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Characterizations on The Effect of Iron (III) Nitrate Catalyst on the Synthesis of Reduced Graphene Oxide from Bamboo Derived Graphite

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ABSTRACT

Bamboo-derived graphite was used to make reduced graphene oxide (rGO) from pyrolysis with Iron (III) Nitrate acting as a catalyst for significant success in producing rGO from bamboo in a sustainable and renewable manner. Bamboo grown in Peru, Malaysia (Gigantochloa albociliata) was chosen. The process of producing bamboo charcoal involved pyrolysis at 800°C under an inert nitrogen atmosphere, with a heating rate of 10°C per minute and a soaking time of 3 hours. Bamboo charcoal that was prepared was activated using Iron (III) Nitrate at four different concentrations of the catalyst (0 %, 10 %, 20 % and 30 %) before exfoliation to and reduction to rGO. Characterization of structure and morphology were completed with X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Raman Spectroscopy. Results of this study show that activating bamboo charcoal with Iron (III) Nitrate (20 %) made the best rGO in terms of graphitization, surface roughness, and porosity with maintained structural integrity (fine microstructural detail). This study also shows that rGO produced from bamboo charcoal has many uses such as water purification, energy storage, electromagnetic wave absorption and environmental remediation. This work demonstrates that there continues to be a growing need for affordable and environmentally friendly synthetic graphite as bamboo represents a possible and viable renewable alternative to non-renewable carbon sources for the production of advanced nanomaterials.

Keywords: *Reduced graphene oxide, synthetic graphite, bamboo-derived graphite, Iron (III) nitrate catalyst, pyrolysis, sustainable materials*

1. INTRODUCTION

With the increasing emphasis on moving toward a circular economy worldwide, there has been a strong focus on developing sustainable alternatives to materials traditionally derived from fossil fuels. There has also been a significant increase in the number of energy storage systems, flexible electronic devices, and advanced filtration systems being developed; therefore, there is significantly more scrutiny placed on the carbon footprint of the materials used in the development of each of these technologies. As such, there has been a continued need for renewable and environmentally friendly materials, which has opened many opportunities to explore bamboo as a sustainable carbon precursor for creating advanced nanomaterials via the production of reduced graphene oxide (rGO). Within the regional context of Southeast Asia, and specifically Malaysia, bamboo is an underutilized economic asset. Unlike timber, which requires decades to mature, Gigantochloa albociliata can be harvested within 3 to 5 years without destroying the root system, ensuring a continuous supply of biomass. By upgrading this "poor man's timber" into high-value graphene, we create a new value chain that supports rural economies while providing the high-tech sector with a localized, sustainable supply of raw materials. Bamboo is different from traditional carbon sources because it falls within the Poaceae family. Therefore, bamboo has become a viable renewable option due to its ability to produce biomass quickly and to sequester carbon efficiently. Among the many species of bamboo, the Gigantochloa albociliata species has a high carbon content and grows at an accelerated rate and is an ideal candidate for producing rGO via the conversion of this renewable raw material into rGO through the hydrogen reduction of oxidized graphite (GO). [1]. As interest rises in bamboo- based graphite and its derivatives (e.g., graphene oxide [GO] and reduced graphene oxide [rGO]), converting raw biomass into high quality carbon continues to be technically challenging. The complicated (lignocelluosic) structure of bamboo fibre necessitates more intensive thermal and/or chemical treatment to reach the honecomb lattice structure typically associated with graphene. Presently, issues such as chemical contamination, poor crystallinity, and inconsistent synthesis quality pose barriers to large-scale use. Additionally, conventional methods used to create GO or rGO almost always involve non-renewable graphite sources; therefore, they are unsustainable, energy-intensive to extract from the earth, and subject to the unpredictability of global supply chains. [2].

The strategic importance of developing bio-based carbon has never been more critical. Currently, the global market for synthetic graphite—the primary precursor for

graphene—is heavily reliant on petroleum coke and coal tar pitch. These precursors are not only finite but also produced through high-emission manufacturing processes that contradict the very goal of "green" technology. As international regulations, such as the EU's Carbon Border Adjustment Mechanism (CBAM), begin to penalize high-carbon manufacturing, the transition to biogenic carbon sources like bamboo is moving from an academic interest to an industrial necessity [3].

