Gold nanoparticle fortified bamboo biochar Nanocomposite

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Abstract

Gold nanoparticles due to their specific properties and function have found uses in the field of engineering to medical sciences. The Gold nanoparticles are always used in conjugation with other chemicals, metals, proteins, and other organic materials. The addition of other conjugates enhances the properties of Gold nanoparticles. As the insertion of metal nanoparticles into an organic matrix effectively increases the specific surface area of such materials, thereby enhancing the desired properties of the material. The term nanocomposite(NCs) is used for material containing an inorganic moiety, with at least one dimension in a nanometre range of 1–100 nm(nanoparticles) and other materials like metal, ceramics, and polymers. The term "Bionanocomposite" (BNCs)has been assigned to nanocomposites containing a component of biological origin in the mixture. In this work, Bamboo (Bambusa bambos) was used as the organic matrix for the preparation of gold nanoparticle biochar (Au-NPs/BC) nanocomposite. The one-step synthesis approach was used for the treatment of Bamboo with Auric Chloride Salt at room temperature. In addition to the above process, the bamboo was also pyrolyzed at low temperature after treatment, which helped further to reduce the overall cost of the method. This made the method of preparation of the nanocomposite low cost and eco-friendly. Various analytical techniques such as Fourier transform infrared (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDS), and UV–Vis spectroscopy methods were used for the characterization of the synthesized nanocomposite. This nanocomposite was used for the preparation of electrodes and its electrical conductivity was tested. In this method, the nanocomposite was prepared in a good amount via a very simple methodology. The characterization revealed the presence of gold in the nanocomposite, which confirms that this method can be used for the preparation of the (Au-NPs/BC) nanocomposite.

1. Introduction

Nanocomposites are high-end material, exhibit unusual combinations of properties, and provide new material design opportunities. There is a very high demand for the nanocomposite materials, Because of their attractive potential, there significantly improved mechanical and physical properties compared with original grain materials(Camargo et al. 2009; Tasnim et al. 2017). Expected benefits of nanocomposites include module improvement, flexural strength, temperature heat distortion, barrier properties, and other benefits. Their potential areas of application are very vast from structural, sensing to biomedical application(Jawaid and Swain 2017). Over the last few years, "bionanocomposite" has become a typical term assigned to nanocomposites containing a component of biological origin in mixture with an inorganic moiety, with at least a single dimension in a nanometre range of 1–100 nm(Mousa et al. 2016). The term "bionanocomposites" was used for the first time in 2004 also called "nanobiocomposites" (NCs), "green composites," or "biohybrides."(Khan et al. 2017). Bionanocomposites are somewhat similar to nanocomposites, but there are fundamental differences in there preparation methods, properties, functionalities, biodegradability, biocompatibility, and applications(Darder et al. 2007; Shchipunov 2012). These significant change in properties is provided by the biological or inorganic components present in the composite(Li et al. 2004). The structure of the nanocomposite typically consists of the matrix material which contains the nanosized reinforcement components in the form of particles, whiskers, fibres, nanotubes, etc. New nanocomposites material are being developed based on biochar, Endler, Leonardo W., et al has prepared nickel oxide (BC–NiO) nanocomposite by using *Acacia mearnsii* plant for the preparation of biochar(Endler et al. 2020). Similar works were done using Cerium Oxide to form nanocomposite using biochar as a base to test for the sonocatalytic performance of the composite (Khataee et al. 2018). The porous structured carbon materials have been used as electrodes due to their properties of high conductivity, large surface area, and the ease of surface modifications. The unique properties of these carbon materials have generated great interested, but complicated methods of preparation and the high cost have hindered their usability(Chikwendu Okpala). Biochar (BC) is a solid carbon-rich residue obtained by thermal decomposition of biomass in a low oxygen environment. The type of biomass used for the preparation of BC significantly influence its composition and thus influencing its properties(Břendová et al. 2012). The plant's compost of various complex microstructure and networks of highly interconnected channels. These allow efficient distribution of electrolyte in the electrochemical reaction system(Veiga et al. 2017). The other remarkable properties of BC produced by plants is they are nontoxic, heat resistance, and lightweight. Owing to their remarkable properties BC from plants has been utilized to absorb heavy metals, in new battery technologies, and many more. Furthermore, the modification done to the BC by the addition of different modifiers like metal and metal oxide nanoparticles can impart new properties to the BC electrode. The combination of Gold nanoparticle (Au-NPs) and biochar (BC) to form nanocomposites improves the efficiency of the electrode(Ferreira et al. 2018). The Gold nanoparticle (Au-NPs) offer unique electrical, optical, mechanical, and magnetic properties different from their bulk form. These properties can be explored in combination with the properties of BC to develop an analytical device to monitor environmental toxicants, catalysis, and many other biological applications. The use of nanoparticles improves the performance of the sensor and their electrochemical activity of the sensors. Here we have developed a simple method for the synthesis of gold nanoparticles biochar nanocomposites. The Bamboo was used to develop the nanocomposite due to its low cost and its ecofriendly properties. The characterization (SEM, XRD, and FTIR) was done to deduce the properties of the Gold nanoparticle, biochar composite (Au-NPs/BC). The prepared copper electrode was modified with the (Au-NPs/BC) nanocomposites in a very simple low cost method and can be used for different applications in a very simple method.

