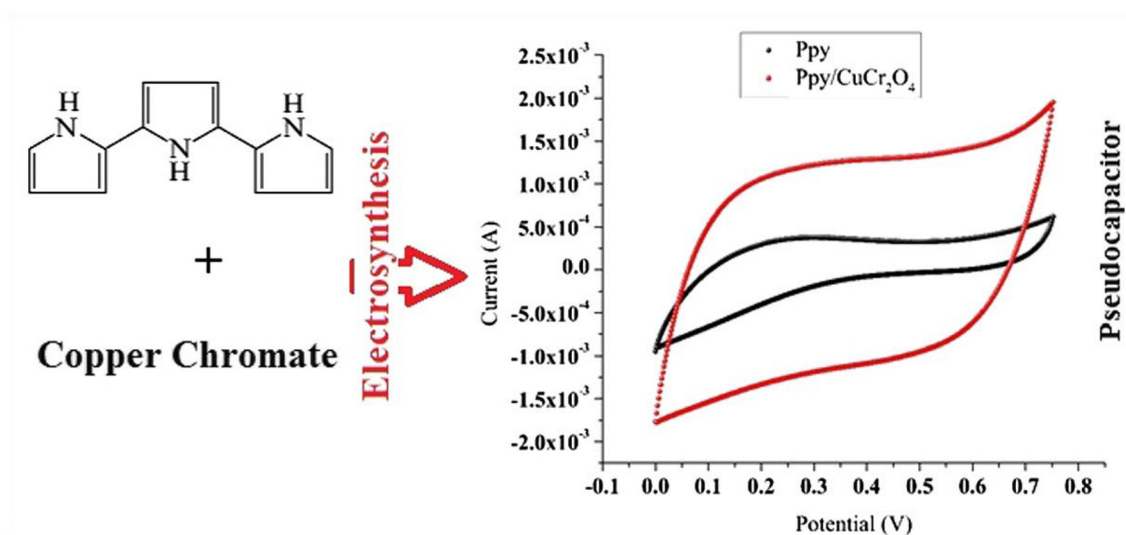


Fig. 33 Ppy/CuCr₂O₄ nanocomposite as a pseudocapacitor.⁴⁷

the highest short-circuit photocurrent density and fill factor. The photoelectric efficiency of the La-doped CuCr₂O₄ electrode was mainly owing to its shape and the synergistic effect of a dispersive active center.⁸³ Bhagwan *et al.* demonstrated the application of CuCr₂O₄ NPs in supercapacitors, achieving a specific capacity of 72.5 mAh g⁻¹ at 1 A g⁻¹. Incorporating MWCNTs significantly enhanced the performance, yielding a higher capacity of 108.5 mAh g⁻¹. The MWCNT/CuCr₂O₄//AC HSC device produced a noteworthy energy density of 32.46 Wh kg⁻¹ and a power density of 775 W kg⁻¹. The tremendous potential of this material for energy storage applications was demonstrated through hands-on demonstrations, which powered gadgets, including LEDs, a kitchen timer, and a toy fan.¹²⁹ The maximum capacitance of 370.5 F g⁻¹ of the CuCr₂O₄/GO nanocomposite was attained with a 0.1 M H₂SO₄ aqueous solution, indicating its potential for capacitive energy storage.^{120,143}

Sarkar *et al.* found that the 5 wt% CuCr₂O₄ NG nanocomposite exhibited a maximum specific capacitance of 530.6 F g⁻¹ at a current density of 0.5 A g⁻¹ and demonstrated excellent cycling stability, retaining 98.3% of its capacity after 5000 GCD cycles. CuCr₂O₄ enhances the supercapacitive performance by serving as a redox-active material and spacer between NG layers, thereby maintaining structural integrity, preventing agglomeration, and minimizing the capacitance loss.¹²¹ According to Susanti *et al.*, CV and EIS measurements showed that the hybrid supercapacitor electrode (N-rGO/CuCr₂O₄) successfully developed a hybrid supercapacitor by combining the characteristics of EDLC and pseudocapacitors.¹³⁰ Kumar *et al.* suggested that the NPs exhibited potential as an asymmetric supercapacitor.⁷³

