

"Chitosan-Loaded Nanocomposite Materials for Biocidal and Photocatalytic Activity– A Review"

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Abstract: Chitosan (CS)-based antibacterial hydrogels, often enhanced with inorganic nanoparticles, antibacterial agents, or CS derivatives, are gaining attention for their potential in wound healing, drug delivery, and environmental cleanup. These materials offer excellent biocompatibility, improved antibacterial efficacy, and controlled release capabilities. Research has shown that CS composites with metal oxides such as SnO₂, CuO, Ag, NiO, MgO, and Fe₃O₄ exhibit enhanced antibacterial, antifungal, photocatalytic, and adsorption properties. However, challenges like solubility issues and potential toxicity need to be addressed. Ongoing research is focused on optimizing these composites to improve both performance and safety for biomedical and environmental applications.

Keywords- nanoparticles, Chitosan, antibacterial, metal oxide, photocatalytic, biocompatibility

I. Introduction

Nanoparticles, with sizes ranging from 1 to 100 nm, exhibit unique physical and chemical properties compared to their bulk counterparts due to their large surface area relative to their volume.¹ They can be synthesized using various methods, categorized into top-down approaches (such as hydrothermal synthesis, ball milling, etching, laser ablation, sputtering, and electro-explosion) and bottom-up approaches (including spinning, sol-gel methods, molecular condensation, chemical reduction, and green synthesis). Nanoparticles are also classified into organic and inorganic types. Organic nanoparticles, such as carbon nanorods, lipids, and polymers, offer intriguing applications due to their enhanced stability.² While bulk organic compounds are generally soluble in water, nanoparticles are soluble in aqueous solvents.³ Inorganic nanoparticles, particularly metal oxides, are of great interest due to their ability to endure harsh conditions and their notable physical and optical properties.⁴

Chitosan is a polymer derived from chitin, a biopolymer found in eukaryotic species such as crustaceans, insects, and fungi. It is primarily composed of N-acetyl-D-glucosamine units, but through deacetylation, these units become β-1,4-D-glucosamine, which is known as chitosan (Figure 1). Chitosan demonstrates significant effectiveness at the nanoscale. To form chitosan nanoparticles, the concentration should be at or below 4 mg/ml, depending on the selected TPP concentration.¹ Chitosan and chitin are commonly sourced from marine environments.

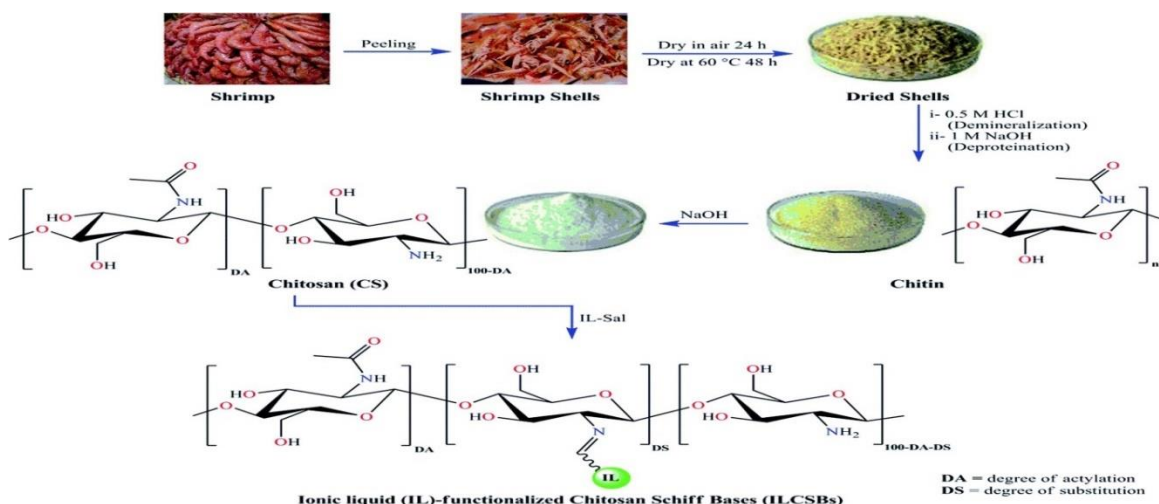


Figure 1. Schematic representation of the extraction of chitosan from shrimp shells and synthetic route to ionic liquid-based chitosan Schiff (5)

Chitosan nanoparticles retain the properties of chitosan while exhibiting enhanced characteristics due to their small size and surface effects.⁶ Unlike chitin, chitosan is soluble in acidic media, which facilitates its processing and application.⁷ While chitosan dissolves in diluted acids, it is typically insoluble in most organic solvents and at neutral pH in water. This insolubility in water poses a challenge for its broader use. Researchers have focused on functionalizing chitosan to improve its water solubility. Each glucosamine unit in chitosan has an amine group and two hydroxyl groups that serve as reactive sites, making chitosan effective for chelation, electrostatic interactions, and film formation. Chitosan has been studied for its applications in color removal, toxic

organic compound removal, biosensors, and antimicrobial agents due to its interaction with various substances. Nanoparticles can be synthesized through chemical methods, such as electrochemical or chemical reduction, which are cost-effective and scalable but may involve toxic reagents and produce hazardous waste. Alternatively, biological methods use organisms or natural extracts, offering a low-cost and potentially less toxic approach but are more complex. The choice of synthesis method significantly impacts the nanoparticles' size, stability, and biological activity, influencing their chemical properties and potential toxicity.⁸⁻⁹