2. Experiments

2.1. Materials

The chemical Used i.e. Auric Chloride were purchased from Himedia chemical reagent Company. All other reagents and chemical compounds were of analytical quality and used without further purification. To prepare the electrode 2.0 mm copper wire, copper disk of diameter 8.0 mm, a PVC tube of 10 mm diameter and 50 mm length, the white cement was use as insulator material.G In the experiments, double distilled water was used as a solvent. The Bamboo (*Bambusa bambos*) was obtained from the botanical garden of Ayurvedic College (Raipur, Chhattisgarh)

2.2. Apparatus

The specimens were analyzed using Scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy (EDS) (X-Max) was done to find the elemental composition. The image was recorded at optimum settings of 20 kV. The powdered sample was used for analysis, which was crushed using a pistil motor. X-ray diffraction patterns (XRD) were obtained on a PANalytical X'pert Pro diffractometer using CuKa as the radiation source. The voltage for the operation was 40 kV, the current was 30mA and the scan rate was 5 min s^{-1}. The Bruker instrument was used for Fourier transform infrared (FT-IR) spectrum analysis. The electrical conductivity was analysized use Digital Multimeter.

2.3. Preparation of Gold Nanoparticle bio-char (Au-NPs/BC) nanocomposite

The Bamboo (*Bambusa bambos*) was obtained from the botanical garden of Ayurvedic College (Raipur, Chhattisgarh). It was used for the production of Bamboo biochar (BC). The synthesis of biochar is schematically presented in Fig. 1. The following steps were followed for the synthesis of (Au-NPs/BC) nanocomposite. The first step was washing; it was done to remove surface impurities from the stems of Bamboo. In the second step, the bamboo was cut into small pieces of 5 mm length. In the third step, 20 gm of the stem were dipped in aqueous HAuCl4 solution (2.5 mM, 50 mL) for three days, till the stem was completely soaked. For control, instead of the HAuCl4 solution, 1 g of the stems is steeped in distilled water. In the fourth step, the steeped stems were washed and dried, to remove any moisture at 80∘C for 24 hours in the oven. In the final step, the dried stems were subjected to thermal decomposition at 350∘C for 2 hours at 2∘C/minute in a muffle furnace. Black biochar was obtained at the end of the process, by slow thermal decomposition later it was crushed into fine powder to make the gold Nanoparticle biochar nanocomposite (Au-NPs/BC)(van Zwieten et al. 2010).

2.4 Preparation of modified electrodes from the nanocomposites

The working electrode was prepared by (Fig. 2.) (A) A 2.0 mm copper (B) copper plate or disk of diameter 8.0 mm (C) PCV casing was added as support, and (D) The insulating and supporting material was added (white cement). At the top 2.0 mm gap was left above the copper disk. The dimensions of the prepared electrode is 10 mm diameter and 50 mm length .The powdered(1 gm) nanocomposite(Au-NPs/BC) and Normal Biochar (Control BC) can be used as a modifier of electrodes in this experiment. The nanocomposite is mixed with Gum Arabic (Conductive and binding polymer)(Bhakat et al. 2018) as a binding agent in the ratio of 3:1. This mixture was filled in the gap in the prepared electrode; the electrode was polished and cleaned before the coating. Then both the modified electrode was dried at 80 ^{o}C in an oven. Then this electrode was used for further experiment.

