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ABSTRACT

Graphene, a thin planar sheet of sp^2 hybridized carbon atoms has created a revolution over the past few decades in materials science and technology since from its invention. Owing to its excellent mixture of physical and chemical properties, it becomes a potential candidate in many interesting research areas such as supercapacitors, batteries, solar cells, nanoelectronics, metal oxides, functional nanocomposites, sensing, catalysis etc. Recently, efforts have been made to develop technologically feasible graphene-based devices. The development of composite materials based on graphene and natural polymers provide an ideal material in the biomedical field. However, the lack of good mechanical and thermal properties limits its applications. This drawback could be overcome by the reduction of graphene oxide into reduced graphene oxide (rGO). The rGO combined with the polymer helps to increase the adsorption of nanocomposites which thereby enhances the properties of the material to suit commercial applications. In addition to these, metal and metal oxide nanoparticles are decorated on the surface of polymer blended reduced graphene oxide that enhances the conductivity of hybrid nanocomposites. This present research is focused on the development of simple hybrid nanocomposites for the electrochemical determination of p-Aminophenol and biological applications. The metal and metal oxides nanoparticles ensembled on chitosan blended reduced graphene oxide nanocomposites are synthesized by chemical reduction method. The as synthesized nanocomposites are analyzed through an assortment of characterization to investigate their structural, morphological, and electrocatalytic properties along with their biological applications.

The graphene oxide is synthesized by modified Hummer's method and the chitosan functionalized reduced graphene oxide is synthesized by chemical reduction method. The structural, spectral, morphological and elemental studies of the synthesized nanocomposites are characterized by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDAX). XRD analysis reveals that the small diffraction peak at 10.3° corresponds to the plane of chitosan

and the broadening of the peak at 24° may be due to the amorphous nature of chitosan. The band at 1640 cm^{-1} corresponds to the NHCO stretching vibrations of amine groups of chitosan. The surface morphology of rGO/CS nanocomposites reveals that the formation of white patches on the surface of flat wrinkled leaf like structure of rGO. The electro-chemical behaviour for the prepared GO and rGO/CS nanocomposites is investigated using cyclic voltammetry. The rGO/CS nanocomposites exhibits high redox peak current for p-AP compared to GO modified GCE.

The various concentrations of (0.002M, 0.004M, 0.006M, 0.008M and 0.01M) zinc nanoparticles embedded on chitosan blended reduced graphene oxide nanocomposites are synthesized by chemical reduction method. The nanocomposites are characterized using XRD, FT-IR, SEM, EDAX, HR-TEM and SAED analysis. The XRD pattern of rGO/CS/ZnO nanocomposites shows the hexagonal wurtzite phase of ZnO and the crystallite size is found to be about 27.3 nm, 33.4 nm and 34.9 nm. The surface morphological analysis reveals that the rod shaped ZnO nanoparticles found to be homogeneously distributed on the surface of rough, thin, wrinkled reduced graphene oxide/chitosan nanosheets. EDAX analysis reveals the presence of carbon, oxygen and zinc without any impurities that further confirms the formation of rGO/CS/ZnO nanocomposites. The electrochemical behaviour of rGO/CS/ZnO nanocomposite towards the detection of p-AP at the modified glassy carbon electrode is studied using cyclic voltammetry. The maximum redox peak current is observed to be $0.06\mu\text{A}$ for pH 7 of 0.008M of rGO/CS/ZnO nanocomposite with a wide linear range of detection about $10\mu\text{M}$ to $180\mu\text{M}$. This is due to its uniform dispersion of ZnO nanoparticles on the large surface area of rGO/CS, leads to good electron transfer rate between p-AP and rGO/CS/ZnO nanocomposite modified GCE.

The various concentrations of (0.002M, 0.004M, 0.006M, 0.008M and 0.01M) copper oxide decorated chitosan blended reduced graphene oxide are synthesized by a facile chemical reduction method. The prepared nanocomposites are characterized by XRD, FT-IR, SEM, EDAX, HR-TEM and SAED analysis. The XRD analysis reveals that the crystallite size of copper oxide nanoparticles is in the

range from 9.16 nm to 18.5 nm. SEM and HR-TEM analysis reveals that the spherical shaped copper oxide nanoparticles well dispersed on to the chitosan blended rGO nanosheets. Elemental analysis reveals the presence of carbon, oxygen and copper without any impurities. Under optimized conditions, the maximum redox peak current is 0.18 μ A for pH 5 of 0.008M of rGO/CS/CuO nanocomposite and the linear range of detection about 60 μ M to 150 μ M.

