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Preparation of Chlorogenic Acid Based Green Zinc Nanocomposite Characterization and Anti - Inflammatory Study

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ABSTRACT

Nanocomposites are heterogeneous (multiphase) materials composed of at least one nanoscale phase (known as nanofiller) that is dispersed in a second phase (known as matrix) to obtain a combination of the individual properties of its constituents. Inflammation is the host body response to physical and chemical stress or injury in order to restore cellular homeostasis and tissue micro environment. "Nanocomposites are those composites in which one phase has nanoscale morphology like nanoparticles, nanotubes, or lamellar nanostructure. They have multiphase, so are multiphasic materials, at least of the phases should have dimensions in the range of 10–100 nm. The aim of the study is to prepare ZnO nanocomposite using coffee seed extract which contain chlorogenic aid.Growth of hydroxyapatite (HA) on gelatin-chitosan composite capped gold nanoparticles is presented for the first time by employing wet precipitation methods and we obtained good yields of HA. The function of starch-based polymers is limited due to poor mechanical properties. However, it is improved with forming a biocomposite of thermoplastic starch (TPS) as matrix and the cellulose fibers (CF) as reinforcement. The surface of cellulose fibers is successfully modified using the air plasma treatment with the aim of improving the matrix/fiber adhesion. Chlorogenic acid (CGA), an important biologically active dietary polyphenol, is produced by certain plant species and is a major component of coffee. Reduction in the risk of a variety of diseases following CGA consumption has been mentioned in recent basic and clinical research studies.

Key Words: *ZnO, NP(nano particle),SEM,FT-IR,, chitosan, coffee seed extract.*

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INTRODUCTION

Composite materials are prepared from the combination of two or more different materials with distinct chemical or physical characteristics [1].The resultant composite exhibits properties which are distinct to its constituent materials, which remain separate and distinct within the finished structure and are not held together by formal chemical bonds. In nanocomposites, either one of the constituents has dimensions on the nanoscale or instead the composite structure exhibits nanosized phase separation of the individual components.

Nanocomposites are the materials having multiphase solids with nano-size particles (<100 nm). Due to the presence of nano-size particles (reinforcement material), a discontinuous phase is created over a matrix (continuous phase) of standard material [2].This specific condition of nanocomposite provides mechanical strength, toughness apart from the expansion in both mechanical and thermal conductivity . Because of these properties, nanocomposites get their wide applicability as a functional and structural material[3].The characteristic advantages of nanocomposite are also attracting interest from different areas of applications, including catalysis, sorption, separation, fuel cell, etc.,

Fibers are available in nature and are mostly constituted of holocellulose (cellulose and hemicellulose together) and lignin, with minor contents of sugars, starch, proteins, extractives, and ash . Fibers from plants include leaf fibers (e.g., oil palm, banana, sisal, pineapple, abaca leaf); vast fibers (e.g., kenaf, jute, and flax); and seed fibers (e.g., cotton, rice husk, kapok, and coir)[4].The unidirectional cellulose microfibrils of plant fibers create the reinforcing elements in the matrix blend of hemicellulose and lignin]. Several advantages, such as availability, lower density and cost, environmental friendliness, biodegradation ability without emission of toxic gas along with good mechanical properties make natural fiber as one of the potent candidates for polymer-based nanocomposite[5].

Composites and nanocomposites can be prepared from a variety of different materials, depending on their intended application[6].The following areas are currently under exploration in the department. By far the most extensive industrial usage of composite materials relates to polymeric matrices reinforced

with glass or carbon fibres. Within the medical materials field, resorbable composites offer huge potential for controlled degradation and tailored temporary support to a healing site. Particle size and composition can be used to influence the mechanics and degradation profiles and active agents may be incorporated with sustained release tied to the degradation kinetics [8].

MATERIAL AND METHODS

Extract Preparation

Coffee seed first washed and then kept under the temperature of 15°C for 6h before being used for nanoparticle synthesis. Next, about 10 g of CS samples were powdered and dissolved into 250 mL DDW in an Erlenmeyer flask which was then subjected to continual stirring for 30 minutes at 60° C. After the boiling process, the extract (which was suspended in DDW) was cooled to room temperature. The solution is then filtered so that the extract can be collected and stored under the temperature of –20°C[9].