3. Results And Discussion

3.1. Characterization of Au-NPs/BC nanocomposites

The most appealing aspect of biochar is that it represents a cost-effective, safe, and easy-to-produce process that allows the development of materials with broad applications at a lower cost compared to hydrocarbon or other chemical process materials. While most of the applications are still in their infancy, biochar can already be used with remarkable results in many applications Such applications include soil modification, catalysis, water purification, and storage of energy and gas. The most recent endeavor for the use of Biochar in developing the Sensors. The biochar derived from plants and plant-based sources are been utilized in many applications. The Pomelo fruit-based sensor is being used to develop high capacity batteries(Zhang et al. 2015), bamboo(Noman et al. 2014), coffee(Jagdale et al. 2019), sugarcane(Liu et al. 2019), and even rice husk (Haffiz et al. 2017)based biochar are used for sensors applications. Biochar has been used as a base material, which can be modified with different chemicals and materials(Zhang et al. 2015; Kouchachvili and Entchev 2017; Xiang et al. 2018) (Nanoparticles, Gold, silver). In general, the elemental composition of nanocomposite is affected by the type of plant used for the synthesis and thus effecting its properties(Břendová et al. 2012; Liu et al. 2012). The Change in structure and composition, of biochar, mainly occur during the heat treatment(Veiga et al. 2017). The electrocatalytic properties were analyzed of the Au-NPs/BC-350 nanocomposites prepared from Bamboo (Bambusa bambos)(Crombie et al. 2013). The fresh Bamboo stems were used as the permeation process is employed in the preparation process of the nanocomposite. The low cost and easy availability are some of the reasons to use this plant for the process. The peculation of the HAuCl4 aqueous solution in the precursor is very important to synthesize the nanocomposite. The duct and sieve tubes naturally present in plants stems helps in peculation of the HAuCl4 aqueous solution to the interior of the plants. The impurities and inorganic salts are removed during soaking; this is moreover due to the presence of large amounts of hydrogen ions in the HAuCl4 aqueous solution. The process of thermal decomposition carried on after the above treatment the functional groups such as CO, CO_2, and H_2O are removed as volatile elements and thus forming BC(Ahmad et al. 2014; Godlewska et al. 2017). The above process makes the BC more porous and leave the carbon structure(Singh et al. 2017). The decomposition of $HAuCl_4$ occurs due to heating and this process is further enhanced by the reducing environment formed by the BC. Thus, the nanocomposite of Au-NPs/BC is produced in the process.

(A) SEM and EDS Analysis

As-synthesized BC-350 Its morphology and structure were analyzed using SEM (Fig. 3A). In the SEM images, the sieve tubes and ducts are visible in the biochar. These structure facilities entry of metal salts and chemicals in the biochar. The Au-NPs / BC nanocomposite were powdered and analyzed for the presence of AuNPs in the samples. The Au-NPs / BC-350 SEM images (Fig. 3B) reveal many particles on the BC surface. These findings suggest the nanoparticles are inserted into the BC by the established method. The SEM image (Fig. 3B) and the EDS (Table 1.) study further supports Au's presence in the sample. The distributions of C, O, K, and Au were uniform in the sample. In the sample, the presence of C, O, and K suggests BC's plant origin(Hernandez-Mena et al. 2014). The SEM and EDS analysis thus show convincingly the BC fortification with gold nanoparticles and the nanocomposite formation.

Table 1
EDS analysis of the Au-NPs/BC nanocomposites

Element	Weight%	Atomic%
C	34.75	47.21
O	49.74	50.73
K	2.30	0.96
Au	13.22	1.09
Totals	100.00	

(B) XRD Analysis

The microstructure of Au-NPs/BC nanocomposites can be studied by XRD analysis. The XRD patterns exhibit peaks at 38.72, 44.94, 65.13, and 78.26, which correspond to the (111), (200), (220), and (311), standard phase of Au (refer JCPDS file reference no. 04-0784) (Fig. 4). This indicates the biosynthesized gold nanoparticles have high crystallinity. In the XRD patterns of the nanocomposite samples, two large diffraction peaks at 22.3 and 43.4 are due to graphite diffraction pattern (002) and (100)(Singh et al. 2017). Note, the potassium present in bamboo is associated with a self-activation effect which helps to form micro-pores in large amounts (Fahmi et al. 2018). This finding shows that nanocomposites of the Au-NPs / BC are partly graphitized.

(C) FTIR Analysis

Fourier-transform infrared (FTIR) spectroscopy has been used to analyze the effect of thermal decomposition on the surface functional groups of bio-char(Fig. 5). Most of the FTIR typically provides features from organic functional groups that are used to examine bio-char organic components. It would be expected the peak at 3420 cm^{-1} from organic O-H stretching with the Contribution of any water molecule that may remain in the sample or other hydroxyl group-derived minerals. The band at 2920 cm^{-1} is associated with C ≡ C alkyne stretching in hemicellulose. The lignin aromatic group gives rise to asymmetric stretching of C = C at 1576 cm-1 indicating a band of G(Chen et al. 2015). The C-H bending modes decrease at 876 cm^{-1} and emit CH_4 gas as the temperature rises(Chia et al. 2012). The FTIR analysis confirms all cellulose, hemicellulose, and some lignin content in the bamboo.