This research introduces a new catalytic method to overcome the structural and environmental constraints of bamboo charcoal by using Iron (III) nitrate [Fe(NO3)3] as a catalyst in converting bamboo charcoal to enable easier graphitization of the carbon structure at much lower temperatures (under 1000 degrees C) as opposed to graphitizing graphite-filled calciners which reach temperatures over 2500 degrees C. Fe3+ ions facilitate a more orderly rearrangement of the atomic carbon to form ordered structure of the carbon into larger areas which is important in increasing conductivity (due to higher type II conductivity) and surface characteristics for next generation applications [4]. The ordered carbon structures facilitate the production of porous materials with uniform pore structure at an atomic level. Using Iron (III) nitrate, or [Fe(NO3)] is a strategic response to these challenges. The use of an iron-based catalyst does not only lower the temperature but also produces a "graphitization flux." During thermal treatment, the iron nanoparticles function as mobile catalysts, dissolving amorphous carbon and re-precipitating it as highly ordered graphite flakes. The catalytic "dissolution and precipitation" mechanism helps to overcome the disordered nature of lignocellulosic carbon during this process. By refining our precursor material at the molecular level prior to oxidizing the precursor, we are able to create rGO that has superior mechanical and electrical structure.

To carbonize raw bamboo into carbonized material, a process will consist of burning it. Then this will be used in combination with the Modified Hammers Method to produce reduced graphene oxide (rGO) from graphene oxide (GO), and chemically reducing GO for the same purpose. The Modified Hammers Method is recognized as the best method for producing graphene oxide because it can be scaled to large quantities of material, but because of the harsh oxidizing agents used to convert graphite to graphene oxide, this can create a lot of damage to the lattice structure. Additionally, when low-cost biomass-derived carbon is used, it usually contains higher levels of inorganic impurities (i.e., ash content), which exacerbate oxidation-induced damage to the lattice. Therefore, without a catalyst being used to improve conductivity during the reduction of GO to rGO using chemical methods, rGO will have poor electrical properties typically because of having a very high remaining oxygen and significant fragmentation of the rGO structure. [5].

Using Raman spectroscopy to evaluate defects, SEM for morphology, and XRD for characterisation of production materials; we're establishing a method for creating high-quality (pure) rGO from bamboo charcoal at low temperatures. This work also shows a connection between agricultural waste and advanced nanotechnology, in which sustainability and high performance are considered together in materials science. Through connecting the management of agri-waste, the links are starting to be made with advanced nano-technology; therefore, this project could develop a 'standard' protocol for converting bamboo into rGO.. This not only addresses the environmental burden of biomass disposal but also provides a blueprint for the mass production of high-purity carbon materials that can compete with, or even exceed, the performance of traditional fossil-fuel-derived counterparts [6].

2. METHODOLOGY

2.1. Materials

This research used raw biomass of bamboo culms, (species) Gigantochloa albociliata. A species specified for its short growing time and high lignocellulosic density. The samples were deliberately harvested from long-established plantations in Beseri and Padang Besar, Perlis, Malaysia. The selected locations were intended to provide biomass that reflects the respective soil chemistry and climatic conditions of Northern Peninsular Malaysia, both of which will greatly influence the resulting ash-to-carbon precursor ratio. The chemical that has been used to complete the exfoliation process to produce the graphene oxide (GO) is Nitrogen gas (N2), sulphuric acid (H2SO4), potassium persulfate (K2S2O8), phosphorus pentoxide (P4O10), potassium permanganate (KMnO4), hydrogen peroxide (H2O2) and hydrochloric acid (HCl). All of the chemical was strictly used without further purification. For rGO, Sodium Borohydride (NaBH4) has been used as the reducing agent without further purification.