Chitosan, derived from crustacean by-products like lobster cephalothorax, is a valuable material for industrial production due to its high chitin content (at least 20%). For optimal chitosan production, methods that yield chitosan with stronger bioactivities, such as lower degree of acetylation (DA) and lower molecular weight (MW), should be prioritized

Chitosan, a natural green polymer, is limited in applications due to its insolubility in water, poor acid resistance, and inadequate mechanical properties. Chemical modification is an effective way to improve its solubility and enhance its physicochemical properties, such as thermal stability, rheological properties, oxidation resistance, and antibacterial activity. Chitosan's structure has three types of active groups: amino, primary hydroxyl (C6-OH), and secondary hydroxyl (C3-OH). The amino group is the most reactive, followed by the primary and then the secondary hydroxyl groups. Modifications can target the amino, hydroxyl, or both, resulting in N-, O-, or N,O-modified derivatives, expanding its applications.¹⁰⁻¹¹

Antibacterial and Photocatalytic Activity of chitosan-Based Composite

Semiconductors, such as TiO₂, ZnO, MgO, WO₃, Fe₂O₃, and CdS, are often chosen as photocatalysts due to their narrow band gaps, which allow them to absorb photons and generate electron-hole pairs that act as reducing and oxidizing agents, respectively. However, their photocatalytic efficiency can be limited by their response to visible light. To address this, doping with transition metals or non-metals is used to enhance their visible light absorption. While transition metal doping can introduce localized d-states that serve as trapping sites for charge carriers, reducing their lifetime and efficiency, non-metal doping (e.g., nitrogen, boron, sulfur) is a more promising approach. For instance, nitrogen doping in TiO₂ introduces p-states above the valence band, narrowing the band gap and improving visible light absorption. Nitrogen-doped TiO₂, first reported by Sato et al., uses sources like urea, ammonia, and nitric acid. Additionally, transition metal ions and complexes have been immobilized on biopolymers to synergize homogeneous and heterogeneous catalysis in a cost-effective manner.¹²

The study explores biopolymer-based hydrogels, like those made from cellulose, chitosan, and alginate, enhanced with metal ions (e.g., Cu²⁺, Fe³⁺) for catalytic applications. These hydrogels provide an eco-friendly platform for sustainable catalysis, showing high efficiency in degrading organic dyes and reducing pollutants. The hydrogels' properties, such as porosity and mechanical strength, can be tailored to optimize metal ion distribution and catalytic performance, presenting a promising approach for environmental remediation and green chemistry.¹³

This study reviews CS-based antibacterial hydrogels, categorized into those with inorganic nanoparticles, antibacterial agents, or CS derivatives. These hydrogels offer benefits like biocompatibility and controlled release but face challenges like solubility and potential toxicity. Chemical modifications of CS can enhance its properties, and combining it with inorganic substances reduces side effects. While promising for treating infected wounds, more research is needed to fully understand CS's antibacterial mechanisms.¹⁴

CS-SnO₂

SnO₂ nanoparticles and chitosan-SnO₂ (CS-SnO₂) nanocomposites were synthesized by vinod kumar et.al. using a cost-effective precipitation method and sonication. XRD analysis confirmed that the SnO₂ nanoparticles maintained a tetragonal structure with well-defined peaks, indicating high crystallinity. Incorporation of chitosan led to broader, less intense peaks in the XRD patterns, suggesting reduced crystallite sizes. TEM images showed that the CS-SnO₂ (50:50) nanocomposite had better dispersion of spherical nanoparticles compared to CS-SnO₂ (75:25). Band gap measurements from DRS spectra revealed values of 3.38 eV for pure SnO₂, 3.52 eV for CS-SnO₂ (50:50), and 3.91 eV for CS-SnO₂ (75:25) (Table 1), correlating with nanoparticle size. Photocatalytic tests under 348 nm UV light demonstrated that CS-SnO₂ (50:50) achieved significantly higher degradation rates for methyl orange and rhodamine B compared to both CS-SnO₂ (75:25) and pure SnO₂, with improvements of up to 2.74 times for methyl orange and 1.99 times for rhodamine B. Furthermore, the CS-SnO₂ (50:50) nanocomposite showed excellent stability and reusability, maintaining its photocatalytic efficiency over multiple cycles. This underscores the enhanced performance of CS-SnO₂ (50:50) due to its optimal nanoparticle size, increased surface area, and the synergistic interaction with chitosan.¹⁵

Table 1 Crystallite size, TEM Particle size and band gap of the synthesized binary CS-SnO₂ photocatalysts. (15)

Samples	Crystallite size D (nm)	TEM Particle size (nm)	Reflectance edge (nm)	Band gap based on KM function (eV)
Pure SnO ₂	13.2	15	364	3.38
CS-SnO ₂ (50:50)	8.4	11	348	3.52
CS-SnO ₂ (75:25)	6.7	Agglomeration	318	3.91

