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Synthesis and characterization of gold nanoparticle-mediated bamboo biochar nanocomposite-based electrode and analysis of its electrochemical behavior

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The current study describes a low-cost synthesis of gold nanoparticle biochar (Au-NPs/BC) employing *Bambusa bambos* (bamboo) as an organic matrix for the Au-NPs/BC and bio-electrochemical application. Synthesized nanoparticles were characterized by fourier transform infrared (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and UV–Vis spectroscopy. This nanocomposite is employed for electrode coating, and its Bio/electrochemical characteristics were investigated using the microbial fuel cell. Results showed that the nanocomposite production and BC fortification with gold nanoparticles are conclusively by SEM and EDX. The SEM analysis of the Au-NPs/BC revealed the distribution of gold nanoparticles on the BC surface, confirming successful integration. Furthermore, the EDX results indicated anAu-NPs of 13.22% (*w/w*). The XRD patterns exhibit peaks at 38.72, 44.94, 65.13, and 78.26, which correspond to the standard phase of Au. Furthermore, FTIR analysis confirms that biochar contains cellulose, hemicellulose, and a trace amount of lignin. Finally, the modified electrode exhibited higher conductivity in bio-electrochemical experiments than the conventional carbon electrode. The CV graph showed the maximum potential of 0.75 V at 50µA and 0.70 V at 25µA for modified and carbon electrodes respectively. The modified electrodes are suitable for the development of high-efficiency bio/electrochemical systems.

Keywords: Bio-nanocomposite, Electrode, Green energy, Green synthesis, Microbial fuel cell

Nanocomposites are high-end material that demonstrates unusual combinations of qualities and provides new potential for the development of materials. Nanocomposite materials are in great demand because to their enticing potential, which includes considerably better mechanical and physical qualities[1-2]. Nanocomposites are believed to provide benefits such as module enhancement, flexural strength, temperature heat distortion, barrier characteristics, and other advantages. Their possible application areas range from sensing to biomedical applications[3]. Over the past several years, the term "bio-nanocomposite" has evolved to refer to nanocomposites that comprise a biological component mixed with an inorganic moiety and have at least one dimension in the nanometer range of 1-100 nm[4]. The term "bio-nanocomposites" was coined in 2004, and it is also known as "nanobio-composites" (NCs), "green composites," and "biohybrides."[5]. Bio-nanocomposites and nanocomposites have certain similarities. However, these materials are vastly different in terms of their manufacturing processes, characteristics, functions, biodegradability, biocompatibility, and applications[6]. The biological or inorganic components that are present in the composite are responsible for bringing about these dramatic changes in characteristics[7]. The matrix material includes the nanosized reinforcing components in the form of particles, whiskers, fibres, nanotubes, *etc*.

Recently, nanocomposites materials based on biochar and nickel oxide (BC-NiO) nanocomposite have been synthesized by employing the *Acacia mearnsii* plant for energy storage devices. The nanocomposite was produced by pyrolysis at a temperature of 700°C, and it was studied through the use of X-ray diffraction, Raman spectroscopy, and transmission electronic microscopy[8]. Similar research was conducted by utilizing cerium oxide to construct nanocomposites with biochar acting as the basis material. These nanocomposites were then evaluated for their catalytic activity[9]. As a result of their high conductivity, huge surface area, and simplicity of surface modification, porous structured carbon materials have been used as electrodes. The unusual features of these carbon-based materials have aroused considerable attention, but their expensive preparation

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processes and prohibitive costs prevent their widespread application.

The composition and properties of plant biochar (BC) are significantly influenced by the type of biomass used in its synthesis process. It is a solid carbon-rich residue produced through the thermal decomposition of plant biomass under low oxygen conditions[10]. The complex microstructures and networks of highly linked channels in plant compost facilitate effective electrolyte distribution in the electrochemical reaction system. Plant-derived BC has several notable properties, including non-toxicity, heat resistance, and low weight, which style it appropriate for a wider choice of applications. It can be used to remediate heavy metals and in the development of novel battery types of machinery. Additionally, incorporating different modifiers such as metal and metal oxide nanoparticles can enhance the characteristics of BC electrodes. For instance, the efficiency of BC electrodes can be improved by creating nanocomposites with gold nanoparticles (Au-NPs)[11]. Au-NPs exhibit distinct electrical, mechanical, magnetic, and optical properties, which set them apart from their bulk form. These unique features can be effectively combined with the inherent attributes of BC. Such integration allows for the design of an analytical apparatus suitable for monitoring environmental pollutants, catalysis, and various biological applications. The incorporation of nanoparticles elevates the sensor's efficacy and its electrochemical activity.

