

Synthesis of Cadmium Sulphide Reduced Graphene Oxide Nanocomposites and Evaluation of Their Antibacterial Activity

Binigha M¹, Dr. Rajesh Kanna Gopal²

¹Undergraduate, Saveetha Dental College and Hospitals, Saveetha Institute of Medical and Technical Sciences (Simats), Chennai -77, Tamil Nadu, India.

²Department of Microbiology, Centre for Infectious Diseases, Saveetha Dental College and Hospitals, Simats, Chennai - 600077. Email: rajeshkannag.sdc@saveetha.com, 152001011.sdc@saveetha.com

ABSTRACT

Background Dental caries is one of the most prevalent conditions. It is due to improper oral hygiene and from the frequent ingestion of sweets, which alters the biofilm microbiota that typically lives in the oral cavity in a state of homeostasis to one that is acidogenic, aciduric, and cariogenic. It is seen as a microbiological disease caused by diet, involving the formation of a cariogenic biofilm and frequent exposure to fermentable carbohydrates. This can be prevented by maintaining proper oral hygiene. In the present study, cadmium sulphide reduced graphene oxide was synthesised which is further subjected to characterisation and its antimicrobial activity was assessed against *E.faecalis* and *S.mutans*.

Materials and methods The nanocomposite cadmium sulphide reduced graphene oxide was synthesised by a simple precipitation method. The XRD, scanning electron microscopy, Fourier transform infrared spectroscopy, EDX confirmed the synthesised nanocomposite. Followed by evaluation of anti microbial activity of the synthesised nanocomposite against *E.faecalis* and *S.mutans*.

Results The antibacterial activity of CdS-rGO against *E.faecalis* and *S.mutans* shows increased activity at higher concentrations.

Conclusion From the study it is concluded that the anti-bacterial activity of Cds /rGO against *E.faecalis* is higher when the concentration increases similarly against *S.mutans* is higher with increasing concentration.

Keywords: Cadmium sulphide, reduced graphene oxide, anti biofilm activity, simple precipitation

How to cite this article: Binigha M, Gopal RK. Synthesis of Cadmium Sulphide Reduced Graphene Oxide Nanocomposites and Evaluation of Their Antibacterial Activity. *Int J Drug Deliv Technol.* 2026;16(19s): 852-857. DOI: 10.25258/ijddt.16.19s.99

Source of support: Nil.

Conflict of interest: None

INTRODUCTION

One of the most prevalent and ancient illnesses in humans, according to reports, is dental caries. Because cariogenic bacteria stick to teeth and metabolize carbohydrates to make acid, which gradually demineralizes tooth structure, dental caries is a common chronic infectious illness(1). Dental caries arises from the frequent ingestion of sweets, which alters the biofilm microbiota that typically lives in the oral cavity in a state of homeostasis to one that is acidogenic, aciduric, and cariogenic. This change may have a clinically indiscernible effect or cause a net mineral loss in the tooth's hard structures, which would manifest as an obvious carious lesion(2). Dental caries is seen as a microbiological disease caused by diet, involving the formation of a cariogenic biofilm and frequent exposure to fermentable carbohydrates(3). As per the World Health Organization, dental caries ranks fourth in terms of treatment costs, leading to a substantial worldwide

disease burden(4). The treatment for dental caries depends on the extension of the caries. It can be an invasive procedure or non-invasive procedure(5).

For the past few years, various research has been done on nanoparticles because of their unique properties. The size range of nanoparticles is typically 1–100 nm(6). They can manifest as high surface area nanopores, grains, fibers, films, nanorods, dots, and clusters of atoms. The antiviral, antimicrobial and antifungal properties of nanoparticles are demonstrated(7). Metal-based and metallic oxide-based nanoparticles have emerged as an advanced dental treatment option because they disrupt bacterial metabolism and stop the production of biofilms(8). In dentistry, nanoparticles are mostly used as fillers in nanocomposites. The nanoparticles in filling materials give mechanical strength to the materials. Nanocomposites contain nanofillers, resin matrix, photoinitiators, etc, that can be used for filling the

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prepared cavity(9).The primary benefits of nanocomposites over other types of composites are their high surface/volume ratio, which permits small filler sizes and decreased inter-particle separation; additionally, they are said to have improved mechanical, high ductility, scratch resistance, improved optical, and improved thermal properties. High gloss and gloss stability, as well as exceptional polishability and adaptability, are simplified and improved aesthetic qualities, particularly for dental nanocomposites. Nanocomposites may have drawbacks such as diminished toughness and impact performance(10).