2.2. Materials Preparation

Upon collection, the bamboo culms underwent an initial cleaning process to remove surface debris and organic contaminants. The culms were then mechanically processed and cut into uniform rectangular slices. The goal of maintaining this shape is to maximise the surface area-to-volume ratio, allowing for more efficient water loss during the drying stages. The slices were then air-dried in ambient air for many days, allowing the free water in the bamboo's vascular bundles to evaporate slowly and thus prevent structural warping or cracking of the fibres. The seasoned bamboo was then transferred into a rotary dryer to achieve the bone-dry state to enable the efficient and consistent carbonization of the bamboo. The samples were then dried in an oven at 105 °C for three hours. Maintaining the wood at 105 °C is important because it effectively removes bound water (intercellular moisture) without causing premature thermal degradation of the hemicellulose, which begins at slightly higher temperatures. The rotary dryer used provides a uniform heating of the samples and avoids the occurrence of localized overheating of the biomass. Once all moisture had been removed from the dried bamboo sections, the bamboo was mechanically processed into smaller, uniform chips. This process is critical for the

subsequent catalytic activation and graphitization processes. By ensuring that all bamboo chips are the same size, the heat transfer of the Iron (III) nitrate [Fe(NO3)] catalyst will be consistent throughout the batch, leading to a more uniform reduced graphene oxide (rGO) product.

2.2 Pyrolysis of Bamboo Chips

The bamboo chips were pyrolyzed in a tube furnace with a continuous nitrogen flow, avoiding oxidation. The temperature was raised at a controlled rate of 10 °C/min to a maximum of 800 °C, and held for three hours. The bamboo charcoal was allowed to cool to room temperature under a nitrogen atmosphere and then ground with a mortar and pestle to produce a fine powder, referred to as bamboo-derived graphite.

2.3 Catalyst Activation

Iron (III) nitrate nonahydrate ($Fe(NO_3)_3{\cdot}9H_2O$), a catalyst, activated the bamboo-derived graphite during its preparation for phase separation. Solutions with four Catalyst concentrations (Blank 0% solution, 10% solution, 20% solution, and 30% solution) were prepared by dissolving $Fe(NO_3)_3{\cdot}9H_2O$ in distilled water and stirring for at least 24 hours. The bamboo charcoal powders were mixed with the solutions at room temperature for 48 hours; each sample received equal amounts of $Fe(NO_3)_3{\cdot}9H_2O$ to ensure uniform catalyst distribution. Afterwards, the samples were filtered, oven dried at 100 °C and stored until further fabrication steps were performed. The bulk quantity of charcoal was washed with hydrochloric acid (HCl) to remove $Fe(NO_3)_3{\cdot}9H_2O$.

2.4 Synthesis of Graphene Oxide and Reduced Graphene Oxide

Graphene oxide (GO) was produced from activated bamboo on top of graphite using the modified Hummer's method in a process that started with concentrated sulfuric acid (H2SO4) as the oxidant, added incrementally with potassium permanganate (KMnO4) at controlled temperatures and mixing rates to prevent excessive heating of the mixture materials; once the mixture had been completed, the GO materials were diluted, separated, and washed until neutral pH was reached to produce GO; rGO was produced from GO by chemical reduction using sodium borohydride (NaBH4), which regained a portion of the sp2 hybridization of the carbon in the material improving the electrical conductivity and the graphitic order of the final product; and finally, rGO materials were washed, separated, and dried under vacuum at 60°C. Concentrated sulphuric acid (H2SO4) was used as the oxidative medium, with incremental additions of potassium permanganate (KMnO4) made while controlling temperature and agitation during mixing to avoid excessive heating of the materials. The mixed materials were then diluted, separated, and cleaned until a neutral pH was reached for GO production. The GO material was then chemically reduced with sodium borohydride (NaBH4) to yield reduced graphene oxide (rGO). This reduction process returned some of the sp2 hybridization to the carbon structure, thereby improving the electrical conductivity and graphitic ordering of the final materials. The final rGO materials were cleaned, separated, and dried in a vacuum oven at 60 degrees Celsius.