Understanding the electrochemical behavior is crucial for tailoring the electrode's design to specific applications, such as sensing, catalysis, or energy storage. Moreover, this analysis contributes to the broader field of electrochemical research, providing valuable information for the development of efficient and versatile electrochemical devices based on Au-NPs-coated biochar electrodes. Several studies on the electrochemical behavior of the bio-based modified electrode with Au-NPs in microbial fuel cell (MFC) systems have also been published[12-13]. An MFC is a kind of bio-electrochemical device that employs the biochemical processes of microbes to produce energy. In an MFC, microorganisms consume organic matter and produce electrons, which can be collected by electrodes and used to generate an electrical current. The process of electricity generation in an MFC is driven by the oxidation of organic matter, which releases electrons that flow through an electrical circuit to produce an electrical current. MFCs have been explored as an auspicious technology for the production of renewable energy and the treatment of wastewater. They have the potential to produce electricity from a broad range of organic waste streams, including agricultural waste, food waste, and wastewater, making them a promising technology for sustainable energy production and waste management. Additionally, MFCs can also play a role in dropping greenhouse gas discharges by capturing carbon dioxide from the atmosphere and using it as a source of energy. The compost-based modified electrode in MFCs has recently become popular due to their bio-electro-catalytic action and power production capability[14-15]. The lacing of Au-NPs led to a substantial enhancement in the reliability and consistency of these electrodes in MFC[16]. There are many reports on MFC processes, and to the best of our understanding, no research on the influence of biochar-based Au-NPs on the behavior of MFC for wastewater have been published. Keeping such considerations in mind, the current research has been segmented into two sections. First, we demonstrated the environmentally friendly synthesis and characterization of an AuNPs-based electrode using bamboo biochar. Second, the above green synthesized AuNPs have been combined on to carbon electrode to determine their influence on the bio-electro-catalytic action in MFC.

In this research, a green and cost-effective process was utilized for the synthesis of gold nanoparticle biochar nanocomposites using bamboo plants. The choice of bamboo as the plant source was based on its eco-friendly properties and low cost. To deduce the properties of the Au-NPs/BC nanocomposite, characterization techniques such as SEM, XRD, and FTIR were employed. A copper electrode was then improved with the Au-NPs/BC nanocomposites in a simple and cost-effective manner, allowing for its potential use in various applications. To analyze the electrochemical behaviors, the MFC system is a potential candidate used in this study. The electrode's electrochemical behavior is evaluated through techniques such as CV. These methods allow for the examination of key electrochemical parameters, including electron transfer kinetics, charge storage capacity, and electrocatalytic activity. The resulting findings could contribute to the development of improved and sustainable methods for the synthesis of nanocomposites and potentially aid in the advancement of fuel cell technology.

Materials and Methods

Materials

The Himedia chemical reagent Company was the source of Auric Chloride, whereas all other chemical compounds and reagents were of analytical quality and used without purification. The electrode was constructed using a 2.0 mm copper wire, a copper disk with an 8.0 mm diameter, a 10 mm diameter, and a 50 mm long PVC tube, with white cement serving as the insulating material. Double-distilled water served as the solvent during the experiments. The plant, *Bambusa bambos*, was obtained from the botanical garden of Ayurvedic College Raipur, Chhattisgarh, India.