Vinothkumar *et al.* explored the potential of CuCr₂O₄/Cr₂O₃@NF as an efficient material for energy storage. Fig. 34(a) depicts the galvanostatic charge-discharge (GCD) profiles recorded at different current densities (from 1 A g⁻¹ to

15 A g⁻¹), which display noticeable potential plateaus with non-linear features, indicating the pseudocapacitive behavior of the material. As shown in Fig. 34(b), it is evident that the specific capacitance decreases as the current density increases. Despite this trend, the material exhibits a high specific capacitance of approximately 1027.5 F g⁻¹ at 1 A g⁻¹ and maintains a good performance even at higher rates. The comparison chart in Fig. 34(c) highlights the superior electrochemical performance of CuCr₂O₄/Cr₂O₃ NCs over other spinel chromite-based materials. Finally, Fig. 34(d) presents the effective redox pseudocapacitance mechanism involving Cu and Cr ion pairs, enhanced by the well-dispersed nanoparticles and high surface area, which facilitates rapid ion diffusion and redox activity. Furthermore, the asymmetric device (CuCr₂O₄/Cr₂O₃@NF//AC@NF) delivers an energy density of 38.89 Wh kg⁻¹ at 800.02 W kg⁻¹ and retains 88.67% of its capacitance after 5000 charge-discharge cycles.¹³⁷

5.3.2. Hydrogen storage/water splitting. Lachini *et al.* studied the hydrogen storage properties of CuCr₂O₄ NPs employing cyclic voltammetry and charge-discharge chronopotentiometry in a 3 M KOH solution. CuCr₂O₄ NPs produced by the sol-gel and green techniques have specific capacitance values of 2427 and 1503 F g⁻¹, respectively. Their discharge capabilities were 3011 and 4304 mAh g⁻¹ after 11 cycles, respectively. The sol-gel method demonstrated a superior hydrogen storage performance due to the enhanced porosity.⁴⁹ Ghorai *et al.* studied the photoelectrochemical oxygen evolution reaction (OER) of a 100% CuCr₂O₄/TiO₂ nanocomposite and found a current density of 1.513 mA cm⁻². This was 1.1 and 1.7 times greater than that of the pristine CuCr₂O₄ (1.40 mA cm⁻²) and TiO₂ (0.91 mA cm⁻²), respectively. When exposed to visible LED light, the 100% CuCr₂O₄/TiO₂ NC demonstrated extremely strong OER kinetics. In addition to being a suitable option for the oxygen evolution process, it is also a viable choice when exposed to visible LED light at