CS-Ag

The study focused on synthesizing silver nanoparticles (AgNPs) and incorporating them into PVA/CH hydrogel films, with an emphasis on evaluating their properties and performance. AgNPs synthesized at 75 °C had a particle size of 4.59 nm with a PDI of 0.84, and increasing EC extract concentration from 2 to 10 mg/ml led to larger particles. Zeta potential decreased from -17.6 to -12.3 mV, indicating changes in stability, but six-month stability studies showed no significant changes in size, absorbance, or zeta potential. FTIR spectra revealed PVA bands at 2946.67 cm^{-1} (C–H stretching) and 3631.72 cm^{-1} (–OH group), while CH showed peaks at 3287.49 cm^{-1} (–OH stretching) and 1661.58 cm^{-1} (Amide I). Shifts in characteristic peaks for PVA/CH films indicated interactions with AgNPs. SEM images showed that neat PVA films had a porous structure, which became more uniform with increased PVA content. TEM analysis revealed that AgNPs synthesized at room temperature and 45 °C were oval and larger (20–40 nm, 20–30 nm), while those at 55 and 65 °C were spherical and smaller (10–20 nm, <10 nm). XRD patterns confirmed a face-centered cubic structure for AgNPs with peaks at 38.08°, 44.29°, 64.50°, and 77.52°. TGA results indicated that AgNPs in hydrogel delayed thermal degradation, with a slight increase in degradation temperature. The swelling ratio increased with decreasing PVA content, and in-vitro weight loss decreased with higher PVA content. Viscosity increased with higher PVA concentration. Content uniformity and silver ion loading were consistent, with AgNPs solution releasing 90.92% silver ions in 24 h and PVA/CH hydrogels releasing 82.21% to 91.83% over 48–72 h. Hemocompatibility tests showed no significant hemolysis, and antimicrobial assays demonstrated effective inhibition of *S. aureus* and *E. coli*, with AgNPs showing larger inhibition zones compared to controls.¹⁶

Cs-CuO

Firnanely *et. al.*, synthesized a chitosan-CuO composite. The chitosan-CuO composites successfully absorbed heavy metals, achieving an 87% absorption rate for mercury (Hg). Important functional groups are visible in the IR spectra of the sensor material at every stage of fabrication. Bands at 1082, 1382, and 1655 cm^{-1} , which represent C–O, C–N, and C=O bonds, respectively, are visible in the pure chitosan (CS) film. The presence of the ionic liquid is indicated by a prominent O–H band at 3277 cm^{-1} and a C–H doublet at 2928 and 2872 cm^{-1} in the chitosan film with ionic liquid (CSeIL). A unique band at 435 cm^{-1} is seen in the final sensor material (CS-IL-CuO NPs), which is attributed to the Cu–O bond and verifies the integration of CuO nanoparticles.^{17–18}

The antimicrobial activities of the synthesized CuO/CS nanocomposite spheres were tested against *Pseudomonas aeruginosa* using a disc diffusion method on Nutrient Agar plates. The results showed that pure chitosan (CS) exhibited minimal antibacterial activity, with bacterial growth observed without inhibition zones. In contrast, the composite spheres demonstrated strong antibacterial properties, with clear inhibition zones around the discs. X-ray diffraction (XRD) analysis confirmed the presence of CuO within the CS matrix, with characteristic peaks of CuO observed at $2\theta = 16.3, 22.6, \text{ and } 34.3^\circ$, consistent with pure CuO. FTIR analysis indicated the presence of both CS and CuO in the composite. Field emission scanning electron microscopy (FESEM) revealed rough surfaces with aggregated CuO particles embedded in the CS. Thermogravimetric analysis (TGA) showed similar thermal degradation patterns to pure CS, suggesting no significant impact of CuO on thermal stability. The adsorption study demonstrated that the composite spheres were more effective than pure CS in removing methylene blue (MO), with 94% adsorption by the composite spheres compared to 76% by pure CS after 180 minutes (Figure 2). The adsorption was pH-dependent, with optimal removal observed at pH 6 to 8.¹⁹

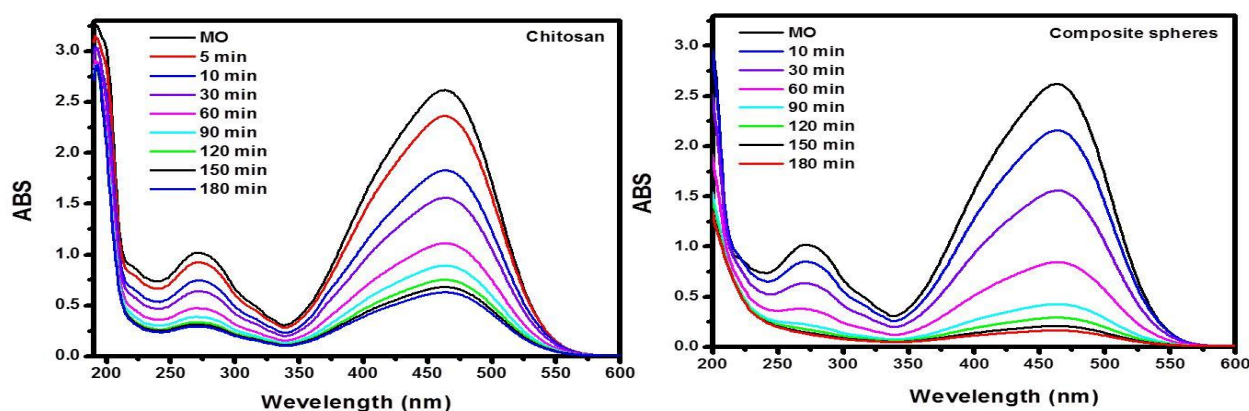


Figure 2 Change in absorption spectrum of MO using (a) CS and (b) composite spheres (19)

The Cs/PVA/GO/CuO patch shows strong antibacterial activity, promotes cell proliferation, and supports wound healing. It maintains good cell viability at lower concentrations and offers effective antimicrobial action at higher levels. The patch also regulates moisture in wounds, absorbs excess exudate, and shows minimal hemotoxicity, making it a promising option for wound care.²⁰

