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Antibacterial Properties of Simple Chemically Fabricated ZnS/Graphene Composite

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ABSTRACT

In the present work a simple wet chemical method has been adopted for synthesis ZnS and ZnS-Graphene composite in low temperature range of 50-90 0C. XRD analysis shows cubic phase of thus prepared sample and morphology analysis by SEM indicates mono dispersed particle morphology. The graphene composite has been prepared and investigated, which was found to show enhanced antibacterial activity towards E.Coli in comparison to neat ZnS.

KEY WORDS: SEMICONDUCTING MATERIAL, GRAPHENE COMPOSITE, ZINC SULPHIDE, ANTIBACTERIAL ACTIVITY.

INTRODUCTION

To combat bacterial infections, antibacterial agents are widely used because of their powerful outcomes. Reagarding this, many researches has been focussed on the search for new antibacterial agents to combat the gradual developing resistance against potent antibiotics. However studies have indicated that widespread use of antibiotics has led to emergence of bacterial resistance to multiple drugs. Such abuse of antibacterial drugs are known to be the cause of development of superbacteria which are reportedly resistant to nearly all antibiotics owing to presence of gene NDM-1. In addition, reckless use of antibiotics has led to various health hazards to public health such as superbugs that do not respond to any existing drugs . Therefore, this necessitates a search for effective antibacterial materials. Regarding this, different nanoparticles have been used as alternative to antibiotics, which is because of the fact that nanoparticles are capable of preventing microbial drug resistance in certain cases .

Now-a-days intensive research is going on composite materials in which the matrix is usually a conducting substrate such as conducting polymer, graphene, metal powder etc. Further, it is known that, combination of such semiconducting nanomaterials with some typical conducting polymer matrix is expected to improve some of their useful properties such as thermal, mechanical properties, electro chemical properties and physicochemical qualities because of synergistic effects.

In this regard, graphene stands out as the most promising candidate to be a major filling agent for composite applications. At very low loading of fillers, such matrix have found to improved in multifunctional aspects in comparison to bare filler material. These materials are additional advantages like their lightness and simple and easy method of processing. In addition, these also make the material stronger for various multifunctional applications with simultaneous improvement in the physicochemical qualities of the host matrix upon distribution. This has additional benefits of increased interfacial bonds between the layers of graphene and the guest filler matrix. Such a bonding leads to reinforcement

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of improvement in synergistic properties in composites such as catalytic as well as antibacterial activities.

Synthesis method: All the Chemical reagents were of analytical grade without further purification and they are listed billow. Graphene powder, hydrogen peroxide, zinc nitrate, sodium sulfide, methylorange powder, sodium nitrate, potassium permanganate, these reagents werepurchased from Nice chemical private limited, Sulphuric acid, disodium salt of ethylene diaminetetraacetic acid (EDTA) and sodium dodecyl sulphate (SDS) wereprocured from CDH chemicals private limited, while Sodium borohydrade and Ethylene diamine were taken from Spectrochemprivate limited. The bacterial strain Escherichia coli (HO25), Nutrient agar, Nutrient broth,Mueller-Hinton agarwas provided from the Department of paramedics, CUTM.Odisha.

Preparation of ZnS: In a typical procedure 0.73 g of Zinc nitrate and 5g of EDTA/ 5mL of Ethylene diamine was added into 50 mL of water at 50 °C under stirring for half an hour. After that 5.9 g of SDS (5 times of CMC) was added followed by 0.6 g of sodium sulfide and temperature was increased up to 50 °C at reflux condition for 2hours. Then it was filtered and washed for several times to remove excesses amount of soluble impurities. Then the resulting mixture was allowed to dry in the vacuum oven at 60°C. The same procedure was repeated at 70 and 90 °C

Preparation of Graphene Oxide (GO): Aqueous suspension of GO was synthesized by modified Hummers method. In a typical procedure, 1.5g of graphite powder and 1g of Sodium nitrate was slowly added into 34 mL of sulphuric acid under stirring in an ice bath at below 10oC. After 30min 6g of potassium permanganate was added slowly, after 2 h of continuous stirring the resulting dark green suspension was removed from the ice bath to maintain normal temperature then 100 mL of water was slowly added to it under stirring which results in an increase in temperature up to 98°C. after that 10 mL Hydrogen peroxide was added, after addition of hydrogen peroxide a yellowish brown solution was obtained and it allowed to settle for hours, then the final mixture was separated and washed with water for several times to remove excess of sulphuric acid along with soluble impurities. After that the resulting black pasty mass was allowed to dry vacuum oven at 60 °C

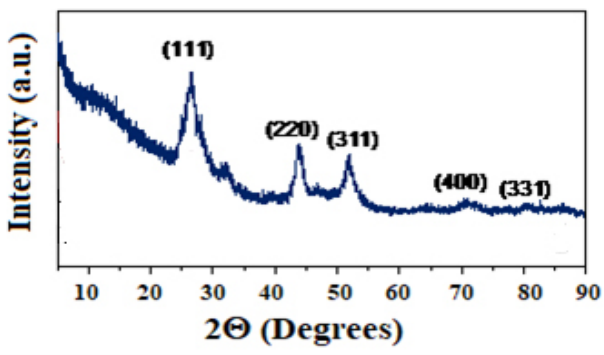
Preparation of reduced graphene oxide: To prepare reduced graphene 0.5 g of graphene oxide was suspended into 50 mL of water followed by the addition of 0.07 g of sodium borohydrade at 90 °C under reflux condition for 2 hrs with continuous stirring.