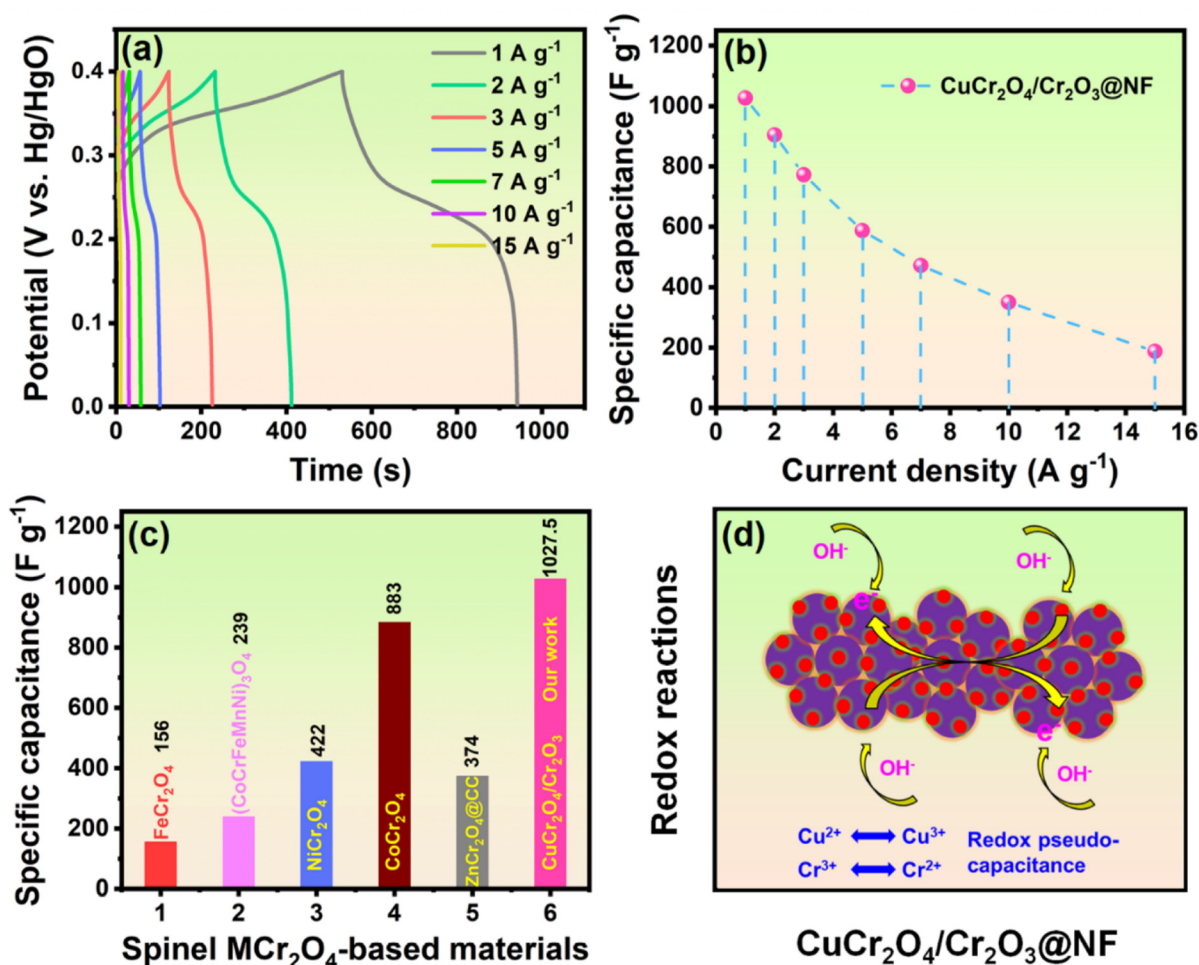


Fig. 34 (a) GCD curves of CuCr₂O₄/Cr₂O₃@NF from 1 to 15 A g⁻¹, (b) C_{sp} vs. current density of CuCr₂O₄/Cr₂O₃@NF, (c) C_{sp} comparison with earlier Cr-related spinel electrodes, and (d) redox reactions of CuCr₂O₄/Cr₂O₃@NF.¹³⁷

home. They also examined [•]OH production in both the DFL and PFL reactions employing the coumarin fluorescence probe approach. This process creates highly fluorescent 7-hydroxycoumarin when the fluorescence-inactive coumarin combines with [•]OH.⁴⁸ Shi *et al.* reported that the CuCr₂O₄/SiO₂ composite shows reduced diffuse reflectance when wet, turning deep-black with strong solar absorbance, and achieving a water evaporation rate of 1.319 kg m⁻² h⁻¹, underscoring the importance of wet-state optical characteristics for improving photothermal materials.¹³³

5.3.3. Electrochemical activity. Interestingly, cyclic voltammetry is a popular electrochemical technique for determining the oxidation and reduction potentials of materials. The oxidation potential, which is tied to electron extraction from the highest occupied molecular orbital (HOMO), coincides with the ionization potential. In contrast, the reduction potential is related to electron affinity and reflects the lowest unoccupied molecular orbital (LUMO) of the material. Cyclic voltammograms were produced at various scan rates (20, 50, 200, and 500 mV s⁻¹) in studies using BiOBr, CuCr₂O₄, and 20% CuCr₂O₄/BiOBr, indicating repeatability in anodic and catho-

dic peak potentials. BiOBr, CuCr₂O₄, and 20% CuCr₂O₄/BiOBr exhibit HOMO values of ~5.27 eV, ~5.21 eV, and ~5.24 eV, while the LUMO levels are ~2.97 eV, ~3.12 eV, and ~2.98 eV, respectively. Under controlled conditions, the oxidation and reduction potentials of these materials showed minor differences (Fig. 35).¹¹²