Apparatus

The Energy Dispersive X-ray Spectroscopy (EDX) and Scanning Electron Microscopy (SEM) (EVO 15, Carl Zeiss India) were employed for specimen analysis to determine their elemental composition[17]. An image was captured at an optimal setting of 20 kV, utilizing a powdered sample that had been crushed with a pestle and mortar. X-ray Diffraction patterns (XRD) were obtained in the range of 2θ angles at 20-80°C using a PANalytical X'pert Pro diffractometer. CuKα radiation served as the source, operating at 40 kV and 30 mA, with a scan rate of 5 min s^{-1}. Fourier Transform Infrared (FT-IR) spectrum analysis was conducted within the range of 4000-500 cm^{-1} using a Bruker instrument[18]. Electrical conductivity was analyzed using a Digital Multimeter according to Kumar and Jujjavarappu (2023)[19].

Nanocomposite preparation with Gold Nanoparticle biochar (Au-NPs/BC)

Bambusa bambos obtained from the botanical garden of Ayurvedic College in Raipur, Chhattisgarh, India, was utilized to produce biochar (BC), as illustrated in (Fig. 1). The synthesis of the (Au-NPs/BC) nanocomposite involved several steps. Initially, the plant sample (stem) underwent thorough cleaning with sodium hypochlorite and distilled water to eliminate any surface contaminants. Subsequently, the stem was sliced into small 5.0 mm pieces, and 20 g of the plant sample was soaked in an aqueous chloroauric acid ($HAuCl_4$) solution (2.5 mM, 50 mL) for three days. A control was also prepared by immersing 1.0 g of the stems in distilled water[20-21].Following this, the steeped stems were washed, dried, and subjected to thermal decomposition at 350°C for 2 h at 2°C min^{-1} in a muffle furnace to obtain black biochar. The resulting biochar was then finely ground into a powder to create the gold nanoparticle biochar nanocomposite (Au-NPs/BC)[22].