Synthesis of nanoparticles

The ZnO NPs were prepared according to the earlier report with slight modification 0.53 g of Zinc sulphate was dissolved in 50 mL of deionized water to prepare 0.2 M. This solution primarily appeared as a milky whiteclear solution, and to this 1 mL of acetic acid was added. In another vessel 1 g of NaOH was dissolved in 25 mL of deionized water to get 1 M NaOH. In a drop wise manner, NaOH was added to the contents of $ZnSO_4$ with continuous stirring, and was subjected to heating at 90°C. In an instant, white precipitate was obtained. The precipitate was then centrifuged at 1000 ppm for 10 min, repeatedly washed 4 to 5 times with deionized water and then dried at 80°C for 2 hours. The fine powder of ZnONPs obtained was used for the characterization.

Green synthesis of ZnO composite

For green synthesis of ZnO nanoparticle 10 mL extract were added before the addition of NaOH. About 25mL of acetic acid (1 %) was added in 0.5 g of chitosan, stirred well for 30 min at 50 ◦C. Then, 25 mL of 0.5M zinc sulphate solution was added drop wise to the Cs solution and stirring was continued for 3 h at100 ◦C. The obtained plant extract (25 mL) was added slowly to the Cs/ZnO mixture and stirred continuously for 2 h at 80 ◦C. Then about 25 mL of NaOH (1M) was added drop by drop to the reaction mixture untilthe formation of precipitate. The resulting precipitate was washed with double distilled water several times and dried at 50 ◦C in hot air oven.The ultraviolet-visible (UV-Vis) spectra of Zno-NPs were measured over a wavelength range of 200 to 1200 nm by using Perkin Elmer Lambda UV-Vis spectrophotometer

RESULTS AND DISCUSSION

The UV–Vis absorption spectrum of zinc oxide nanoparticles synthesized using coffe seed extract s an oxidizing agent agent is show in figure 1. UV–Vis absorption spectrum reveals the formation of zinc oxide nanoparticles by showing surface plasmon absorption maxima at 280-290 nm. The position and shape of the plasmon absorption depends on the particles size and shape. The TLC analysis shows both the NP and chlorogenic acid have same Rf value 0.68(plate 1)

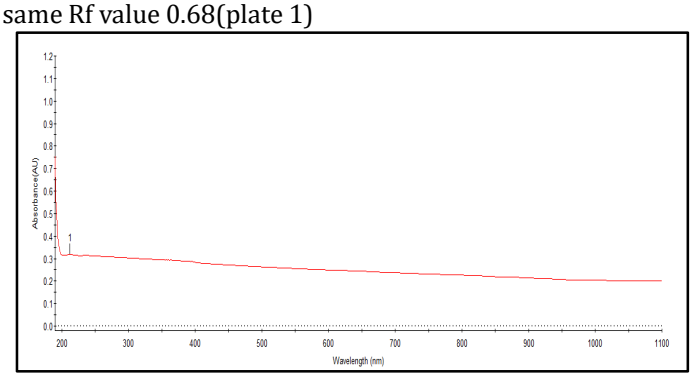


Figure 1 UV spectrum of zinc oxide nanoarticle NameNo.Peak(nm)Peak(AU)

Sample-AVN 1211.90 0.3180

Characterization of green ZnO Nanoparticle

SEM (FIG 2A) images of the ZnO nano partile seen, the spherical particles appear to be quite distinct and uniform, and the size of the particles ranges from 40 to 65 nm. XRD patterns of ZnO Np are shown in Figure 2. The major peaks at scattering angles (2θ) of 31.8°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, 66.3°, 68.1°, and 69.3° correspond to the lattice planes of (100), (002), (101), (102), (110), (103), (200), (112), and (201), respectively. These represent the wurtzite hexagonal phase of ZnO, confirming the formation of ZnO particles. The observed diffraction reflections are well-matched with the reported literature as well

as standard JCPDS data card No. 36–1451 [46] Other diffraction peaks referring to any impurities were not detected, suggesting that precipitated Zn(OH)2 was completely decomposed to ZnO.