Shafique *et al.* described the electrochemical stability of a nanoelectrode material, which was tested for commercial uses by scanning modified electrodes for 500 cycles in 1 M KOH and 0.1 M H₂SO₄ electrolytes at a scan rate of 100 mV s⁻¹. EIS spectra show a quicker electron transfer rate in 0.1 M H₂SO₄, with a maximum specific capacitance of 445.5 F g⁻¹ determined using CV.¹²² Furthermore, a three-electrode setup using CuCr₂O₄-CTAB as the electrode material produced capacitance values of up to 965 F g⁻¹ at a scan rate of 1 A g⁻¹. Additionally, it provides excellent cyclic stability in a 1 M Na₂SO₄ electrolyte, retaining 91% of its initial capacitance after 5000 cycles.⁷³

Using a simple drop-coating method, Kong *et al.* successfully fabricated a non-enzymatic electrochemical H₂O₂ sensor. The broad apparent active area of the CuCr₂O₄/GCE probe, low charge transfer resistance, and the synergistic redox coupling

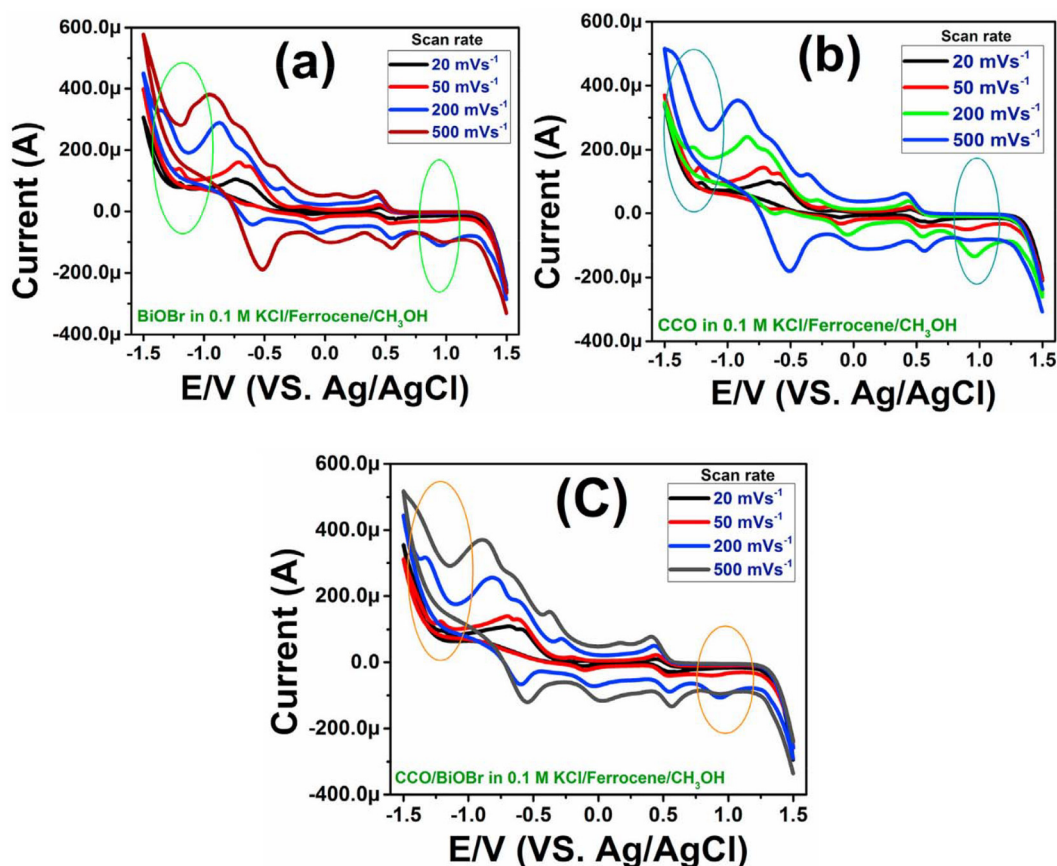


Fig. 35 Highlighted area in the CV of (a) BiOBr, (b) CuCr₂O₄, and (c) 20% CuCr₂O₄/BiOBr in 0.1 M KCl/CH₃OH/ferrocene shows the change in potential in each case, with GC serving as the working electrode, Pt wire serving as the counter electrode, and Ag/AgCl serving as the reference electrode.¹¹²

of CuCr₂O₄ and GCE were responsible for its high sensitivity (886 $\mu\text{A mM}^{-1} \text{cm}^{-2}$), low detection limit (160 nM), and wide linear detection range (0.3 μM to 8.1 mM) in 0.1 M NaOH.⁶⁰ Lao *et al.* reported the electrochemical performance of a CuCr₂O₄Ag composite. Compared to the uncoated CuCr₂O₄, the Ag coating on CuCr₂O₄ improves its coulombic efficiency, cycling stability, and initial discharge capacity, making it a more efficient anode material for lithium-ion batteries.⁴⁶