Khan *et al.* synthesized chitosan-copper oxide (CH-CuO) nanoparticles. Due to their unique three-dimensional structure, electrospun nanofibrous membranes are beneficial for wound care, drug delivery, and tissue engineering. This study aims to develop

these membranes by combining PVA, chitosan, and varying amounts of copper oxide. The researchers successfully synthesized chitosan-stabilized copper oxide nanoparticles and confirmed their structure. After UV irradiation, the membranes were analyzed using several techniques, including XRD, SEM, DSC, FTIR, UV-Vis, and DLS. The results indicated that the nanofibers had diameters ranging from 147 to 207 nm, that CH-CuO nanoparticles were effectively incorporated, that UV treatment improved thermal stability, and that antibacterial activity increased. In vitro tests confirmed the nanoparticles' safety and demonstrated controlled release of copper ions.²¹

The XRD analysis shows chitosan's crystalline peaks at $2\theta = 10^\circ$ and 20° , while the CS/PVA matrix displays amorphous characteristics, and CuO exhibits distinct monoclinic peaks. FTIR spectra reveal shifts in functional group peaks for CS/PVA/CuO after Pb (II) adsorption (Figure 3), indicating interactions between metal ions and functional groups. SEM images show smooth chitosan surfaces and rougher CS/PVA/CuO beads post-adsorption, confirming successful Pb(II) binding. EDS analysis confirms the presence of Cu in CuO nanorods and Pb in the adsorbent post-adsorption. TEM images depict well-dispersed CuO nanorods within the CS/PVA matrix, with a diameter of 20–50 nm.²²

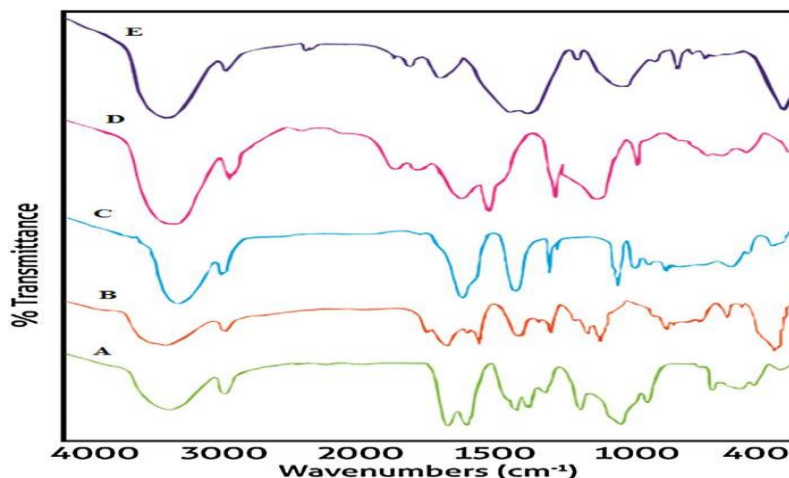


Figure 3 FTIR spectra of (A) pure chitosan, (B) CS/PVA (C) CuO (D) CS/PVA/CuO (E) Pb(II) loaded CS/PVA/CuO (22).

The electrospun nanocomposites were characterized using various techniques, including AFM, FT-IR, TEM, EDS, elemental analysis, SEM, DTA, and TGA. The Pt/PVA-CuOeCo₃O₄/CH, Pt/PVA-CuOeCo₃O₄, and Pt/PVA/CH catalysts were examined by FT-IR, TEM, and EDX. The morphology of the PVA-CuOeCo₃O₄ nanocomposite was confirmed by SEM and TEM, showing spherical metal oxides with a mean size of 37.54 nm and PVA nanofibers averaging 80 nm. TEM images revealed well-dispersed spherical Pt nanoparticles around the metal oxides. FT-IR spectra confirmed the presence of PVA and metal oxides, with characteristic peaks for OeH stretching and CeO stretching. TGA analysis indicated that PVA decomposes in two stages, while the presence of CuO and Co₃O₄ decreased the final degradation temperature of the PVA-CuOeCo₃O₄ composite. AFM images showed a smooth and interconnected nanofiber structure with metal oxides, and EDX confirmed the formation of the composites. Electrocatalytic studies were conducted to assess CO tolerance, hydrogen adsorption, and methanol oxidation. CO stripping tests showed that Pt/PVA-CuOeCo₃O₄/CH had superior CO tolerance and higher electrocatalytic activity for methanol oxidation compared to Pt/PVA-CuOeCo₃O₄ and Pt/PVA/CH. The ECSAC values indicated that Pt/PVA-CuOeCo₃O₄/CH had the highest electrochemically active surface area. The CV curves for hydrogen adsorption revealed that Pt/PVA-CuOeCo₃O₄/CH had the highest ECSA, indicating more active sites. Methanol oxidation reactions demonstrated that Pt/PVA-CuOeCo₃O₄/CH had better performance with higher peak current densities and lower onset potentials. The stability of Pt/PVA-CuOeCo₃O₄/CH was superior in chronoamperometry tests and the effects of methanol concentration and scan rates were examined, showing increased catalytic activity with higher concentrations and scan rates. Electrochemical impedance spectroscopy confirmed that Pt/PVA-CuOeCo₃O₄/CH exhibited enhanced electrochemical activity and improved ion diffusion.²³