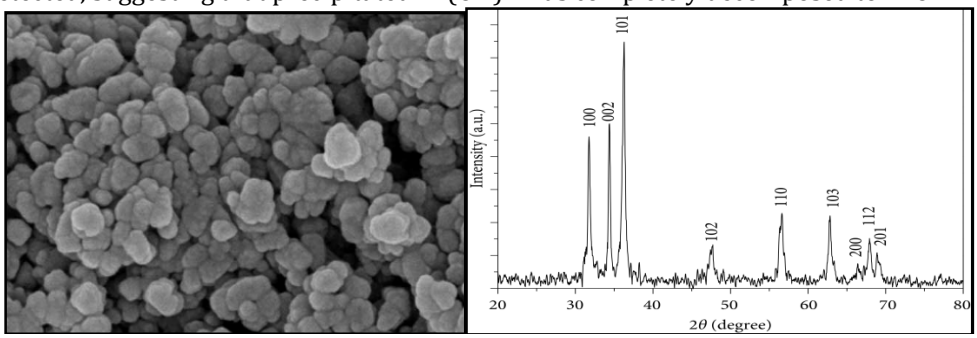


Figure 2 SEM and XRD pattern of green nano Composite

FTIR characterization NanoComposite

Polyemer of chitosan with ZnO and without ZnO were prepared (plate 2) .Figure 3 shows the FTIR spectra of ZnO nanoparticles, chlorogenic acid, chitosan, and ZnO/chitosan/Extract nanocomposite. Figure 3a the FTIR spectrum of chlorogenic acid shows the peak at 3449 cm^{-1} corresponds to the O–H stretching vibration; the peak at 1634 cm^{-1} may be due to the O–H bending vibration; the H–O–H bending vibration or the absorbed CO_2 bands may be responsible for the peak at 1382 cm^{-1}; and the band in the range of 528–435 cm^{-1} refers to the stretching mode of Zn–O. In the spectrum of chitosan (Figure 3b), the broad peak at 3463 cm^{-1} is due to the –OH/–NH2 stretching vibration; the peaks at 2930 and 2860 cm^{-1} are attributed to the C–H stretching vibration; the peak at 1651 cm^{-1} corresponds to the amino group bending vibrations; the peak at 1564 cm^{-1} may be due to the deformation of amide II; and the peaks at 1064 and 1023 cm^{-1} may refer to the C–O stretching vibration. Compared to the spectrum of chitosan, a new band from 528 to 412 cm^{-1} referring to the Zn–O stretching appears in the spectrum of ZnO/chitosan nanocomposite (Figure 3c). This indicates the existence of ZnO in the structure of the nanocomposite. In addition, the peak relating to the –OH/–NH2 stretching vibration in chitosan (at 3463 cm^{-1}) is broader and shifted to the lower wave number (3446 cm^{-1}) in the nanocomposite, suggesting the strong intermolecular hydrogen bonding interaction between chitosan and ZnO.