Graphene is a carbon allotrope made of honeycomb-structured single-atom-thick carbon nanosheets. Reduced graphene oxide and graphene oxide are the primary derivatives of graphene. When oxygen-containing groups alter the graphene sheet, a derivative called graphene oxide (GO) is created(11). Excellent in terms of biocompatibility, GO has the ability to stimulate the directed differentiation of stem cells as well as cell adhesion and proliferation. The remarkable capacity of graphene oxide to prevent caries, The remarkable capacity of graphene oxide to prevent caries, tooth demineralization, and cariogenic bacteria(12). By removing GO's oxygen groups, rGO is produced and has less oxygen containing groups(13). In a previous study done by Priyadarsini et al., evaluated the use of graphene and its derivatives graphene oxide in various biological applications(14). Nizami et al., did an in vitro study on how the graphene can be used for the management of periodontal diseases and dental caries(15).

A great deal of interest has been shown in the manufacture and use of semiconductor nanoparticles, such as cadmium sulfide (CdS), because of their many uses as biosensors, photocatalysts, solar cells, diodes, and quantum dots for targeted medication delivery and treatment(16). Cadmium has a cytotoxic effect that is largely due to release of Cd²⁺ ions from the particles. Therefore, a reduction in nanoparticle size results in an increased surface to volume ratio and raises the possibility that the bacterial cells will be exposed to Cd²⁺(17).Malarkodi et al., evaluated the antimicrobial activity of cadmium sulphide against various oral pathogens(18)

The aim of the study was to synthesize cadmium sulphide reduced graphene oxide nanocomposite and evaluate its anti microbial efficacy..

Materials and Methods

Preparation of r-GO CdS

Synthesis of r-GO

Graphene oxide was prepared from graphite powder (325 mesh, 99%, Alfa Aesar Chemicals) by a modified Hummers method(59). In a typical procedure, 50 ml of H₂SO₄ (98%) was added into a 500ml flask and cooled by immersion in an ice bath. Then, 1.5 g NaNO₃ was added into the sulfuric acid solution under vigorous stirring to avoid agglomeration. After potassium nitrate was well dispersed, 1g graphite powder was added along the beaker wall. Then 8 g KMnO₄ was added gradually under stirring, the rate of addition was carefully controlled to keep the reaction temperature below 10 °C. After 2h, the ice bath was removed and the mixture was maintained at 35 °C by thermostatic oil bath for 6h. After the reaction, 200ml of H₂O was slowly added under vigorous stirring, the rate of addition was 3–5 drops per second, successively. Then the reaction temperature rapidly increased to 80 °C. After 30 min, the mixture was further treated with 200 ml of H₂O and 20 ml of 30% H₂O₂. For purification, the mixture was filtered and washed with 5% HCl aqueous to remove metal ions and through the centrifugal cleaning with DI water several times, the pH of the supernatant was neutral. After centrifugation and freeze drying in low temperature (–50 °C), graphene oxide was obtained.

Development of CdS coated r-GO

The CdS coated r-GO were obtained via a one-step hydrothermal method using supercritical water as green reductant. Briefly, 0.2 g of CdS were first added into the calculated amount of r-GO solution followed by ultrasonic dispersion for 30 min and vigorous stirring for another 2 h to disperse CdS sufficiently. Then, the mixing solution is transferred into a 50 ml Teflon-sealed autoclave at 150 °C for 5 h and cooled down to room temperature naturally. The resulting hybrids were recovered by centrifugation, washed with DI water and alcohol several times, and fully dried in vacuum at 60 °C for 12 h.