3.2. Electrochemical behavior of Au-NPs/BC nano-composites

Nanocomposite-modified electrodes were built to investigate the electrochemical activity of the modified electrode. Both the electrodes the one modified with Au-NPs/BC and the control electrode modified with BC were tested for comparative resistance using digital multimeter. The Au-NPs/BC electrode showed less resistance compared to BC modified electrode. The resistance of both the electrode was less than 5 ohms, which is a mark of a good conductor and thus a good electrode.

3.3. Stability and reproducibility of Au-NPs/BC

The study of the stability and reproducibility of the modified electrode should be tested for the storage at room temperature. Despite the benefits of composite nanoparticles-biochar, such as increased biochar production, different surface area, and nanoparticles, the potential risk of metal oxide nanoparticles' environmental and biological toxicity are to be considered(Kahru et al. 2008). Further research should therefore be carried out to improve the stability of the Nano-composites based on the biochar. There are many factors in the production and synthesis processes of biochar-based nanocomposites which can have a significant influence on the properties of the resulting materials. With the production conditions, Biochar properties will change, making the properties of biochar-based nano-composites vary accordingly. the properties and adsorption ability of biochar-based nanocomposites are affected by Biomass type and pyrolysis conditions[27]. The Biochar effects the absorption ability of the sensor, this can be attributed primarily to the different composition and content of biochar lignin, cellulose, hemicellulose, and inorganic salts. Besides the contents, the pyrolysis conditions (thermochemical conversion technology, the temperature of pyrolysis, residue time, etc.) can also have a major influence on the adsorption performance of biochar

based materials[28]. In our assessment, the amount of nanocomposite (Au-NPs/BC) in the electrode is very small (1 gm) there the risk of environmental contamination is very less and the coating is very stable. Table 2 summarizes the biochar modified with different materials and their applications in different fields. The modified biochar are being used in various electrical and electrochemical applications. The focus of our study is to develop biochar for sensor applications, biochar has been used to develop, Humidity sensor(Ziegler et al. 2017; Jagdale et al. 2019), Pressure sensors, electrochemical sensors(Kalinke et al. 2016; de Oliveira et al. 2017; Liu et al. 2019). The final approach of this experiment is to utilize the different properties and to develop a sensor from the biochar developed by the above procedure.

Table 2
Biochar and their applications

	Name of Biochar Composite	Base material	Modifies	Used for sensing / Application's	Special features	Reference and Year
1.	Gold nanoparticle-decorated biochar (Au-NPs/BC) nanocomposites	The leaves of *Dracaena sanderiana* plant was used	Gold (Auric Chloride Salt)	Used for electrochemical sensing of **hydroquinone** and **catechol** by coating the GCE electrode	Simultaneous determination of HQ and CA with very low detection limits and high sensitivities.	(Xiang et al. 2018)
2	Selenium loaded macro-/micro-porous biochar-based (Se/MMPBc) Composite	Inner spongy layer of **Pomelo** fruit	Selenium	Applied as the cathode material of lithium-selenium batteries (Energy Storage)	Helps in excellent electrochemical durability of the composite.	(Zhang et al. 2015)
3	Ag/BC composite	BC formed by carbonization of ash tree residue at 700°C	BC was chemically activated in an Silver salt (Ag_2SO_4/HNO_3)solution	Ag/BC composite Was used to make electrodes That showed high specific capacitance(Energy Storage)	Ag/BC composite based cell Showed excellent energy density	(Kouchachvili and Entchev 2017)
4	SWP700 biochar(Commercial Biochar) and Polyvinylpyrrolidone (PVP) was added as binder	SWP700 biochar(Commercial Biochar)	Polyvinylpyrrolidone (PVP) was used as binder to adhere biochar onto ceramic substrates having platinum electrodes	Humidity sensors	biochar materials are behaving as p-type semiconductors under low amounts of humidity. .	(Ziegler et al. 2017)
5	Coffee ground biochar (CGB) based Ink	Coffee ground biochar	Polyvinyl butyral (PVB) as a binder and ethylene glycol monobutyral ether	Humidity sensors	Screen-printed films tested for impedance	(Jagdale et al. 2019)
6	Biochar activated with nitric acid	Biochar	Nitric acid	Carbon paste electrode modified with biochar for Electrochemical Sensing	Detection of **Methyl Parathion**	(de Oliveira et al. 2017)
7	Biochar/PDMS foams	Biochar from pyrolysis of rice husk	Biochar/PDMS foam is sandwiched between copper electrodes	Pressure sensors based on polymer foams	foams display negative pressure coefficient of resistance	(Haffiz et al. 2017)
8	Biochar nanoparticle/ Tyrosinase Enzyme / Nafion membrane/ Glassy Carbon electrode(BCNPs/Tyr/Nafion/GCE)	Sugarcane derived biochar	Modified with Tyrosinase Enzyme	Used GCE electrode for electrochemical sensing of **Bisphenol A**	Due to high conductivity property electode Show higher sensing signal, decreased impedance and lowered reduction potential	(Liu et al. 2019)
9	Carbon paste electrode modified (CPME-BC400) with biochar	Biochar obtained from castor oil cake	Carbon paste was modified with Biochar at 400ºC	Determination of **paraquat (PQ^{2+})**	Differential Pulse Adsorptive Stripping Voltammetry (DPAdSV) was used for sensing	(Kalinke et al. 2016)