CS- Fe₃O₄

XRD and FTIR methods were used to analyse the structure and content of the chitosan-coated Fe₃O₄ nanoparticles that P Nehra et al. synthesized. The XRD examination confirms the spine structure and primarily magnetite composition of the Fe₃O₄ nanoparticles by revealing diffraction peaks. Consistent diffraction angles show that the chitosan coating does not change the crystal structure. Utilising the Debye-Scherrer equation, the mean particle size was determined to be roughly 10.4 ± 4.9 nm for Fe₃O₄ NPs and 11.4 ± 5.2 nm for Fe₃O₄ NPs coated with chitosan. The presence of chitosan on the surface is verified by FTIR analysis, which shows distinctive absorption bands that match FeO bonds and primary amine groups. Peaks connected to the stretching vibrations of OH, NH, and CO provide additional evidence for the effective chitosan coating of Fe₃O₄ nanoparticles. Surface modification of Fe₃O₄ nanoparticles with chitosan significantly enhances their antibacterial and antifungal properties. These chitosan-coated Fe₃O₄ nanoparticles, which can be synthesised using the coprecipitation method, have a size of less than ~15 nm, making them effective in combating various microbial threats.²⁴

X-ray diffraction (XRD) using X'Pert Pro with Cu K α radiation scanned from 10° to 80° revealed that pure chitosan exhibits a 20° peak, attributed to its crystalline structure. This peak disappears in HC composites, suggesting successful integration of α -Fe₂O₃ nanoparticles into the chitosan matrix. The XRD patterns of HC nanocomposites show α -Fe₂O₃ with peaks at 24.1°, 33.1°, 35.6°, 40.7°, 49.4°, 54.0°, 57.4°, 62.3°, 63.9°, 71.8°, and 75.3°, corresponding to indices (012), (104), (110), (113), (024), (116), (018), (214), (300), (1010), and (220), and reveal maghemite and magnetite phases. Semi-quantitative analysis gives 85.6% hematite, 12.4% maghemite, and 2.0% magnetite. TEM images show that nanoparticles have hexagonal shapes (7-11 nm) with some aggregation, while IR spectra confirm chitosan coating through Fe–O bonds (560 and 475 cm⁻¹) and chitosan-related bands. Raman spectra reveal hematite, maghemite, and magnetite phases with peaks at 213, 288, 402, 495, 611, 702, 525, and 660 cm⁻¹. Magnetization curves indicate a decrease in saturation magnetization (Ms) with increasing chitosan content, attributed to non-magnetic carbon residue, but the values remain higher than typical α -Fe₂O₃ nanoparticles due to small particle size (7-11 nm) and aggregation effects. HC0.5 shows superparamagnetic behavior with low coercivity (Hc) and remanent magnetization (Mr), which increase slightly with higher chitosan content, making the samples suitable for biomedical applications.²⁵

Cs-Graphene oxide

The electrical conductivity of the reduced graphene oxide/chitosan (rGO/CS) composite was measured using a potentiostat at room temperature (23°C, ~20% relative humidity). Conductivity tests were conducted with 100 μ L of rGO/CS composite containing different chitosan amounts (0, 50, 100, and 150 mg) in 96-well plates, with a probe distance of 5 mm. Conductivity was also evaluated based on composite shape using cellulose paper molds. X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) were used to analyze the rGO/CS composite's surface characteristics, with SEM performed at 5 kV and XPS using an Al X-ray source (1486.6 eV) at 15 kV $\times$ 24.1 W. Raman spectroscopy was conducted with a 532 nm laser and a $\times$ 20 objective lens. For sensor fabrication, various substrates (O.H.P. film, A4 coated paper, and cellulose paper) were used, and molds (0.5 mm $\times$ 0.5 mm $\times$ 5 mm) with shapes like heart, star, and circle were tested. The paper-based rGO/CS composite sensor was exposed to NO₂ gas (1–100 ppm) using a mass flow controller and conductance changes were analyzed with a current-voltage meter.²⁶

CS- MgO /CS- Nio Polymer

The development of novel antibacterial agents is crucial in combating biofilm-associated infections and drug-resistant bacteria. Metal oxide-based polymeric materials, such as chitosan-coated MgO and NiO nanocomposites, show promise in distinguishing mammalian cells from bacterial cells, providing long-term antibacterial effects. Synthesised via a simple in situ chemistry approach, these nanocomposites demonstrated effective antibacterial activity, particularly against *Staphylococcus aureus*. Notably, at a concentration of 5 μ g/mL, CS-NiO significantly reduced the viability of *E. coli* and *S. aureus*, with up to 98% inhibition of *S. aureus* at 15 μ g/mL after 12 hours of incubation.²⁷

CS-MO nanocomposites outperformed chitosan and metal oxides alone in terms of antibacterial effectiveness. Notably, CS-NiO was the most effective, reducing *S. aureus* and *E. coli* viability by 2-8% after 12 hours. Thus, chitosan/NiO nanocomposites may serve as effective antibacterial agents against harmful pathogens.²⁷

The research on heterogeneous photocatalysis focusses on using chitosan (CS) as a support material for metal oxide photocatalysts. CS improves dispersion, surface area, and pollutant removal efficiency, achieving significant results like 99% Pb(II) removal in 30 minutes. While CS is cost-effective, further research could help reduce costs even more.²⁸

Heterogeneous photocatalysis is a key area of research for degrading pollutants and splitting water. Metal oxide photocatalysts, when supported by materials like chitosan (CS) biopolymer, show improved performance due to enhanced dispersion, surface area, adsorption sites, and stability. CS provides strong adhesion, high surface area, and adsorption affinity for pollutants. It helps overcome limitations of nano-sized photocatalysts by improving stability, recyclability, and preventing coagulation. For instance, CS-supported metal oxides can remove 99% of Pb(II) from wastewater in 30 minutes. Further improvements are possible by increasing CS porosity and finding cost-effective extraction methods.²⁸