5.4. Biological activities of CuCr₂O₄ nanomaterials/composite

In the study by Ahmed *et al.*, they examined the toxicity of CuCr₂O₄ NPs in MCF-7 cells using the MTT test. The rosemary essential nanoemulsion helps prevent cancer. This study found that rosemary-mediated CuCr₂O₄ NPs had a stronger cell-killing effect than Nolvadex-D. The IC₅₀ value of the synthesized NPs was 247.7 ppm. Furthermore, a hemolysis assay was performed on red blood cells (RBCs) using different concentrations of the compound. The results revealed no hemolysis at any tested concentration, suggesting that the NPs are non-toxic. This confirms the absence of cytotoxic effects on RBC in the toxicity screening test.⁴¹ Mirzaei *et al.* tested the antibacterial activities of bare or modified NPs against Gram-

positive (*S. aureus*) and Gram-negative (*E. coli*) bacteria in silicone rubber and PVDF matrices. They discovered that the size of the NPs and the matrix medium are critical for inhibiting bacterial growth.⁴² Khan *et al.* studied antibacterial activities achieved through the photoinhibition of both *E. coli* and *S. aureus* bacteria. V₂O₅/CuCr₂O₄/MCM-41 inhibited *E. coli* and *S. aureus* in a zone of 18 ± 0.3 mm and 22 ± 0.2 mm, respectively, when exposed to visible light. The nanostructures efficiently inhibited bacterial growth and survival, demonstrating their suitability for applications in water disinfection and V₂O₅/CuCr₂O₄/MCM-41 antibacterial coatings.¹³⁵ Heydari *et al.* evaluated the bone-forming ability of human adipose-derived stem cells (hADSCs) using a nanocomposite scaffold synthesized from glycerol and azelaic acid (GI-Az) with CuCr₂O₄ NPs. The findings demonstrated that the GI-Az-1%CuCr₂O₄ scaffold considerably raised bone differentiation markers in hADSCs after 14 days and exhibited good mechanical strength, biocompatibility, and degradability.⁴⁴ CuCr₂O₄-BiI₃O₉ NCs were tested for their potential genotoxicity on *Allium cepa* root tips. The mitotic cycle and nuclei index impact the genotoxic nature of NCs and phase disruption. Modifying the stages of the cycle impairs protein synthesis and DNA replication, thereby enhancing the cytotoxicity. Furthermore, the NCs

Table 7 Summary of the biological activities of CuCr₂O₄ nanomaterials/composites

Nanomaterials	Applications	Cell line/ bacteria	Results	Mechanism of action	Observations	Ref.
Rosemary mediated-CuCr ₂ O ₄ NPs	Anti-breast cancer (MTT assay)	MCF-7	IC ₅₀ 247.7 ppm	Generation of reactive oxygen species (ROS), induced oxidative stress, mitochondrial damage	Dose-dependent cell death: up to 64% reduction at 320 ppm after 24 h; apoptosis observed	41
Modified CuCr ₂ O ₄ /silicone rubber (soaked in Tween80)	Antibacterial activity (Agar diffusion method)	<i>E. coli</i> <i>S. aureus</i>	ZOI 11 mm 12 mm	Damage cell membrane	Size and medium are crucial factors in preventing bacterial development because a higher surface area-to-volume ratio promotes better interactions	42
Bare CuCr ₂ O ₄ /silicone rubber (soaked in Tween80)		<i>E. coli</i> <i>S. aureus</i>	12 mm 14 mm			
V ₂ O ₅ /CuCr ₂ O ₄ /MCM-41 (light)	Antibacterial activity (Agar well diffusion method)	<i>E. coli</i> <i>S. aureus</i>	ZOI 18 ± 0.3 mm MIC 50 µg mL ⁻¹ ZOI 22 ± 0.2 mm MIC 40 µg mL ⁻¹	Disruption of the cell membrane, and generation of ROS	Blocked bacterial growth and viability	135