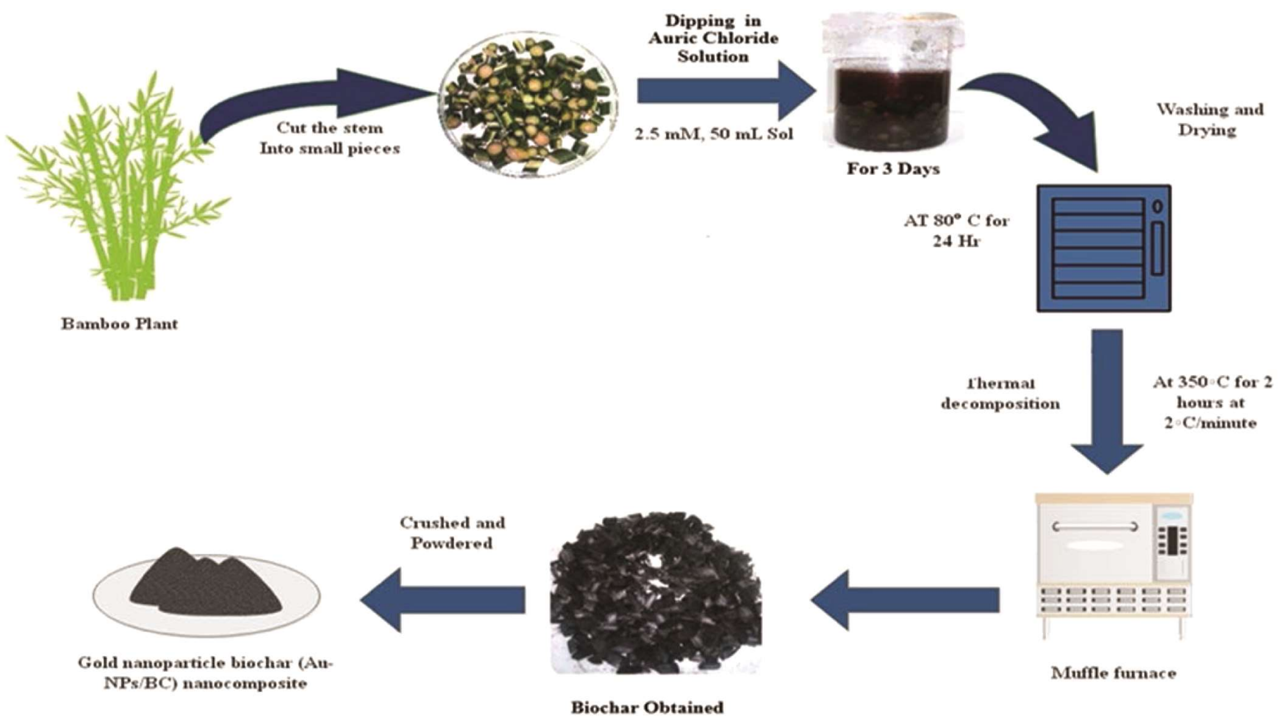

Fig. 1 — Steps Involved in Biochar Synthesis

Modified electrodes preparation from the nanocomposites

Figure 2 illustrates the electrode preparation process, involving the use of a copper plate or disk with a diameter of 8.0 mm and a thickness of 2.0 mm (A). A PVC casing was introduced as support (B), and white cement served as an insulating and supporting material (C). A 2.0 mm gap was maintained above the copper disk, and the resulting electrode had dimensions of 10 mm in diameter and 50 mm in length. For the experimental electrode modification, a powdered nanocomposite (Au-NPs/BC) and normal biochar (Control BC) were utilized. The nanocomposite was blended with a conductive and binding polymer[23]. Gum Arabic, in a ratio of 3:1. This mixture was applied to fill the gap in the prepared electrode, which was subsequently polished and cleaned before coating. Both modified electrodes underwent drying at 80°C in an oven before being employed in subsequent experiments.

Application of the modified electrode in microbial fuel cell

Two plastic containers with a working volume of 600 mL were used to construct the double chamber MFC according to Kumar and Jujjavarappu (2024)[24]. Each chamber had a total volume (v) of 572.55 cm^3, a radius (r) of 4.5 cm, and a height (h) of 9 cm. In the middle of each plastic box, a hole (r =1.5 cm) was drilled for the salt bridge connection. Then, two more holes were drilled in the box's cap to accommodate the insertion of copper wire and electrodes. The anodic chambers then were filled with wastewater as an inoculum and potassium permanganate ($KMnO_4$) as a catholyte in the cathode chamber[25]. The anodic chamber was firmly closed and sealed with glue, to maintain the anaerobic condition. The experiments were done in triplicate; therefore, three MFC setups were prepared.In distilled water, 5% agar and potassium chloride were mixed to make the salt bridge. The solution was boiled for 20 min to allow the salt and agar to dissolve and mix properly. The salt solution waspoured into a falcon tube (r=1.5 cm^2) and allowed to solidify. After that, the prepared salt bridge was implanted into MFC configuration and sealed with elastoseal glue[26].

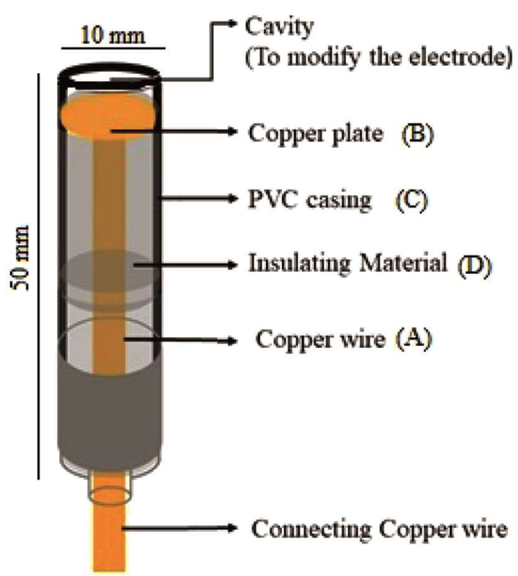

Fig. 2 — Schematic representation of the working electrode (A) Copper wire (B) Copper Plate (C) PVC casing (D) Insulating Material

The electrochemical behavior of the catalyst material used was studied using cyclic voltammograms of gold-nanoparticle coated electrodes. The MFC electrode was electrochemically characterized by cyclic voltametric (CV)by using potentiostat (910 PSTAT mini, Metrohm, India). CV graphs can be used to describe the behavior of cathodic and anodic reactions[27]. CV was employed to define the oxidation–reduction reaction of substrate on the anode biofilm. At a constant scan rate (0.03 V/s) the current response at a surface of the electrode to a particular range of potentials was evaluated. A three-electrode system was used, with the anode serving as the working electrode, the cathode serving as the counter electrode, and an $Ag/AgCl_2$ reference electrode. Peak currents were measured in the -1.5 to +1.5 mV range[28].