The FT-IR spectra of PVP and PVP/NiO nanocomposite show a broad peak around 3450 cm⁻¹, attributed to O-H bonds and water molecules, with a peak at 1675 cm⁻¹ corresponding to C=O absorption. Peaks at 2950 cm⁻¹ are due to CH₂ stretching, while 1440 cm⁻¹ indicates CH₂ bending, and a sharp peak at 441 cm⁻¹ in PVP/NiO corresponds to Ni-O stretching. XRD patterns reveal broad peaks for PVP at 11° and 20°, with lower intensities in PVP/NiO, signifying reduced crystallinity, and sharp peaks at 2 θ of 37.3°, 43.4°, and others confirming cubic NiO formation. TGA-DSC analysis shows initial mass losses of 7% for PVP and 3% for PVP/NiO due to water removal, with significant decomposition starting at 453°C for PVP and 435°C for PVP/NiO. At 800°C, PVP completely decomposes, while PVP/NiO retains about 45% mass. SEM images show that NiO nanoparticles (40-70 nm) in PVP/NiO create pores, enhancing interaction and adsorption sites. EDS spectra reveal decreased C and H contents and increased O and Ni contents in PVP/NiO, confirming successful NiO deposition. UV-Vis spectrophotometry shows a decrease in the absorbance of eosin Y (EY) over time, indicating efficient dye removal by PVP/NiO. Adsorption efficiency decreases with increasing pH, with maximum removal (99.98%) at pH 3 using 0.025 g of PVP/NiO.²⁹ the variance analysis of experimental results. A low probability (< 0.05) of the F-value (302.03) implies the model's accuracy. The acceptable lack of fit value with an F-value of 0.7022 (> 0.05) indicates the method is suitable for presenting experimental data. The model showed a high R² value of 0.9959 for MB removal, indicating strong agreement between experimental and predicted results. The predicted R² (0.9852) is also in good agreement with

the adjusted R^2 (0.9926). The model's signal-to-noise ratio was 63.374, indicating a strong signal and suitability for navigating the design space. Response surface plots showed the interactive effects of pH, initial MB concentration, adsorbent dosage, and temperature on MB adsorption. Higher adsorption efficiency was achieved with pH 7.0 and optimal conditions were found to be pH 7.28, dosage 0.47 g/L, initial MB concentration 19.37 mg/L, and temperature 309.76 K, resulting in 94.51% removal (desirability = 0.829). Adsorption kinetics indicated that pseudo-second-order kinetics best fit the data, and the process involved multiple mechanisms, including chemisorption³⁰

This study meticulously explores the synthesis and morphological characterization of magnesium oxide nanoparticles (MgONP) and their chitosan-functionalized counterparts (CS-MgONP), employing advanced analytical techniques such as X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy. XRD analysis, utilizing a Regaku diffractometer with Cu-K α radiation ($\lambda=1.5418$ Å) over a 2θ range of 10–80 degrees at a scan rate of $0.05^\circ \text{ s}^{-1}$, unveiled distinct diffraction peaks emblematic of both MgO and chitosan, thereby corroborating the successful fabrication of the composite material. Notably, diffraction peaks observed at 200, 380, 620, and 740 correspond to the crystalline phases of chitosan and MgO, while minor peaks at 500 and 580 suggest incipient hydration of MgO to $\text{Mg}(\text{OH})_2$. Concurrently, FTIR spectroscopy delineated the presence of critical functional groups, with characteristic bands below 1000 cm^{-1} indicating Mg-O bonds, a band at 1055 cm^{-1} attributed to C-O-C stretching vibrations, and an expanded band at 1638 cm^{-1} signifying the presence of amide groups associated with chitosan. Additional significant absorptions include those at 1383 cm^{-1} for COO^- vibrations, 2350 cm^{-1} for CO_2 absorbed by MgO, and at 3000 cm^{-1} for C-H stretching vibrations. The narrow bands at 3420 cm^{-1} and 3699 cm^{-1} correspond to -OH and -NH $_2$ groups, respectively, both strongly perturbed by hydrogen bonding. Pertinent spectral changes observed at 3699 cm^{-1} and 1663 cm^{-1} in the FTIR spectrum of MB-treated CS-MgONP suggest the pivotal role of -OH and N-H (amide) groups in facilitating MB adsorption. Furthermore, the study elucidated the kinetics of MB adsorption, revealing a rapid initial uptake of 60.25% within the first 5 minutes, with equilibrium achieved at 70 minutes, culminating in 85.6% adsorption. This rapid adsorption phenomenon is attributed to the abundant active sites on the CS-MgONP surface, which become progressively saturated, resulting in a plateau in adsorption capacity. The findings of this research underscore the substantial potential of CS-MgONP as an efficacious adsorbent for the remediation of dye pollutants, particularly methylene blue, from aqueous environments.³⁰

Heba Ali et al. investigated the antibacterial activity of the synthesised NiO/chitosan/PVA nanocomposite films using the disc diffusion technique. The NiO/chitosan/PVA nanocomposite films exhibit greater efficacy against Gramme-negative bacteria, according to the findings of an antimicrobial test. Because the adsorption characteristics of the NiO were inhibited in the membrane, the removal of MO dye tests revealed that the NiO/chitosan/PVA nanocomposite membrane demonstrated more than two times greater decolorisation effectiveness than the pure chitosan/PVA membrane.³¹