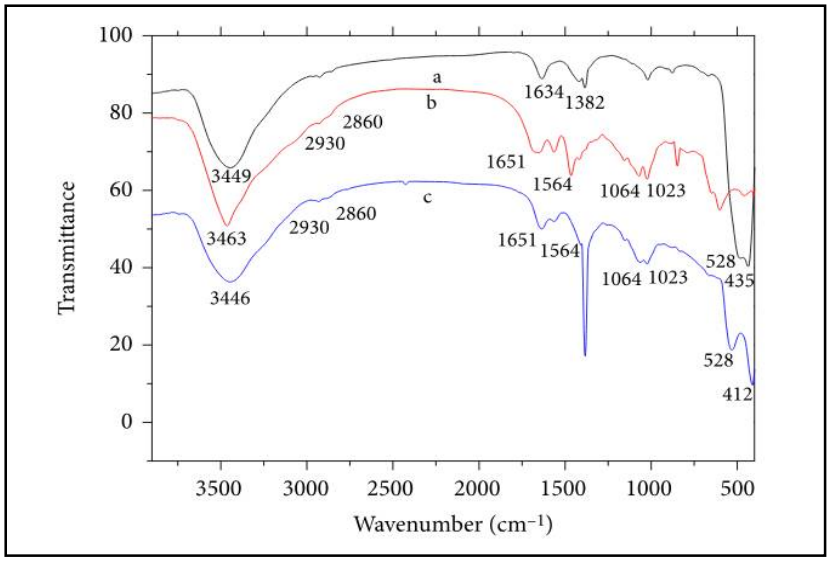
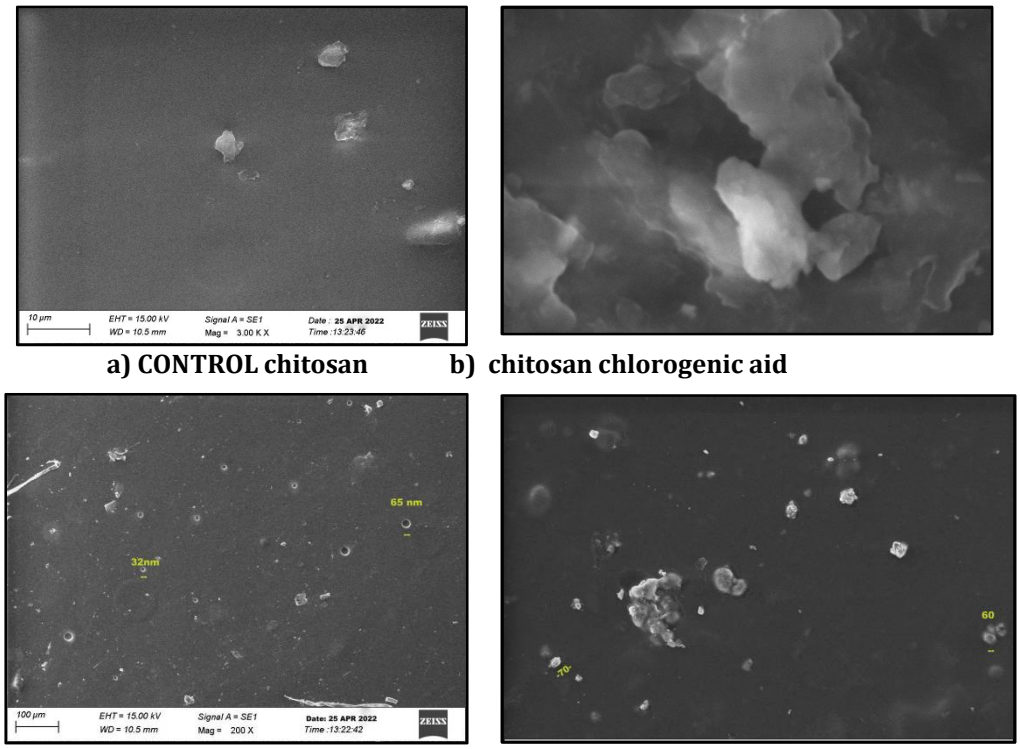


Figure 3 FTIR of nanocomposite

SEM characterization

Scanning Electron Microscopy (SEM) images of the zinc oxide nanocomposites were obtained using a Scanning Electron Microscope. Fig. 4 shows the SEM images of synthesized Zinc oxide nanoparticles on nanocomposite. Fig 4a control film shows uniform sheet without any nanoparticle and 4b shows large agglomerization of chotosan film. Fig. 4(c) shows SEM images of extract-ZnO nanocomposite. The ZnO nanocomposite show the sheet and spherical morphology and some particles have the tendency to dispreesed and the average particle size is about 32- 65 nm. These images show that after polymerization, on each of the particles, the polymer has grown and the dispersion is better. Fig 4d shows chemical mediated np on composite with 60-70 nm ZnO and agglomerization of np. Chemical mediated particles

were formed with size in the nano range, and also the particle separation is not good and this method of preparation was highly affected by particle agglomeration [14].