Results and Discussion

Characterization of Au-NPs/BC nanocomposites

BC is an attractive option due to its cost-effectiveness, safety, and ease of production, which enables the creation of versatile materials at a low cost as compared to other chemical process materials. Although many applications are still in the early stages, biochar has already demonstrated exceptional performance in numerous applications, including catalysis, soil modification, water purification, and energy and gas storage. One of the latest developments for BC is its application in sensor technology. Furthermore, BC derived from plants and plant-based sources has been employed in a variety of applications.

The proposed mechanism for the phyto-synthesize of the BC-AuNP nanocomposite using bamboo involves a meticulous process. Bamboo undergoes controlled pyrolysis to yield biochar, a process further enhanced through surface activation, providing functional groups for subsequent interactions[29-30]. Bamboo extract, rich in phytochemicals, plays a pivotal role in the reduction of gold ions, orchestrating the formation of AuNPs[31]. The activated biochar surface acts as a stabilizing agent during this process, preventing agglomeration and ensuring the uniform distribution of AuNPs. This intricate dance of reduction and stabilization results in the in-situ growth of the BC-AuNP nanocomposite. Characterization techniques validate its structural properties, setting the stage for potential applications in catalysis, sensing, or biomedical fields. This sustainable and green synthesis approach capitalizes on bamboo's inherent properties, presenting a novel avenue for the development of versatile nanocomposites[32].

Biochar serves as the base material, which can be modified with various materials and chemicals, including gold and silver nanoparticles, affecting the elemental composition of the resulting nanocomposite, as well as its properties. Heat treatment causes changes in biochar's structure and composition. Electrocatalytic properties of Au-NPs/BC-350 nanocomposites obtained from bamboo were analyzed, with fresh bamboo stems used for permeation in the synthesis process. The easy availability and low cost of bamboo make it a suitable plant for the process. Peculation of the $HAuCl_4$ aqueous solution into the plant's interior is aided by duct and sieve tubes naturally present in bamboo stems. inorganic salts and other impurities are removed during soaking due to the presence of hydrogen ions in the $HAuCl_4$ aqueous solution. After the above treatment, thermal decomposition occurs, removing functional groups like CO, CO_2, and H_2O as volatile elements, thus forming BC. This process enhances the porosity of the BC and leaves its carbon structure. Biochar heating causes the decomposition of $HAuCl_4$, which is further accelerated by the reducing environment formed by the BC, resulting in the production of the Au-NPs/BC nanocomposite[33].

SEM and EDX analysis

The structure and morphology of BC-350 as-synthesized were evaluated using SEM (Fig. 3A), revealing visible sieve tubes and ducts that allow entry of metal salts and chemicals into the biochar. The presence of AuNPs in the Au-NPs/BC nanocomposite was analyzed after powdering the sample. SEM images (Fig. 3B) of the Au-NPs/BC-350 show many particles on the BC surface, indicating the successful insertion of nanoparticles by the established method as it was previously utilized by many researchers[34–36]. In this investigation, the SEM images unveiled a porous structure present in biochar samples. Porosity, a commonly observed outcome, results from the release of matter in the form of small volatile molecules, including CO, CO_2, CH_4, and H_2O, during the thermal conversion process. The Figure 3B clearly illustrates the widespread distribution of AgNPs on the biochar's surface. The presence of AuNPs in the sample is further supported by EDX analysis (Table 1), which shows uniform percentage distribution of C, O, K, and Au with