2.5 Characterization Techniques

The synthesized rGO samples were characterized using various analytical techniques to evaluate their structural, morphological, and chemical properties: X-ray Diffraction (XRD): XRD analysis was conducted to determine the crystallinity, interlayer spacing, and graphitic structure of the rGO samples. Scanning Electron Microscopy (SEM): SEM was employed to observe surface morphology and pore structure. Prior to SEM analysis, the rGO powders were dispersed in deionized (DI) water, drop-cast onto silicon wafers, and dried. Raman Spectroscopy: Raman spectroscopy was used to assess the degree of reduction of GO and to analyze the D, G, and 2D bands, providing insight into structural disorder and graphitization.

For XRD analysis, the powder samples were examined directly without further modification. This systematic approach ensured consistent synthesis and reliable characterization of bamboo-derived rGO, addressing the common limitations of low crystallinity and contamination observed in biomass-based graphene materials.

3. RESULTS AND DISCUSSION

3.1 Analysis of Graphite

3.1.1 XRD Analysis of graphite

X-ray diffraction (XRD) studies confirmed that carbon structures from bamboo-based charcoal were successfully converted into graphite. The sample area's display of distinct graphitic (002) and (100) diffraction peaks indicates that part of the carbon material has been converted to graphite.

Addition of iron (III) nitrate produced remarkable improvements to both the sharpness and intensity of the (002) and (100) peaks that correlate with graphite's crystalline orderliness, resulting in the presence of crystals becoming increasingly well defined. Among the sample areas that received iron (III) nitrate, the 20% concentrations that received iron (III) nitrate exhibited the most distinct and best-defined (002) diffraction peak, suggesting an optimal amount of graphite was produced. This is likely reflective of the alloying actions of the cation (Fe^{3+} ion), which aid and facilitate crystalline formation when amorphous carbon will become crystallized as graphitic structures as a result of the catalytic nature of iron during pyrolysis.

Iron will aid in the dissolution and precipitation of carbon continuously, generating a greater extent of graphitization, with associated improvements in graphite's level of defects [6].

Higher concentrations of catalyst (30 % for example) showed a slightly greater broadening of XRD peaks, indicating possible oversaturation of the catalyst and an increase in the formation of iron oxide particles, which negatively affect the uniform growth of the crystals produced. Moderate catalyst loadings (20%, for example) were found to provide optimal production of well-crystalized graphite with a greater degree of structural integrity than that produced at higher catalyst concentrations. The comparative XRD patterns for the blank, 10 %, 20 %, and 30 % catalyst-treated samples are depicted in Figure 1; showing a significant increase in both the definition of the peaks and the degree of crystallinity observed in the prepared samples when the catalyst was used at optimal concentrations.

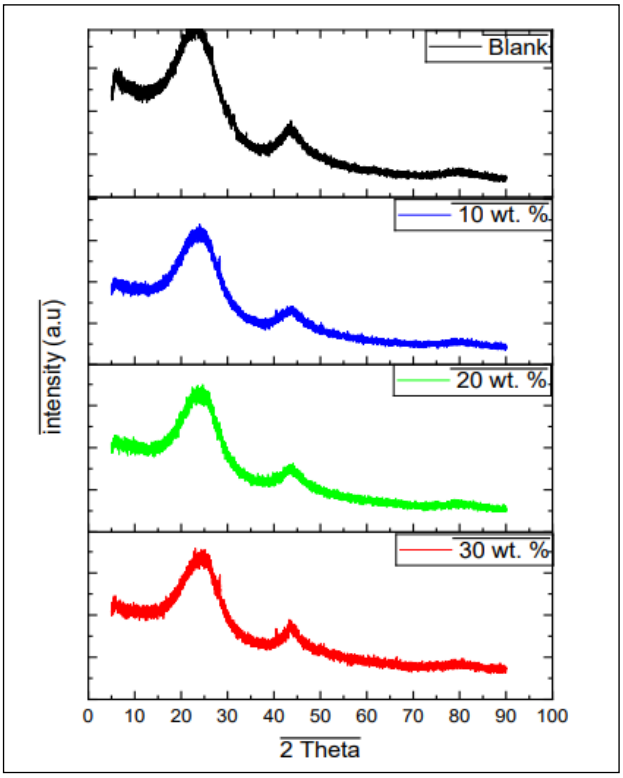

Figure 1. X-ray diffraction patterns of graphite synthesized with Blank,10 wt. %, 20 wt. % and 30 wt. %.