XRD

The Shimadzu model XRD 6000 powder X-ray diffractometer, which uses CuK α radiation and has a diffraction angle range of 10° to 70°, was used to record the XRD pattern of the CdS/GO nanocomposite.

FTIR

The surface functional groups of the biosynthesised CdS-rGO nanocomposite were analysed and discovered using Fourier transform infrared spectroscopy (FTIR, Perkin Elmer 65) at the scanning range of 4000-400 cm⁻¹.

SEM Analysis

Energy dispersive X-ray spectroscopy coupled with field emission scanning electron microscopy was used to analyse the biosynthesized CdS-rGO nanocomposite (FE-SEM).

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EDX analysis

The CdS-rGO nanocomposite sample's morphology and elemental makeup were examined using energy dispersive X-ray (EDX, Röntech xflash)

Antibacterial activity

Using a 96-well microtiter plate test, the anti-biofilm activity of CdS-rGO nanoparticles against *Enterococcus faecalis* and *Streptococcus mutans* was assessed.

Cultures were diluted in fresh broth after being adjusted to an optical density (OD) of 0.5 at 600 nm overnight. Wells containing CdS-rGO nanoparticles at doses of 25, 50, 75, and 100 ug/mL were filled with bacterial suspensions. The positive control (PC) was ciprofloxacin, and the negative control (NC) was untreated bacterial cultures. To enable the production of biofilms, plates were incubated for 24 hours at 37 °C. Following incubation, non-adherent cells were extracted, and phosphate-buffered saline was used to gently wash the wells. After fixing the adhering biofilm with methanol and staining it with 0.1% crystal violet, any extra stain was eliminated. Biofilm biomass was determined by measuring absorbance at 600 nm after the bound dye was dissolved in 33% acetic acid.

RESULTS

The development of a crystalline cadmium sulphide (CdS) phase and reduced graphene oxide is confirmed by the XRD pattern of the CdS/rGO nanocomposite. The graphitic structure of rGO is primarily responsible for the sharp peak at about $2\theta = 26^\circ$, which corresponds to the (002) plane and indicates restoration of sp^2 carbon layers following reduction. The (100), (101), and (110) planes of hexagonal CdS correspond to the extra peaks seen at roughly 28° , 44° , and 52° . This demonstrates that the CdS nanoparticles in the composite are crystalline. Good crystallinity is indicated by the comparatively sharp and well-defined peaks. The synthesized material's purity is demonstrated by the lack of further impurity peaks (**Figure 1**). The presence of Cadmium sulfide (CdS), reduced graphene oxide (rGO), and residual functional groups are all confirmed by the FTIR spectra of the CdS/rGO nanocomposite. The graphitic structure of rGO is confirmed by a prominent signal at 1642 cm^{-1} , which corresponds to C=C stretching of aromatic rings. C-H bending vibrations are linked to the band at 1371 cm^{-1} . C-O stretching is indicated by the peak at 1089 cm^{-1} , which suggests that rGO sheets still have functional groups that contain oxygen. The formation of CdS nanoparticles is confirmed by the bands at 790 cm^{-1} and 572 cm^{-1} , which are due to Cd-S stretching vibrations (**Figure 2**). The anti bacterial activity of CdS/rGO

against *E.faecalis* was evaluated. Initially both groups have high OD value indicating high bacterial concentration. With increasing concentration, the OD value is decreasing which indicates strong inhibition of bacterial growth at higher concentrations. The positive control (PC) reduces bacterial growth but comparatively less effectively than CdS-rGO (**Figure 4**). The antibacterial activity of CdS-rGO against *S.mutans* was also evaluated. With increasing concentration both CdS-rGO and PC groups show decreased bacterial growth. At higher concentration PC exhibits slightly higher antibacterial activity than CdS-rGO (**Figure 5**).