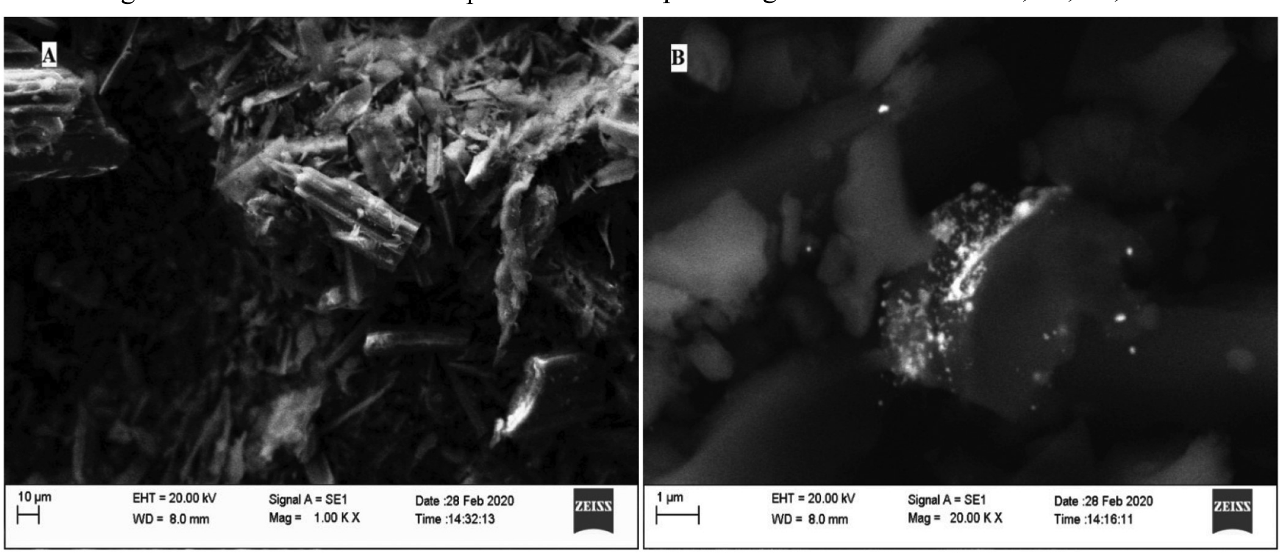

Fig 3 — The image of SEM analysis of (A) BC control; and (B) Au-NPs/BC-350

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	100.00

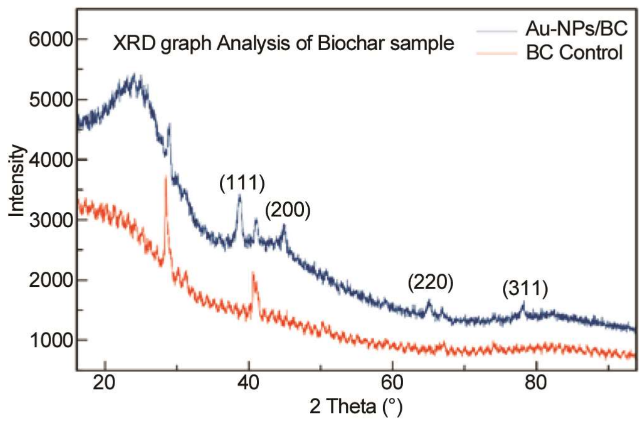

Fig 4 — XRD analysis of the Biochar and AuNP-BC composite

previous findings reported by other researchers[37], providing further validation of the green synthesis of the Au-NPs/BC nanocomposite. The EDX and SEM analysis convincingly demonstrate the fortification of BC with gold nanoparticles and the making of a nanocomposite.

XRD analysis

XRD is a nondestructive technique that furnishes detailed information about the crystallographic structure, chemical composition, and physical properties of a material. XRD analysis can be used to study the microstructure of Au-NPs/BC nanocomposites. The XRD patterns display peaks at 38.72, 44.94, 65.13, and 78.26degree, which correspond to the (111), (200), (220), and (311) standard phases of gold, respectively[38] (Fig. 4). These observations indicate that the green synthesis of AuNP have a high degree of crystallinity. A similar XRD report on AuNPs prepared by a green synthesis method was presented in the research studies. Additionally, in the XRD analysis of the nanocomposite, two prominent diffraction peaks at 22.3 and 43.4 are due to the graphite diffraction pattern (002) and (100), respectively. It is worth noting that the potassium present in bamboo is linked to a self-activation effect that promotes the formation of micro-pores in large quantities.