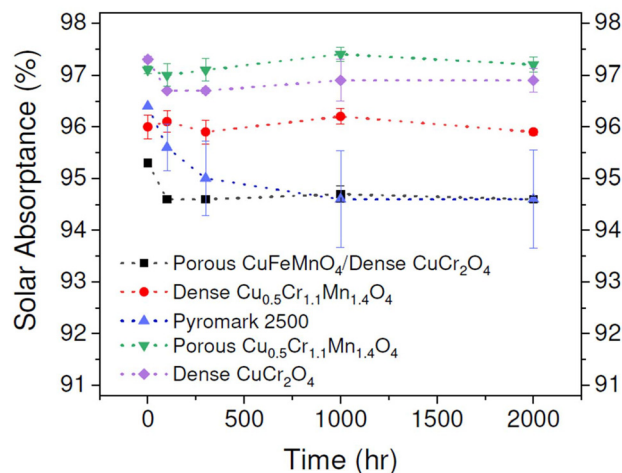
Table 8 Summary of the toxicological activity of CuCr₂O₄ nanomaterials/composites

Applications	Effective dose	Mechanism of action	Observations	Ref.
Homolysis (blood cells)	50, 250, and 500 µg mL ⁻¹	Disruption of cell membranes	Non-toxic nature for all concentration (no homolysis)	41
Genotoxicity (<i>Allium cepa</i> root tips)	—	DNA damage, chromosomal aberrations	Dependent on the NCs concentration	45

showed no chromosomal aberrations or micronucleus production in *Allium cepa*, indicating their non-genotoxic nature.⁴⁵ Moreover, Tables 7 and 8 describe the biological and toxicological activities of CuCr₂O₄ nanomaterials/composites.

5.5. Miscellaneous applications of CuCr₂O₄ nanomaterials/composite

Pakzad *et al.* investigated spectrally selective absorbers in thermoset acrylate-based coatings on aluminium substrates. The larger particle sizes of the produced CuCr₂O₄ NPs exhibit superior solar selectivity. This results in lower thermal emittance (ϵ_T) in the infrared region (2.5–20 µm) and higher solar absorption (α_s) across the solar spectrum (0.3–2.5 µm).¹⁷ The η of DSSC was 44% higher than that of the pure TiO₂ film electrode. CuCr₂O₄/TiO₂ composite electrodes can reduce charge recombination by slowing down reverse electron transfer due to their insulating properties. Furthermore, spinel CuCr₂O₄ is a narrow-bandgap semiconductor with a gap energy of 1.14 eV, which facilitates the absorption of solar energy in the visible light range, thereby improving the performance of DSSCs.⁷² Hu *et al.* described the doping of MCr₂O₄ into TiO₂ film electrodes, which boosted their absorbance. CuCr₂O₄/TiO₂ showed superior light responsiveness

**Fig. 36** Dotted line displaying the anticipated trend for solar absorbance value at 800 °C after 100, 300, 1000, and 2000 h.¹⁴

than NiCr₂O₄/TiO₂, 2% CuCr₂O₄ increased the photoelectric conversion efficiency by 54%, whereas 1% NiCr₂O₄ improved it by 30%.¹²⁵ Rubin and colleagues investigated solar absorber coatings. The maximum solar absorbance was observed in the porous Cu_{0.5}Cr_{1.1}Mn_{1.4}O₄ (Fig. 36), which was 97.1% prior to thermal annealing and remained unchanged during thermal testing, reaching 97.2% after 2000 h. Alternatively, the solar absorbance of Pyromark 2500 significantly decreased, increased from 96.4% to 94.6%.¹⁴