3.2 Analysis of RGO

3.2.1 XRD Analysis of rGO

The structural analysis of iron-doped reduced graphene oxide (rGO) was carried out using X-ray diffraction (XRD). The rGO sample derived from bamboo-based graphite had a sharp 2θ = 26° diffraction peak corresponding to the (002) plane of graphitic carbon, indicating that graphene oxide had been fully reduced and its graphitic structure re-established due to a relatively uniform arrangement of graphene sheets and very few remaining (> 0.1%) oxygen functional group defects within it [7]. The distinctive sharpness and high intensity of the peak indicate the carbon frameworks' high crystallinity and excellent alignment. On the other hand, rGO doped with 20% $Fe(NO_3)_3$ showed a significantly less-sharp, very weak signal peak at the same packing density. Therefore, it is likely that the rGO material has lower crystallinity than that observed in the undoped rGO sample and that the rGO doped with 20% $Fe(NO_3)_3$ has a higher degree of structural disorder. A broader (002) reflection has been observed as a result of Fe species being introduced into the interlayer space of the graphene layers or Fe having been attached to the residual functional groups containing oxygen and having disrupted the regular π–π stacking of the graphene sheets. The structural distortion induced by these two processes hindered the complete realignment of the graphene lattice upon reduction, resulting in smaller graphitic domains with higher defect densities than those in undoped rGO.

The minor peaks observed in the sample doped with Fe correspond to crystalline phases that are likely due to iron, such as Fe_2O_3 or Fe_3O_4, and indicate that the iron catalyst is partially oxidized or not uniformly distributed throughout the graphite structure. Although structural order was lost in the rGO sample, the presence of defects and dopant interactions are known to promote the functionality of rGO material by providing additional potential active sites, enhancing surface area, and improving catalytic and adsorption capabilities. Hence, although the pure rGO is more crystalline than the Fe-doped rGO sample, its higher level of structural disorder increases its potential for functional use. The incorporation of iron into the bamboo-derived rGO through precise control increases its ability to be tuned and, ultimately, would support its application in catalysis, energy storage, and surface-based electrochemical systems. The geometric alterations in the samples resulting from incorporating iron can be identified from X-ray Diffraction (XRD) patterns (see Figure 2) of the blank and 20% iron-doped rGO. Higher levels of graphitization in the precursor do not always yield higher

degrees of crystallinity in the final rGO. Often, more ordered graphite structures can also lead to greater oxidation of the final product due to oxidation-induced defects. This poses an apparent contradiction between the enhanced graphitization of the precursor and the reduced crystallinity of the final rGO. This discrepancy may be attributed to the chemical reactivity of the precursor and oxidizing agents during the synthesis. Graphitization of the Precursor: The addition of iron (III) nitrate (Fe(NO3)) acts as a catalytic agent for the reorganization of amorphous carbon into more organized graphitic domains during the activation process, creating a larger and more well-defined graphite precursor. Increased Reactivity during Oxidation: Structures with high degrees of graphitization experience a greater degree of reactivity when using the modified Hummers' method. The incorporation of iron in the precursor creates a higher surface area and more uniformly sized pores on the precursor, allowing the reactive oxidizers (KMnO4, H2SO4) to penetrate more deeply and effectively between carbon layers. The process of creating defects in a material during the oxidation phase is characterized by a combination of characteristics collectively referred to as the formation laser. Specifically, this factor accounts for the number of O-functional groups the rGO will attach to the carbon lattice due to an efficient oxidation of the Fe-doped precursor compared with the original carbon precursor. Chemical reduction of these O-functional groups creates holes or vacancies in the hexagonal lattice structure of the carbon associated with the rGO, thus leading to the creation of disorder. Furthermore, compared to a blank sample, the rGO produced from using a Fe-catalysed graphite precursor displays a much greater number of structural defects as well as a more disordered Raman spectrum, and consequently, a much larger amount of disorder than the blank sample. In addition, since the blank sample contained less graphitized carbon prior to oxidation, the carbon in this sample underwent a less efficient oxidation-reduction cycle, thereby yielding a more crystalline yet less functionally crystalline rGO. While Iron (III) nitrate effectively enhanced graphitization in the bamboo charcoal by promoting carbon rearrangement, this superior graphitic structure actually facilitated a more thorough and aggressive oxidation during the Hummers' process. The increased density of oxygen-functional groups on the highly graphitized precursor leads to more significant lattice distortion and vacancies upon reduction, which explains the decrease in crystallinity observed in the final rGO product compared to the blank. [10]