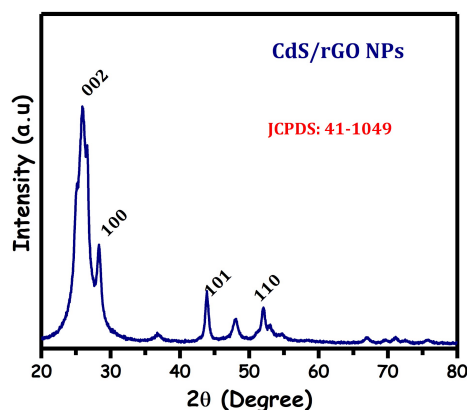


Figure 1: XRD Pattern of CdS/rGO nanocomposites

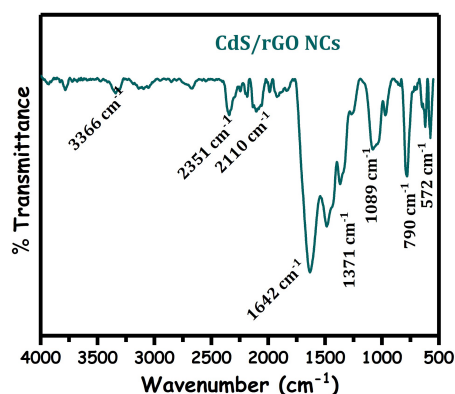


Figure 2: FTIR spectrum of CdS/rGO nanocomposites

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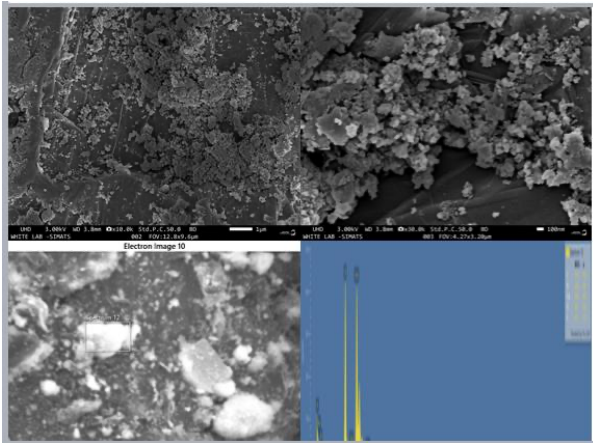


Figure 3: SEM and EDX spectrum of CdS/rGO NCs

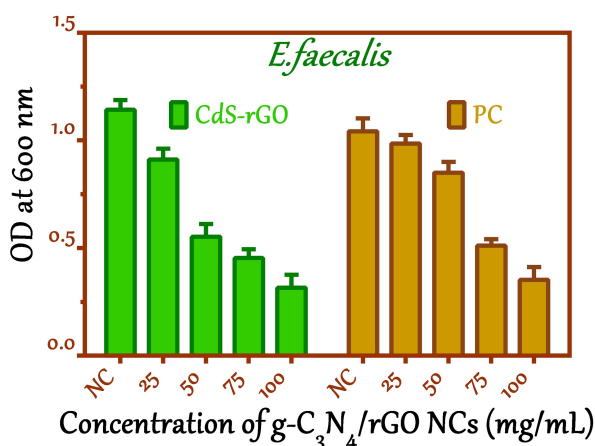


Figure 4 Antibacterial activity of CdS/rGO against *E. faecalis*

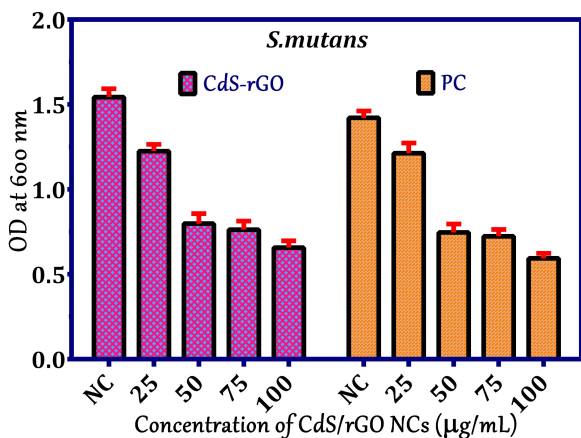


Figure 5: Antibacterial activity of CdS/rGO against *S. mutans*

DISCUSSION

One of the most important markers of overall well-being, and quality of life is dental health. It is well-known that the bacteria that colonize the oral cavity's hard and soft tissues have a major impact on oral health and can lead