Zinc ions exhibit various hydrolysis and precipitation behaviors, forming species like $[\text{Zn}(\text{OH})(\text{H}_2\text{O})_x]$ and $[\text{Zn}_5(\text{OH})_6(\text{CO}_3)_2]$ in different conditions. At pH ~ 10 , $\text{Zn}_5(\text{OH})_6(\text{CO}_3)_2$ predominates when carbonate concentration exceeds 0.3 M, while ZnCO_3 forms between pH 5–9. The reaction of zinc with ammonium carbamate yields zinc oxide (ZnO) and is influenced by ammonium carbamate concentration, with a maximum precipitation yield of $\sim 83\%$ at 1.1 M concentration. Higher concentrations lead to soluble $\text{Zn}(\text{NH}_3)_4^{2+}$ complexes, reducing yield. SEM and XRD analyses confirm the formation of $\text{Zn}_5(\text{OH})_6(\text{CO}_3)_2$ and its decomposition to ZnO upon calcination. IR spectroscopy indicates carbonate and hydroxyl groups in the precursor, with their disappearance post-calcination. Particle morphology varies with zinc concentration and precipitating agents, affecting the final product's size and shape.³²

CS/ZnO nanocomposite demonstrated strong antibacterial activity against *S. aureus*, a bacterium responsible for skin infections. Additionally, cotton fabrics incorporated with CS/ZnO nanocomposite effectively inhibited the growth of *S. aureus*, *B. subtilis*, and *E. coli* pathogens. Furthermore, in sunlight, the nanocomposite efficiently degraded CR dye, demonstrating significant photocatalytic activity. This raises the possibility of uses in textiles and medicine to stop bacterial contamination and to function as a photocatalyst to remove dyes.³³

The growth of *S. aureus* and *E. coli* was totally inhibited by the synergistic action of CS, ZnO, and GO together. This hybrid composite of CS, ZnO, and GO shows potential in tissue engineering, adhesives, food packaging, water treatment, and pharmaceutical and medicinal applications for stopping the spread of germs and absorbing harmful colours.³⁴

The synthesis of chitosan (CS) and its hybrids with zinc oxide (ZnO) and graphene oxide (GO) was successfully achieved using a one-pot chemical strategy. Fourier transform infrared (FTIR) spectroscopy confirmed the formation of CS and its hybrids, with distinct peaks observed for ZnO and GO functional groups in the CS–ZnO and CS–ZnO–GO samples. X-ray diffraction (XRD) analysis indicated a predominant peak orientation of ZnO in both composites, with a decrease in peak broadening and an increase in crystallite size for CS–ZnO–GO. UV–Vis spectra showed a blue shift in the absorption edge for CS–ZnO–GO, indicating successful hybrid formation. Thermogravimetric analysis (TGA) revealed enhanced thermal stability for CS–ZnO–GO, attributed to the strong interactions and thermal barrier effect of GO. Mechanical testing demonstrated significant improvements in tensile strength and strain for CS–ZnO–GO. Morphological studies using scanning and transmission electron microscopy (SEM and TEM) showed agglomeration and nanosized grains, with increased surface area and pore volume for CS–ZnO–GO as confirmed by nitrogen adsorption-desorption isotherms. The hybrid exhibited superior adsorption capabilities for methylene blue (MB) and chromium complex (CC) dyes, outperforming CS and CS–ZnO in pollutant removal efficiency, highlighting its potential for environmental applications.³⁴

The XRD analysis shows that pure chitosan has peaks at $2\theta = 10^\circ$ and 20° , indicating its crystalline nature, while the CS/PVA matrix displays peaks at $2\theta = 19.7^\circ$ and 11.5° , confirming composite formation. Pure ZnO exhibits characteristic peaks corresponding to its hexagonal wurtzite structure, and the CS/PVA/ZnO composite retains ZnO peaks, indicating proper nanoparticle integration. FT-IR analysis reveals functional groups of chitosan, PVA, and ZnO, with the CS/PVA/ZnO spectrum showing characteristic peaks of all components and altered intensities due to ZnO mixing. SEM images demonstrate smooth chitosan surfaces, nanoflake-like ZnO morphology, and porous, rough CS/PVA/ZnO surfaces. TEM confirms ZnO nanoparticles appear as nanorods and are uniformly distributed on the CS/PVA matrix. Overall, the results confirm the successful synthesis and integration of ZnO nanoparticles into the CS/PVA matrix, maintaining distinct structural, morphological, and functional properties. CS, CS/PVA, and CS/PVA/ZnO treatments accelerated wound healing and reduced bacterial growth more effectively than the control, with CS/PVA and CS/PVA/ZnO showing the best results. Histology confirmed improved skin regeneration in these groups.³⁵

The chitosan/ZnO-PVP (CS/ZnO-PVP) nanocomposite was synthesized by mixing 25 mL of 0.1M zinc chloride with 0.1 g polyvinylpyrrolidone (PVP) and stirring at 80°C for 1 hour. This mixture was then combined with 0.1 g chitosan dissolved in 25 mL of 5% acetic acid and stirred for 30 minutes. A 0.2M NaOH solution was added dropwise, causing the solution to turn from pale yellow to a white precipitate, which was washed and dried at 80°C for 12 hours, resulting in the nanocomposite. The structural, elemental, and thermal properties were characterized using FTIR spectroscopy, XRD, HR-SEM with EDAX, TEM, AFM, TGA, and DTA. FTIR analysis confirmed the presence of functional groups of chitosan, ZnO, and PVP with characteristic peak shifts, while XRD indicated hexagonal ZnO crystalline structures. HR-SEM with EDAX and TEM analyses showed uniform ZnO distribution over the chitosan matrix, and AFM indicated a nanocomposite thickness of about 12 nm. TGA and DTA analyses demonstrated high thermal stability. The nanocomposite exhibited significant antibacterial activity against *S. aureus* and *E. coli*, with inhibition zones ranging from 17 to 24 mm. Overall, the CS/ZnO-PVP nanocomposite demonstrated enhanced thermal stability, crystallinity, and antibacterial properties, making it a promising material for biomedical applications.³⁶