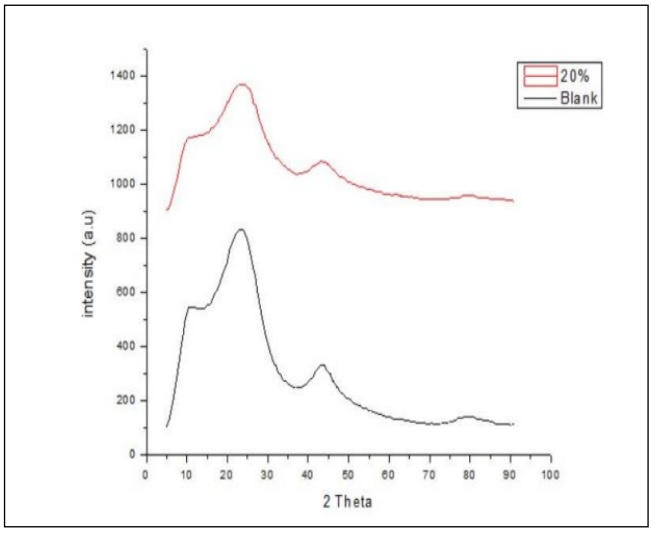

Figure 2. X-ray diffraction (XRD) patterns of (Blank) uncatalyzed rGO & (20 wt. %) catalyzed rGO.

3.3 Surface Morphology by Scanning Electron Microscopes (SEM)

The results of SEM analyses indicated that there are significant differences in the morphology of the blank rGO and the rGO doped with 20% $Fe(NO_3)_3$. Figure 3 illustrates a small number of randomly oriented, graphite-like flakes in the blank rGO, observed at 500× magnification; the layered structure of the blank sample became more apparent at 1000× magnification. The blank sample had smooth, continuous sheets with an intricate pattern of folds and wrinkles (reduced graphene oxide); the presence of wrinkles results from partial elimination of oxygenated functional groups during the reduction process, resulting in contraction of the graphene sheets and accumulation of localised strain resulting in a corrugated surface of rGO, which increases the surface area and flexibility and are characteristics of high-quality rGO. In comparison, the surface morphology of Fe-doped reduced graphene oxide (rGO) (20%) is rougher and more porous. The addition of iron nanoparticles caused a change in the structure, adding nanometer-sized voids, discontinuous folds, and heterogeneities in the layered graphene structure. These nanoparticles likely disrupted the continuous nature of the rGO's graphene lattice structure while increasing the density of defects to the carbon network through localized deformation. The morphological alterations seen indicate that the iron species are functioning as anchor points, which prevents the restacking of the graphene platelets, while creating an open, more porous structure at the same time. The disruptions to the graphene structure will reduce the ability of the delocalised π-electron system to conduct

electricity. However, disrupting the structural integrity of the graphene will lead to higher surface reactivity and increased catalytic properties, due to the additional "rocky" and "porous" surfaces created (to provide more catalytic sites) and the improved interaction of the substrate molecules with the "rough" and "porous" surfaces. Thus, the Fe-doped rGO has desirable properties for use in catalysis, adsorption and electrochemical energy storage [7,9]. Figure 6 shows SEM micrographs (at 500X & 1,000X) of the blank and 20% Fe-doped rGO samples, showing significant surface change due to iron incorporation.