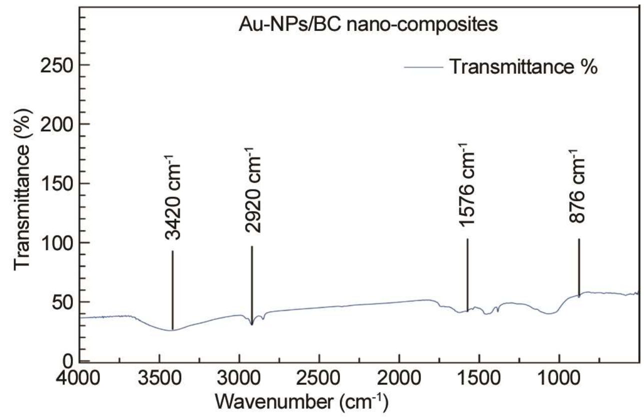

Fig. 5 — Fourier-transform infrared (FTIR) spectroscopy analysis of AuNP/BC nanocomposite

FTIR analysis

Using FTIR spectroscopy, the impact of thermal decomposition on the surface functional groups of biochar was analyzed (Fig. 5). Typically, FTIR offers features from organic functional groups to examine the organic components of biochar[38]. Result showed that the peak at 3420 cm^{-1} is due to organic O-H stretching and may include contributions from any remaining water molecules in the sample or other hydroxyl group-derived minerals. The band at 2920 cm^{-1} is associated with the C≡C alkyne stretching in hemicellulose, while the lignin aromatic group gives rise to asymmetric stretching of C=C at 1576 cm^{-1}, indicating a band of G. As the temperature rises, the C-H bending modes decrease at 876 cm^{-1}, emitting CH_4 gas[39]. The FTIR analysis confirms the presence of all cellulose, hemicellulose, and some lignin content in the biochar nanocomposite.

Electrochemical behavior of Au-NPs/BC nano-composites

To investigate the electrochemical activity of the modified electrode, electrodes modified with nanocomposites were constructed. Both the Au-NPs/BC modified electrode and the control electrode modified with BC were tested for comparative resistance using a digital multimeter. The Au-NPs/BC electrode exhibited lower resistance than the BC modified electrode. The resistance of electrode was less than 5.0Ω, indicating that they were good conductors and therefore good electrodes. This investigation explores the dynamic properties, such as charge transfer processes, conductivity, and redox reactions, exhibited by the nano-composites in the context of electrochemical environments. Understanding the electrochemical behavior is crucial

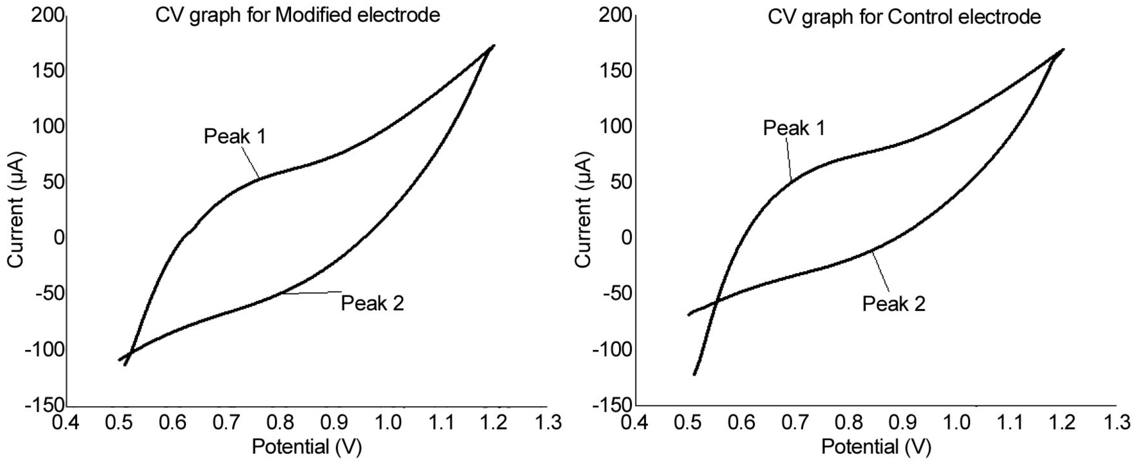

Fig. 6 — Electric current vs potential of CV experiment in microbial fuel cell system (a) CV graph for the modified electrode (b) CV graph for control electrode