Certain vapours are better suited for adsorption on the porous surface of the Cs/PANI nanofiber film. Methyl alcohol, dimethyl amine, and methylamine vapours were detected using thin sheets of doped and dedoped Cs/PANI nanofibers placed onto the QCM electrode. Dipolar interactions and hydrogen bonds may serve as the foundation for the adsorption process. The study examined the impact of temperature on the adsorption of dimethylamine vapours onto the dedoped Cs/PANI nanofiber film, and the adsorption process rate constants were computed.³⁷

The methodologies described are comprehensive and offer a thorough characterization of the hydrogels. The FT-IR, XRD, and SEM analyses provide valuable insights into the chemical structure, crystalline nature, and morphology of the materials. The swelling ratio is calculated using the formula $\text{SR\%} = (W_2 - W_1) / W_1 \times 100$, where W_1 is the initial weight and W_2 is the weight after 24 hours of swelling. Additionally, the swelling behavior and antibacterial activity tests yield functional data that are critical for practical applications.³⁸

The UV-Vis spectrum from 250 to 800 nm for PANI nanostructures with different ablation times in the chitosan solution shows main peaks at 277, 282, and 285 nm, which are related to the π - π transition of nitrogen excitation in benzenoid segments of the PANI nanostructure. The peaks at 445, 451, and 453 nm correspond to the shift of polariton to the π band of PANI. The red shift in the UV-Vis spectra indicates changes in the main chain of PANI.

The X-ray spectrum results for Chi-PANI-NC show main peaks at 8.23° , 17.74° , 26.77° , and 36.07° . A broad peak in the range of 18° to 35° and 36.07° is related to the amorphous nature of chitosan. The sharp peak at 8.50° is associated with acetic acid molecules present in the solution used for chitosan, located in the tunnels between PANI chains. The peaks at 17.74° and 26.77° represent the inter-chain distance between adjoining benzene rings in PANI and the dispersion of PANI chains at interplanar distance.³⁹

SEM images indicate that the CHP substrate has a smooth surface, while the ZnO film deposited on it forms a porous structure with spherical nanocrystallites, suitable for gas sensing. XRD analysis confirms the amorphous phase of the chitosan/PVP substrate (peaks at $2\theta = 9.38^\circ$ and 19.32°) and the polycrystalline hexagonal phase of ZnO (peaks at 31.74° , 34.41° , 36.12° , 47.53° , and 56.60°). Gas sensing tests showed that ZnO films on the CHP substrate responded well to H_2 concentrations of 0.5% and 2.0% at 150°C and 180°C , with sensitivity reaching 24% and 46% at 150°C and increasing to 69% at 180°C . The sensor exhibited good stability over multiple cycles, with response and recovery times of 18 and 35 minutes, respectively. Compared to ZnO on SiO_2/Si , which showed lower sensitivity, the ZnO/CHP sensor demonstrated superior performance, indicating that the biodegradable and flexible CHP substrate is a promising, cost-effective alternative for gas sensing and semiconductor applications.⁴⁰

II. Conclusion

Researchers have explored the synthesis of metal oxide nanoparticles embedded in chitosan and other polymers to enhance their properties for diverse applications. For example, chitosan-stabilized copper oxide nanoparticles have been incorporated into electrospun nanofibrous membranes, leading to improved antibacterial activity, thermal stability, and controlled release of copper ions. These enhancements were verified using techniques like X-ray diffraction (XRD), scanning electron microscopy (SEM), and dynamic light scattering (DLS). Similarly, nickel oxide/chitosan/polyvinyl alcohol (PVA) nanocomposite films demonstrated significant antibacterial effectiveness and dye removal capabilities compared to pure chitosan/PVA films.

Further research has focused on the development of chitosan-coated iron oxide nanoparticles, which exhibit enhanced antibacterial and antifungal properties, indicating potential for medical applications. Additionally, the incorporation of metal oxides such as magnesium oxide (MgO) and zinc oxide (ZnO) into chitosan matrices has shown strong antibacterial properties and photocatalytic activity, making these composites suitable for textile and environmental applications. Photocatalytic tests have revealed significant improvements in dye degradation, underscoring their potential for wastewater treatment and environmental cleanup. Characterization techniques like XRD, FTIR, and SEM are crucial in assessing the structural, chemical, and morphological properties of these nanocomposites, highlighting their promising applications in various fields.

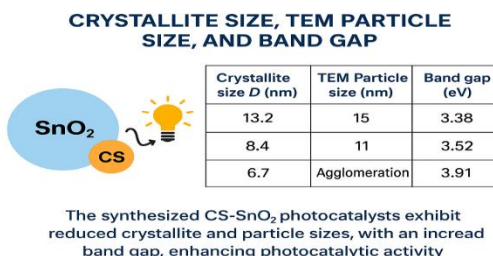
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Graphical Abstract for Table no. 1

Synopsis:- CS-SnO₂ photocatalysts show reduced crystallite size and increased band gap compared to pure SnO₂. The 75:25 ratio exhibits smallest crystallites, agglomeration in TEM, and highest band gap (3.91 eV), indicating enhanced photocatalytic efficiency due to improved charge separation and light absorption.



Pictogram