Preparation of ZnS-Graphene: ZnS-Graphene nanocomposite was synthesised by wet chemical method. Here 0.5 g of graphene oxide, 0.73 g of Zinc nitrate and 5 g of EDTA (or 5mL) of Ethylene diamine was added into 50 mL of water at 50°C under stirring for half an hour. After that 5.9g of SDS (5times of CMC) was added followed by 0.6 g of sodium sulfide and temperature was increased up to 90°C at reflux condition for 2hours. Subsequently 0.07g of sodium borohydrade was added into it, after 30min it was filtered and washed for several times to remove excesses amount of soluble impurities. Then resulting mixture was allowed to dry in vacuum oven at 60°C. Similarly ZnS-Graphene nano composite was prepared.

Antibacterial Study: The bacterial activity was studied by chromogenic medium (chromagar) and the antibiotic sensitivity profile was determined by using Mueller-Hinton agar (MHA*); the pH of the medium was adjusted to 7.0 and sterilized all media and glass wares at 25 lbs pressure and 120 °C temperature in an autoclave. Subsequently 20ml of agar medium was poured into either 100mm or 150mm petri-dishes and let's dry in a laminar air flow to solidify, which takes up to 20min.

Characterization: The structural characterization of the particles were done by X-ray diffraction (XRD) employing Philips X-ray DiffractometerXpert with CuKα radiation in the 2θ range of 5°-90°. Figure 1 and 2 shows XRD patterns of ZnS prepared with different templating agents viz EDTA and Ethylenediamine individually at 50, 70 and 90 oC. The XRD showing peaks at 2θ= 19.085, 28.151, 29.34, 34.55, 43.94, 51.83, indicate cubic phase of the sample according to JCPDS card 77-2100. The prominent peaks were seen to be broadened indicating the nanocrystallinity of all the samples. The analysis of XRD pattern stated that ZnS nanoparticles possess The average crystallite size determined using Debye Scherrer's equation,

Figure 1: XRD pattern of synthesized ZnS particle prepared with EDTA at (a) 50 °C, (b) 70 °C and (c) 90 °C.



$$D=K\lambda/(\beta \cos \theta)$$

Where K, λ, β, and θ are Scherrer constant, wavelength of X-ray radiation target used, maximum peak width in half height and angle of diffraction respectively. The presented data shows the composite synthesized at 90 oC with EDTAand EN, have crystallite size 92 nm and 98 nm respectively. In case of the graphene composite, there is a notable degree of decrease in intensity of the peak at 2θ centered around 25, which indicates appreciable degree of exfoliation of the graphene sheets [29-31].

Scanning Electron Microscope (SEM): The surface structure of the synthesized ZnS nanoparticles prepared with different temperatures were studied with the help of SEM. Figure 4 clearly reveal that the ZnS nanoparticles are irregular in shape and uniformly distributed. Figure 5 shows the SEM images of ZnS-Graphene nanocomposite from which it is evident that ZnS nanoparticles tend to be regularly distributed on the graphene platelets.

Figure 2: SEM images of ZnS in presence of EDTA at 90 °C

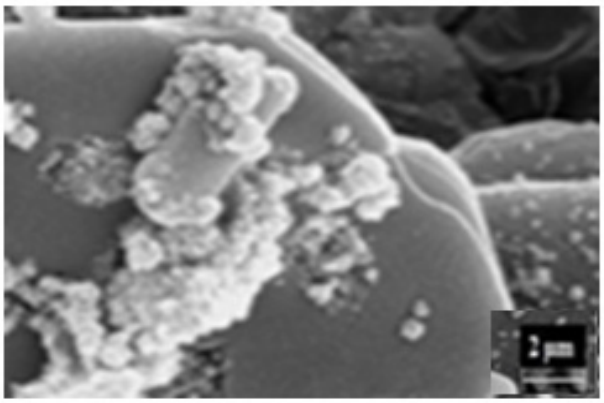


Table 1. Zone of inhabitations against *E.Coli* with respect to relevant specimens

Sl. No.	Types of compound	Zone of inhabitation (Diameter in mm)
1	ZnS/EDTA/90 °C	14
2	ZnSEN/90 °C	13
7	ZnS/Gr/EDTA	21
8	ZnS/Gr/EN	24

Antibacterial activity: The antimicrobial activity was studied using *Escherichia coli* bacteria. The inhibition zone was measured in millimetres around the 'well' having ZnS nanopowder synthesised at different experimental conditions along with graphene composite and has been represented in Figures 9 and 10 and corresponding inhibition zones observed are represented in Table 1. The inhibition zone. was found with ZnS which was prepared with EN at a temperature of 90 oC, which was 17 mm against *E. coli* and that in case of corresponding graphene composites was found to be 20 mm. It should be noted here that in case composite, the amount of ZnS was very small in percentage (~30% or nearly one third) and inspite of this it is showing appreciable antibacterial activity towards *E-coli* bacteria.

Moreover it is important to mention here that, the antibacterial activity performed using neat graphene only, does not indicate any inhibition zone. Thus, from above observations it can easily be concluded that the graphene composites inevitably shows greater antibacterial activity in the present case, which is ascribed due to synergistic effect [32-34]. The ZnS prepared with EDTA at temperature 50 °C had a smallest inhibition zone of 13 mm. While the composite which prepared with EDTA doesn't shows any inhabitation zone. This fact possibly due to formation of some type of organic coating around the sample due to which it inhibits the antibacterial activity and needs further research.

CONCLUSION

ZnS nanoparticles was successfully synthesised by wet chemical processes. XRD data and SEM images confirms that particle size varies at different temperatures as increase in temperature particle size increases. The uniformity of distribution of particles on graphene sheets has been confirmed by morphology analysis. Further, It also has been observed that the antibacterial activity also increases with higher particle size, which in turn, is higher in case of the graphene composite in comparison to neat compound.

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