The incorporation of silver nanoparticles onto the chitosan blended reduced graphene oxide nanocomposites are synthesized by chemical reduction method. The influence of various concentrations of (0.002 M, 0.004 M, 0.006 M, 0.008 M and 0.01 M) of silver nanoparticles on the surface of rGO/CS nanocomposites is investigated using FT-IR, XRD, SEM, HR-TEM and SAED analysis. XRD analysis reveals that the rGO/CS/Ag nanocomposites are well crystallized and the crystallite size is found to be about 23.6 nm. The band formed around 596 cm^{-1} confirms the formation of Ag nanoparticles on the surface of rGO/CS. The surface morphology analysis reveals that the spherical shaped Ag nanoparticles are well dispersed on the surface of rGO/CS sheets. The electrochemical property of prepared rGO/CS/Ag nanocomposites is investigated using cyclic voltammetry. The electrochemical behaviour reveals that the large effective surface area of the prepared nanocomposites allows it to function as an electron transfer medium and enhances the charge transfer rate.

The decoration of various concentrations of (0.002 M, 0.004 M, 0.006 M, 0.008 M and 0.01 M) of gold nanoparticles on the surface of rGO/CS/Au nanocomposites synthesized by chemical reduction method and investigated using FT-IR, XRD, SEM, HR-TEM and SAED analysis. The interaction of functional groups between rGO, CS and Au is confirmed using FT-IR analysis. It reveals that the band formed around 586 cm^{-1} corresponds to the stretching of metallic vibrations. XRD reveals that the nanocomposites are of high crystalline in nature with the crystallite size of about 8.9 nm to 16.5 nm. SEM and HRTEM analysis confirms that the spherical shaped gold nanoparticles are closely anchored onto the surface of rGO/CS nanosheets. EDAX analysis confirms the presence of carbon, oxygen and gold in the prepared nanocomposites. The electrochemical behaviour

reveals that 0.006M of rGO/CS/Au nanocomposites has shown prominent electrochemical behavior towards the detection of p-aminophenol.

The prepared nanocomposites are tested for antibacterial and anticancer activity. The antibacterial activity is done against Gram-positive (*B. subtilis* and *S. aureus*) and Gram-negative (*E. coli* and *K. pneumoniae*) using disc diffusion technique. The cytotoxicity effect is studied against Human hepatocarcinoma (HepG-2) cell lines. The prepared nanocomposite shows the reduction in cell viability by dose-dependent manner. These results attest an efficacy as a substantial material for the treatment of HepG-2 cancer cell lines. Hence the prepared nanocomposites may be applied in biomedical field.

In summary, prepared rGO/CS/ZnO, rGO/CS/CuO, rGO/CS/Ag and rGO/CS/Au show an excellent electrocatalytic activity which acts as a good electrode material and can be applied in environment for the detection of p-Aminophenol. Moreover, the nanocomposites show outstanding bactericidal activity and antiproliferative activity that can be potentially applied in biomedical field.

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ABBREVIATIONS

GO	Graphene Oxide
CNTs	Carbon nanotubes
rGO	Reduced Graphene Oxide
CS	Chitosan
NaNO ₃	Sodium Nitrate
H ₂ SO ₄	Sulphuric acid
KMnO ₄	Potassium Permanganate
H ₂ O ₂	Hydrogen Peroxide
N ₂ H ₄	Hydrazine hydrate
NH ₄ OH	Ammonium hydroxide
AgNO ₃	Silver Nitrate
HAuCl ₃ .3H ₂ O	Gold III chloride trihydrate
C ₄ H ₆ O ₄ Zn.2H ₂ O	Zinc Acetate dihydrate
CH ₃ COO ₂ C ₄ .H ₂ O	Copper II acetate monohydrate
NaBH ₄	Sodiumborohydrate
Ag	Silver
Au	Gold
ZnO	Zinc oxide
CuO	Copper oxide
AgCl	Silver Chloride
KCl	Potassium Chloride
KH ₂ PO ₄	Potassium dihydrogen phosphate
K ₂ HPO ₄	Dipotassium hydrogen phosphate
p-AP	p-Aminophenol
PBS	Phosphate Buffer Solution
GCE	Glassy Carbon Electrode
FT-IR	Fourier Transform Infrared
XRD	X-ray Diffraction
SEM	Scanning Electron Microscope
EDAX	Energy Dispersive X-ray analysis

HRTEM	High Resolution Transmission electron microscope
SAED	Selected Area Electron Diffraction
M	Molarity
CV	Cyclic Voltammetry
E_i	Initial potential
E_p	Peak Potential
I_p	Peak current
E_{pa}	Anodic peak potential
I_{pa}	Anodic peak current
E_{pc}	Cathodic peak potential
I_{pc}	Cathodic peak current
U	Scan rate
cm	Centimeter
mA	Microampere
μ A	Microampere
mM	Millimole
μ M	Micromole
IC50	Half maxima inhibitory concentration
g	Gram
μ g/ml	Microgram per millilitre
DMSO	Dimethylsulfoxide
MTT	3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide
FBS	Fetal Bovine Serum
a.u.	Arbitrary Unit
B. subtilis	Bacillus subtilis
K. pneumonia	Klebsiella pneumonia
S. aureus	Staphylococcus aureus
E.Coli	Escherichia coli
HepG-2	Liver cancer cell line