a) CONTROL chitosan **b) chitosan chlorogenic aid**

c) nano composite/extract **d) Chemical ZnO NP**

Fig 4 SEM image of Nanocomposite

Biological activity of nanocomposite

The control, chlorogenic and ZnO Np were tested against *E.coli.* They showed moderate ativity on chlorogeni aid(12 mm) and enhanced antibacterial performance via larger zones of growth inhibition (15 mm) among coffe seed extract ZnO nanocomposite (plate 3). The nanocomposites have exhibited good antibacterial property against Gram-negative organisms by virtue of the generation of reactive oxygen species (ROS) inside the cells [15]. Denaturation of protein is well documented for the cause of infection. Synthesised compounds of ZnONPs using coffee seed extract were tested for anti inflammatory activity on protein deanturation inhibition (plate 4) and proves that as the concentration (mg/mL) increases, the percentage of inhibition also increases. When the concentration is 100 µg the percentage of inhibition is also nearly 80% which is equivalent to 1000 µg activity of Dichlofenac. Further Chlorogenic aid exhibited 58% anti-inflammatory, chemical ZnO 24% , the control chitosan shows 12% activity (fig 5) and standard 68%.

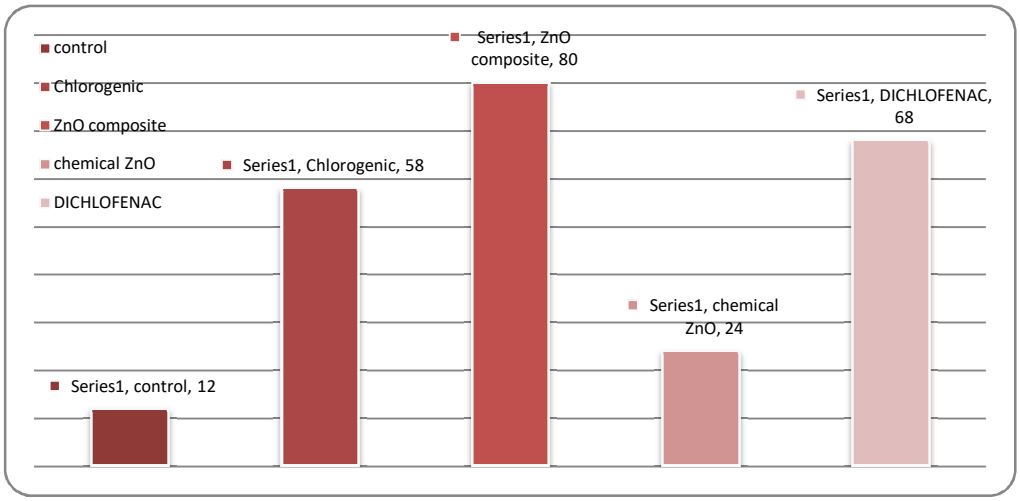


FIGURE 5: Percentage of anti-inflammatory

Plate 1 TLC of Np and chlorogenic acid

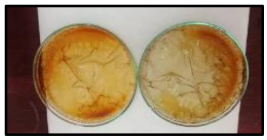


Plate2ZnONano Composite

CONCLUSION

Zinc oxide nanoparticles (ZnO-NPs) have healing, antibacterial, and antioxidant properties. Furthermore, ZnO-NPs also have anti-inflammatory properties. In this study, we synthesized a nanocomposite using ZnO by one step method[10].The structural and morphological properties of nanocrystals and nanocomposite were investigated by X-ray diffraction and scanning electron microscopic. The crystalline spherical structure of ZnO nanocomposite (spherical 65 nm) was determined. The chitosan film is well dispersed and uniform surface in all except chlrogenic acid amended. The nanocomposite demonstrated anti-inflammatory and antibacterial capacities. ZnO extract mediated exhibited antibacterial against MDR *E.coli* 15 mm inhibition zone and not exhibited by control and chemical ZnO synthesized. The anti-inflammatory activity of ZnO nanoparticles was done by protein denaturation assay. The seed extracted nanocomposite showed promising improved anti-inflammatory 82% activity by reducing the protein denaturation process, comparatively higher than standard dichlofenac. The current study has clearly demonstrated that the ZnO NPs are responsible for significant high anti-inflammatory activities. Therefore, the study reveals an efficient, ecofriendly and simple method for the green synthesis of ZnO NPs using green synthetic approach**.**

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest

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