for various applications, including sensing, catalysis, and energy storage[40]. The incorporation of gold nanoparticles into the biochar matrix may introduce unique electrochemical features, influencing the overall performance of the nano-composite in electrochemical applications. Analyzing the electrochemical behavior provides valuable insights into the suitability and efficiency of Au-NPs/BC nano-composites for specific electrochemical applications and helps optimize their design for enhanced performance.

Electrochemical behavior of modified electrode in MFC

In this study, CV was utilized to characterize the electrochemical properties of a dual-chamber MFC with modified electrode. Figure 6A & B depicts the basic features of CV utilizing CV plots of responses for a modified electrode in microbial fuel cell. Figure 6A & B showed a voltage sweep and an electric current response for the modified electrode and control electrode in MFC, respectively. Whenever the electro-active species is utilized or respond near to an anode, the electric current appears to rise as it approaches the oxidation state. The potential was scanned in the progressive direction (oxidation), as shown by the structure of the CV graphs, causing the electric current to rise to a maximum. The generation of electric current is potentially subject to two stages in the overall system: (i) the activity of electroactive compound in the surface of electrode, and (ii) efficiency of the electron transfer process[28]. During the working electrode scan, one oxidizing peak (peak 1) and one reducing peak (peak 2) created, as shown in (Fig. 6A & B) for the modified and control electrodes. During the modified anode and control anode electrode scan, the oxidation peak 1 (Fig. 6A & B) was demonstrated at approximately 0.75 V and 0.70 V, respectively. The reduction peak 2 (Fig. 6A & B) was found at lower 0.80 V and 0.85 V, respectively. Such oxidation and reduction peaks have been found in significant potential area, indicating the presence of two unique redox reactions. These redox couples imply that the modified anode electrode is more efficient for oxidation and reduction reaction at a high rate, as well as more efficient to allowing the formation of biofilm, which adds to the high performance of the MFC.

The highest anode potential is directly associated with peak current density values, implying optimal efficiency in the substrate oxidation process within the MFC system under these conditions. This correlation signifies that the substrate undergoes efficient conversion into CO_2, releasing electrons (e^-) and protons (H^+) during the oxidation process. However, it's important to note that the redox potential of the MFC system is susceptible to various factors, including the type and concentration of the substrate, the microbial community composition, anode design and surface area, operational parameters like temperature and pH, the use of electron mediators, and the presence of mass transport limitations[41].

Conclusion

The eco-friendly synthesis of nanoparticles using plant extracts and the utilization of these AuNP across diverse scientific and technological domains represent a burgeoning and promising field of research. Despite recent strides in phytosynthesis, a research challenge arises due to a lack of understanding of the electrochemical properties of phyto-nanoparticles, impeding their purposeful and effective application as modifiers of electrochemical interfaces. The nanocomposites of Au-NPs/BC exhibit several advantageous features, including a high surface area, excellent electrical conductivity, and moderately high porosity. This study focuses on investigating the physical, chemical, and electrochemical properties of AuNPs synthesized through a pyrolysis method involving bamboo plants. The biochar synthesized through the pyrolysis method proves to be more economical and easier to fabricate, although its characteristics depend on the composition of the plant[43]. Moreover, a biochar-based electrode coated with AuNPs demonstrates a promising electrochemical potential, as evidenced by the findings in the MFC study. The incorporation of AuNPs enhances electron transfer and improves electrode conductivity. In future the modified electrode with AuNPs is expected to exhibit superior analytical characteristics in the determination of various biological components in biosensors.

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Conflict of interests

All authors declare no conflict of interest.

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