Javed *et al.* explored the temperature and frequency effect of the electrical conduction and dielectric relaxation mechanisms in CuCr₂O₄, tetragonally distorted spinel chromite.⁵⁹ The exchange bias behavior was discussed about the exchange coupling between the ferrimagnetic CuCr₂O₄ (130 K) and the

antiferromagnetic Cr_2O_3 (310 K). The exchange bias in CuCr_2O_4 - Cr_2O_3 was attributed to the exchange coupling between ferrimagnetic CuCr_2O_4 (130 K) and antiferromagnetic Cr_2O_3 (310 K). Strong exchange bias was noticed in the field-cooled hysteresis loop at low temperatures, with both the exchange bias field and coercive field decreasing as the temperature increased, vanishing at around 200 K.¹²⁶

6. Challenges and opportunities

Despite significant advancements, several challenges remain in the synthesis and application of CuCr_2O_4 NPs. Achieving precise control over particle size, shape, and crystallinity is challenging, given that the synthesis parameters such as temperature, pH, reaction duration, and surfactants significantly influence the final properties. Scalability and cost-effectiveness also pose hurdles, given that many synthesis methods require high energy inputs or expensive precursors, limiting their commercial viability. Furthermore, to ensure a consistent performance, additional research is also required to determine the stability and reproducibility of CuCr_2O_4 NPs under various environmental conditions. Concerns regarding potential cytotoxicity and environmental impact must be addressed before the widespread use of biomedical and industrial applications. According to the literature, CuCr_2O_4 nanocomposites and metal-doped CuCr_2O_4 NPs have demonstrated a remarkable capacity for energy storage and photocatalytic activity. Additional research is required, especially in the area of green synthesis. Only two studies have reported the green production of CuCr_2O_4 NPs from natural sources to date. This opens up a new avenue for research on sustainability. Additionally, creative approaches are necessary to enhance the compatibility and efficiency when integrating these NPs into practical devices, such as sensors, batteries, and catalysts.

However, numerous opportunities exist to overcome these challenges and expand the potential of CuCr_2O_4 -based materials. Advanced functionalization through metal doping and nanocomposite formation can significantly enhance their catalytic, electrochemical, and biomedical properties. The development of sustainable and green synthesis techniques that use less energy and environmentally benign precursors can improve the scalability, while lessening their negative effects on the environment. CuCr_2O_4 NPs have enormous potential for anticancer treatments, antimicrobial coatings, and targeted drug delivery in biomedical applications. Their excellent electrochemical properties also make them ideal candidates for next-generation energy storage devices, including supercapacitors and Li-ion batteries. Furthermore, their integration into smart sensor technologies might result in extremely sensitive and specific detection systems for industrial applications, healthcare diagnostics, and environmental monitoring. By addressing existing challenges and leveraging these opportunities, CuCr_2O_4 NPs can be further optimized for cutting-edge applications, driving innovation across multiple scientific and technological domains.

7. Conclusion

In this review paper, we provide an overview of breakthroughs in CuCr_2O_4 nanostructures, including pristine NPs, metal-doped variants, and nanocomposites, with diverse applications spanning biological, catalytic, electrochemical, and sensing fields. Several procedures exist for the synthesis of CuCr_2O_4 nanomaterials, including sol-gel, hydrothermal, solution combustion, co-precipitation, and electrospinning approaches. The fabrication of these NPs is significantly influenced by variables such as temperature, pH, reaction time, precursor concentration, and the selection fuel or surfactant, which directly impact their structural and functional properties. In this review, we demonstrated that CuCr_2O_4 NPs demonstrate significant catalytic activity in organic transformations. They also serve as an excellent enhancer of the burning rate for AP-based solid propellant systems. Furthermore, they have demonstrated outstanding potential in energy storage devices such as Li-ion batteries and supercapacitors, as well as electrocatalysis for the OER and HER. CuCr_2O_4 NPs show promise in nanomedicine, including anticancer, antibacterial, and genotoxic properties. Their photocatalytic activity has also been widely investigated for the decomposition of dyes and antibiotics. CuCr_2O_4 NPs have great promise in these fields due to their unique physical and magnetic features. Future studies should focus on developing sustainable and scalable synthesis techniques, while exploring novel applications of CuCr_2O_4 -based nanomaterials. By bridging the gap between synthesis and application, this review aims to contribute to the advancement of copper chromite nanotechnology for next-generation scientific and industrial innovations.

Conflicts of interest

The authors declare no conflict of interest.

Data availability

No primary research results, software or code have been included and no new data were generated or analysed as part of this review.

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