to illnesses. Biofilms, which are highly organized, surface-associated assemblages of bacteria embedded in an extracellular matrix, can be produced by oral microorganisms connected to both hard and soft tissues. A multitude of oral disorders are caused by this colony of microorganisms clinging to surfaces. In recent times nanoparticles are getting more attention, even in dentistry it has significant applications. In the present study, cadmium sulphide reduced graphene oxide was prepared and its antibacterial activity was evaluated.

Cadmium sulphide reduced graphene oxide is synthesised by a simple precipitation method. It was then subjected to UV spectroscopy which confirms the presence of cadmium/ reduced Graphene. From FT-IR spectra the bonding between the metal and the functional groups was determined. From FE-SEM and EDS the morphology and the elemental compositions were confirmed. The antibacterial activity of CdS/rGO against *E. faecalis* is highest at the higher concentration similarly against *S. mutans* is highest at the higher concentration and is comparatively higher than the standard. In a previous study done by Zhao et al., on the inhibitory effect of reduced graphene oxide-silver nanocomposite on progression of artificial enamel caries concluded that rGO/Ag has a protective role on enamel caries progression

In comparison to other articles exploring nanocomposites, the study on binary cadmium sulfide reduced graphene oxide nanocomposites stands out due to its emphasis on antibiofilm activity and the simplicity of the precipitation method. Contrastingly, some studies might prioritise specific material properties or fabrication techniques. For instance, a research paper focusing on advanced fabrication methods could offer insights into alternative ways to synthesise nanocomposites, potentially introducing more complex but efficient approaches.

The focal point of interest lies in the nanocomposites' antibiofilm activity. Biofilm formation poses challenges in diverse sectors, from healthcare to industrial processes, and the ability of these nanocomposites to inhibit biofilm growth opens new avenues for addressing these concerns. The dual-action mechanism, involving the antimicrobial properties of cadmium sulphide and the mechanical stability imparted by reduced graphene oxide, showcases the potential for these nanocomposites as effective agents against biofilm-related issues produced by bacteria.

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Strong bacterial action against the growth of both Gram negative and Gram positive bacteria is demonstrated by rGO/CdS nanocomposites at various concentrations. It was shown that E. Coli was the species most susceptible to the composites(19).Both CdS nanoparticles and CdS/rGO nanocomposites exhibited strong antibacterial efficacy against Staphylococcus aureus and Escherichia coli, according to the zone of inhibition. The results of the above study were similar to the present study(20). The nanoselenium-reduced graphene oxide nanocomposite hydrogel has the capacity to destroy the biofilm formed which has a biomass reduction of upto 80%(21).

5.

Despite their promising features, binary cadmium sulfide reduced graphene oxide nanocomposites face a significant limitation associated with the use of cadmium. Cadmium is a heavy metal with known environmental and toxicity concerns. The potential release of cadmium from the nanocomposites raises questions about their ecological impact and safety in applications involving human contact. Overall, while the nanocomposites demonstrate remarkable properties, the cadmium-associated limitation underscores the importance of a comprehensive and responsible approach to their development and application. Further research could focus on enhancing the eco-friendliness of these materials by exploring alternative elements to replace cadmium, mitigating environmental concerns.

CONCLUSION

9.

The study of binary cadmium sulphide reduced graphene oxide nanocomposites represents a significant stride in materials science, showcasing a synthesis method that balances simplicity with multifunctionality. The demonstrated antibacterial activity holds promise for addressing challenges in healthcare. However, the environmental and toxicity concerns associated with cadmium necessitate careful consideration and mitigation strategies. The future holds exciting prospects, from exploring alternative materials to tailoring properties for specific applications.

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ACKNOWLEDGMENT:

The authors would like to thank all the participants for their valuable support and Saveetha dental college to conduct the study.

CONFLICT OF INTEREST: All the authors declare that there was no conflict of interest in the present study.

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