	Name of Biochar Composite	Base material	Modifies	Used for sensing / Application's	Special features	Reference and Year
10	Modified carbon paste electrode (MCPE)	Biochar obtained from castor oil cake	Prepared using mercury ions (Hg^{2+}) supported at a biochar surface	Determination of zinc(II) ions present in commercial samples (collyrium and ointment)	Interactions between the highly functionalized biochar surface and zinc(II) ions followed by reduction of ions into mercury droplets	(De Oliveira et al. 2015)
11	Biochar functionalized by Biomolecules	Biochar was obtained from (Commercial Biochar)	functionalized by covalent binding between BC and biomolecules by EDC/NHS conjugation.	Used for immunoassay application to detect Hantavirus	Diagnostic is based on the presence of specific hantavirus nucleoprotein (Np), under viremic condition or IgG2b antibodies (Ab)	(Martins et al. 2019)
12	Nanostructured copper hexacyanoferrate Biochar modified carbon paste electrode (nCuHCF-BM-CPE)	Biochar was obtained from (Commercial Biochar)	Modified with nanostructured copper hexacyanoferrate (nCuHCF)	Application for amperometric sensor for isoniazid (INZ).	Electrode showed significant improvement in the current signal for INZ oxidation	(Oliveira et al. 2018)

4. Conclusions

In summary, we report a simple, scalable, and cost-efficient method for the preparation of Au-NPs / BC nanocomposites with high surface area, excellent electrical conductivity, and moderately high porosity. This method resulted in a uniform distribution of gold nanoparticles within the BC as shown in SEM and EDS results. The Au-NPs/ BC nanocomposite produced at low (350^{o}C) temperature and in a muffle furnace. The low temperature and use of simple instruments (instead of Pyrolysis) have contributed to the low cost of the method. Most notably this method can be used for the synthesis of similar types of BC modified with metal (or metal oxide) nanoparticles and other types of plants. The copper electrode is also prepared in a very simple method and is easy to modify to suite any type of potential sensing application. The potential of these BC materials for energy storage systems, environmental monitoring, catalysis, and biological applications and sensing is significant. The experiment showed that BC has excellent electrochemical properties, even though reproducibility is currently lacking. Further investigation will be performed to improve the reproducibility of the nanocomposite and find a suitable application of the modified biochar.

Declarations

Ethics approval and consent to participate

"Not applicable"

Consent for publication

"Not applicable"

Availability of data and material

"Not applicable"

Competing interests

The authors declare that they have no competing interests.

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Authors' contributions

The First author Aditya Lawerence Toppo has contributed, through experimentation work and analysis of the result.

The second (Corresponding) author Jujjavarapu Satya Eswari provided necessary guidance in designing the experiment.

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Figures

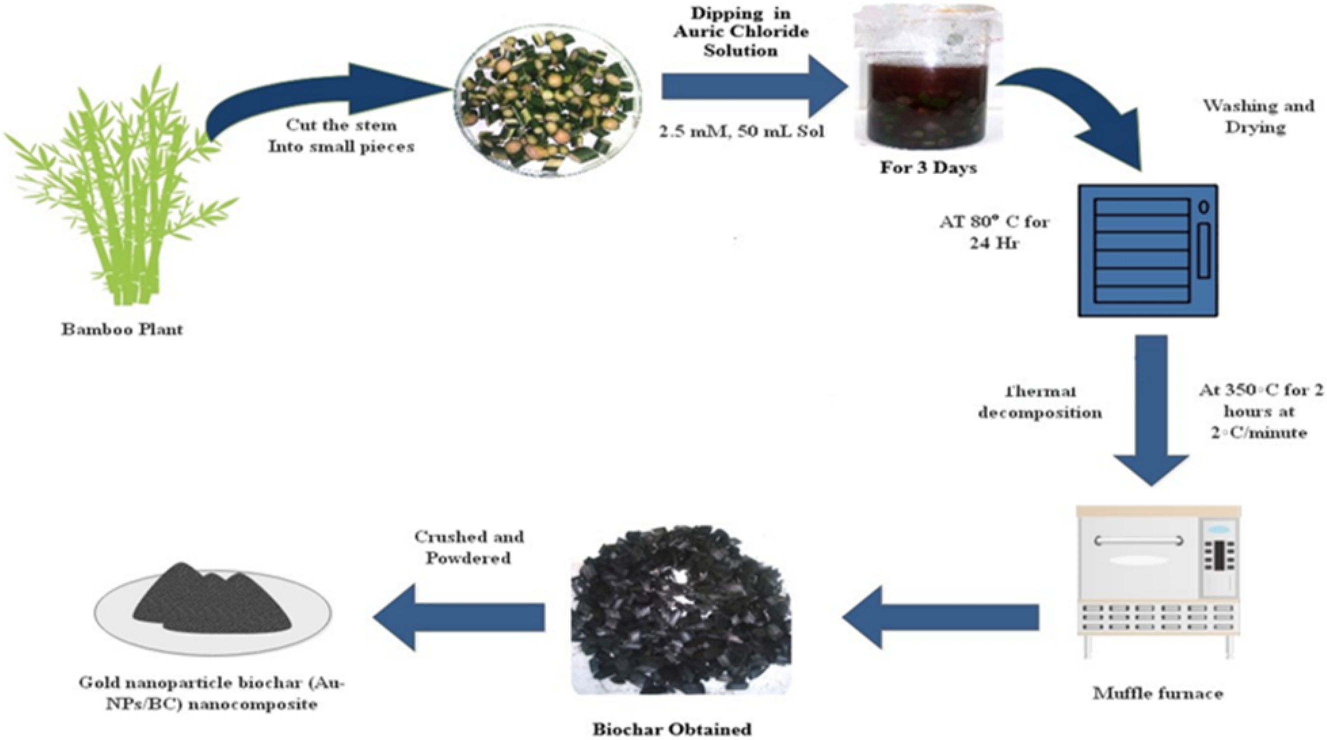

Figure 1

Steps Involved in Biochar Synthesis

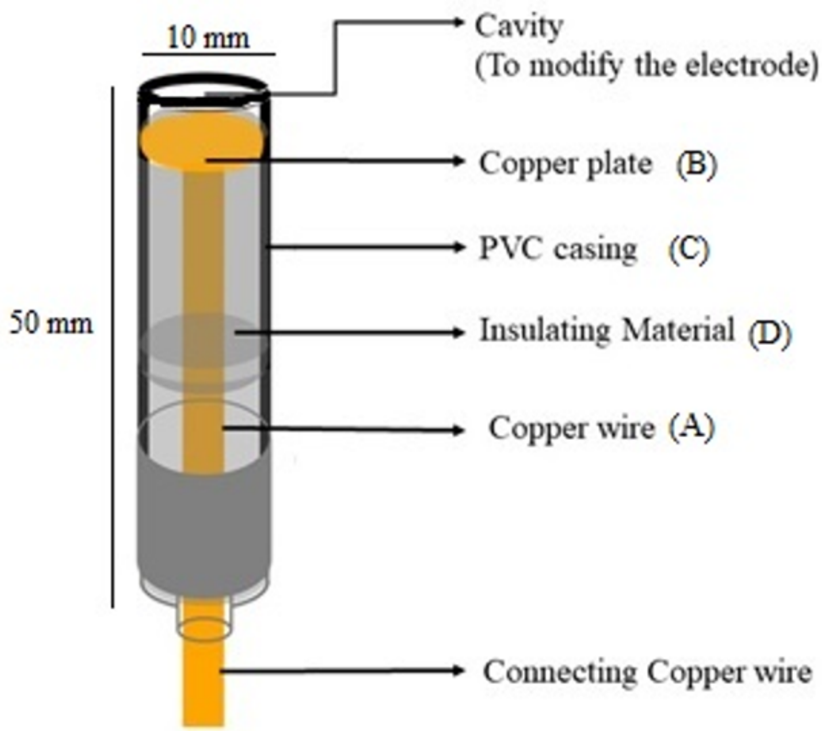

Figure 2

Schematic design of the electrode (A) Copper wire (B) Copper Plate (C) PVC casing (D) Insulating Material

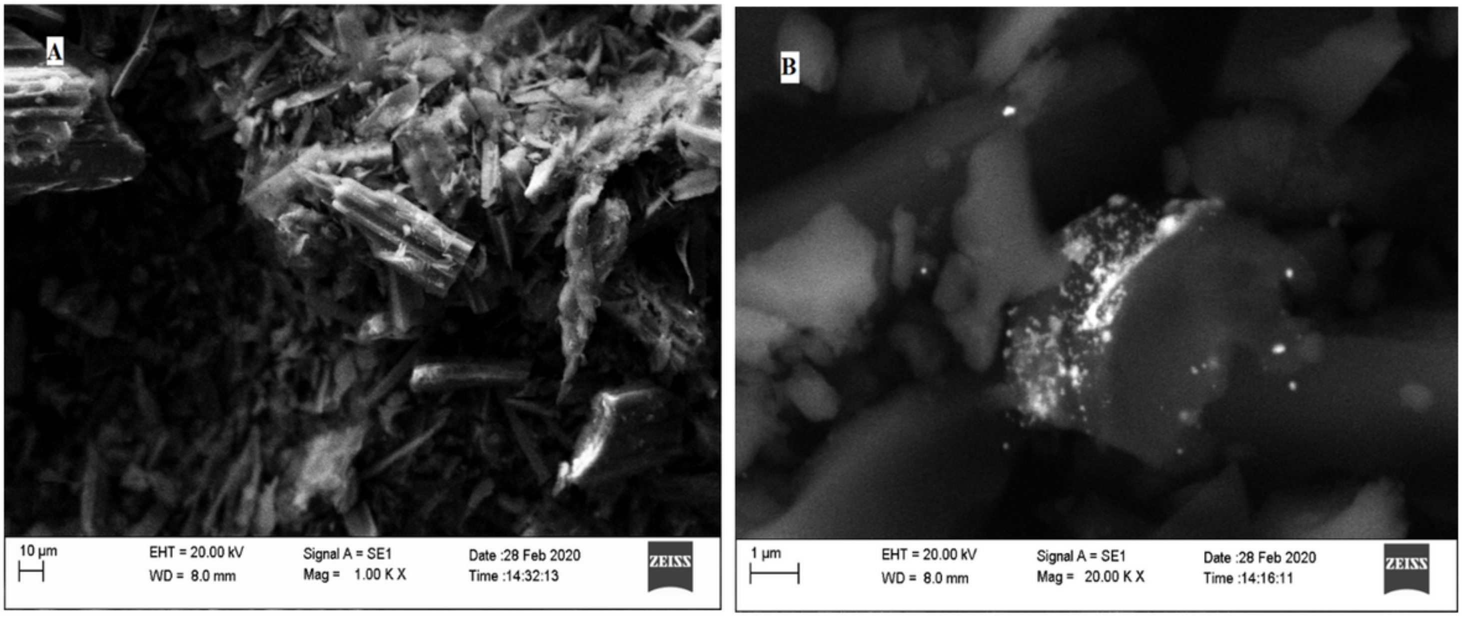

Figure 3

The SEM analysis of (a) BC control (b) Au-NPs/BC-350. The presence of Au-NPs is seen in the image (b)

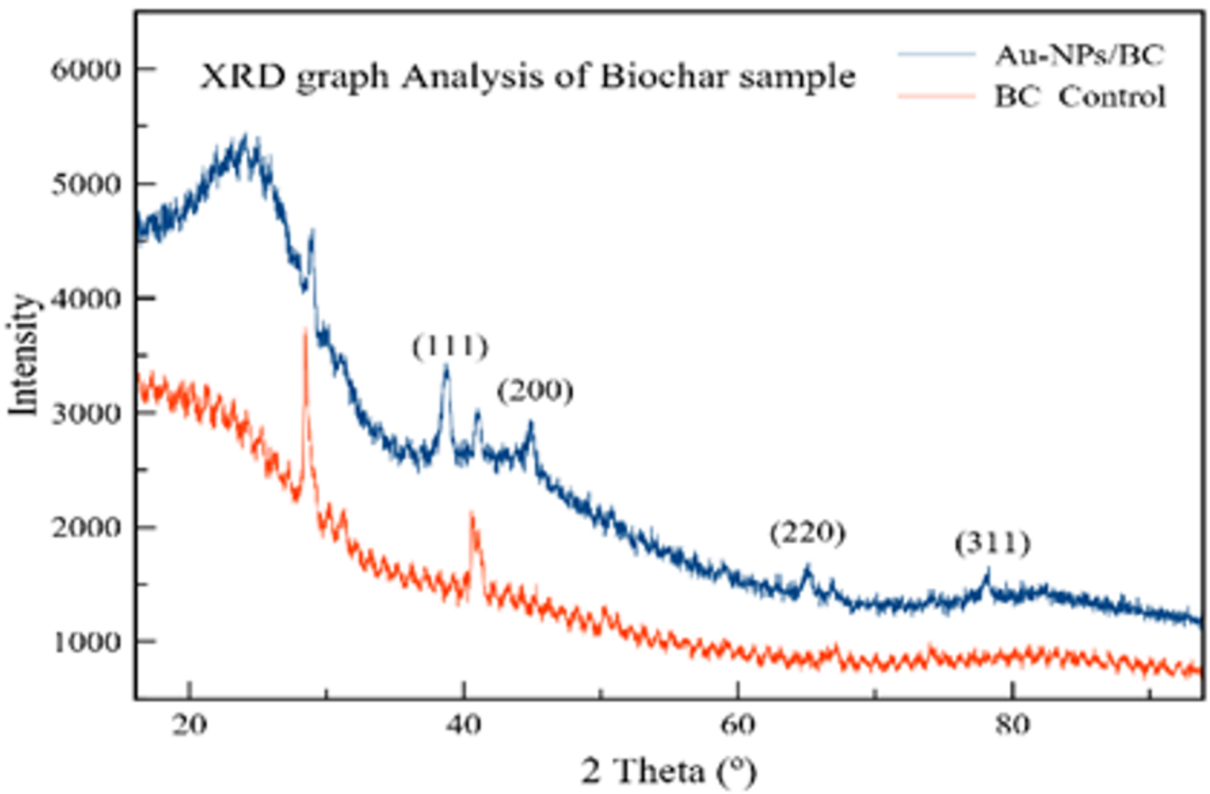

Figure 4

XRD analysis of the Biochar sample.

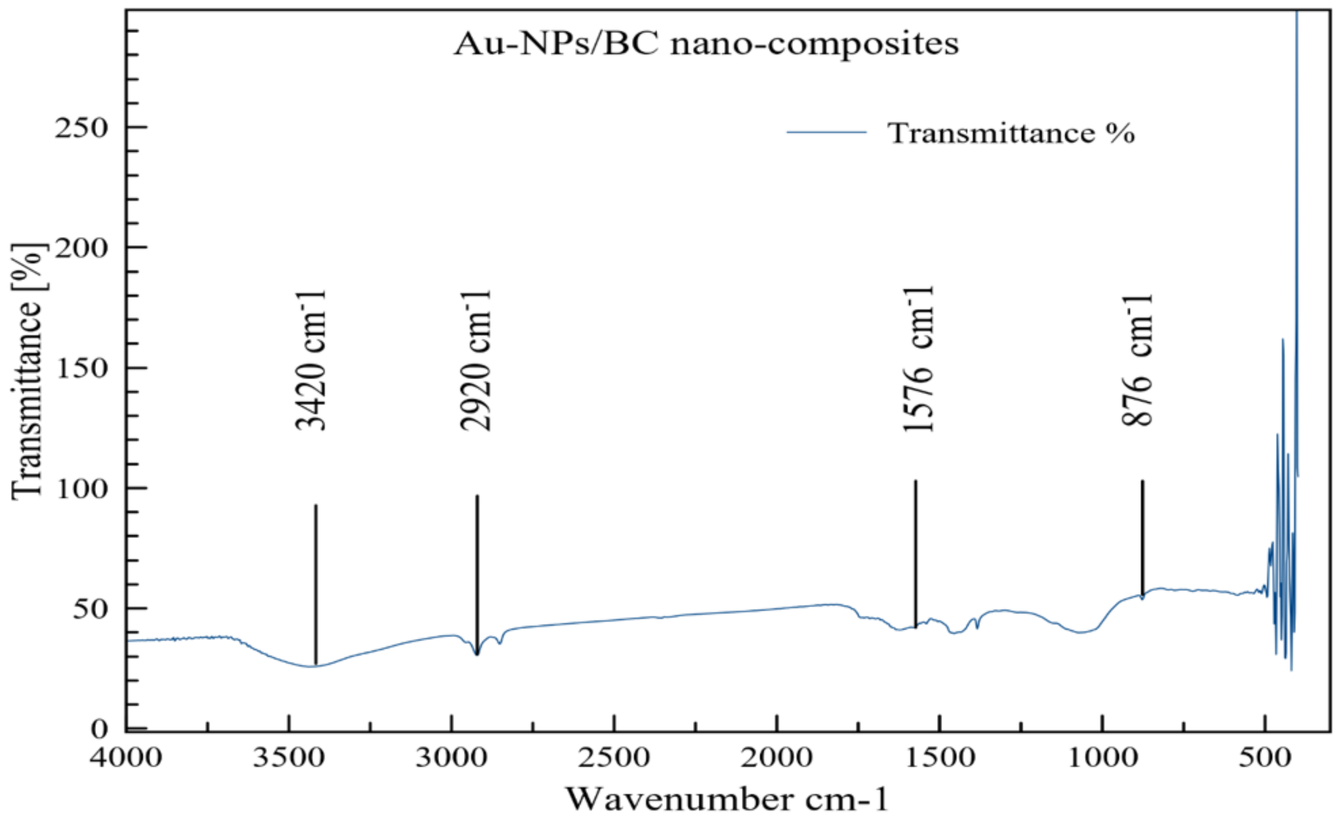

Figure 5

Fourier-transform infrared (FTIR) spectroscopy

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- GraphicalAbstract.docx