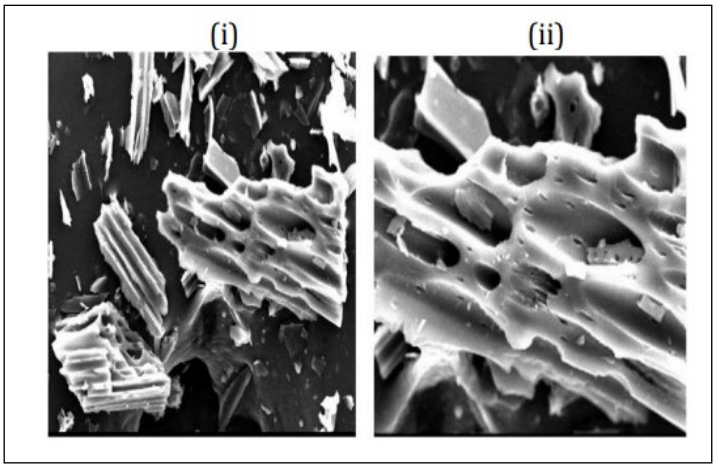

Figure 3. Surface morphologies show the magnification between blank rGO (i) 10µmX500 and (ii) 10µmX1000.

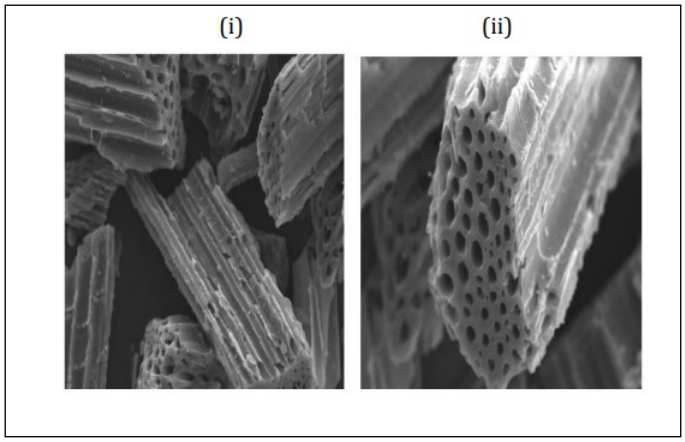

Figure 4. Surface morphologies show the magnification 20% Fe-doped sample (i) 10µmX500 and (ii) 10µmX1000.

3.4 RAMAN Analysis

The results of the Raman spectroscopic analysis of the blank reduced graphene oxide rGO and the 20% Iron (III) Nitrate rGO sample (with catalyst) have been extremely useful for determining the structural properties of the reduced graphene oxide materials. The analysis indicated that both rGO materials possess a graphitic-like structure as evidenced by the presence of the D, G, and 2D peaks (Figures 3 and 4). The D peak at approximately 1335.59 cm^{-1} for the blank sample and 1327.90 cm^{-1} for the Iron sample is indicative of a defect/disorder structure. Additionally, the G peak at approximately 1580.08 cm^{-1} for the blank and 1581.62 cm^{-1} for the Iron confirms the presence of carbon atoms arranged in a 2-dimensional plane in both samples. The presence of a 2D peak is critical for evaluating graphene quality in both rGO materials and was observed at approximately 2700 cm^{-1}. The intensity ratio of the D peak relative to the G peak (I_D/I_G) is slightly higher for the Iron sample (1.07) than the blank sample (1.05), indicating that the addition of iron nanoparticles produces greater defects within the rGO matrix than the blank sample does. The defect density of the rGO-iron composite enhances its catalytic activity and makes it well-suited to catalysis and sensing. The Raman spectrum confirms the presence of vibrational modes associated with Iron (III) Nitrate, demonstrating that the iron nanoparticles have interacted with the graphene matrix of the rGO. The red shift of the D and G bands indicates that graphitization and structural integrity have increased through catalytic activation. Thus, the Raman analysis corroborated the findings obtained from the scanning electron microscopy (SEM) and x-ray diffraction (XRD) studies and supported the conclusion that structural modifications have occurred in the rGO-iron composite and indicate the potential uses of the composite material in advanced applications, including energy storage, catalysis, and sensing, where high levels of conductivity and structural integrity are essential [11].

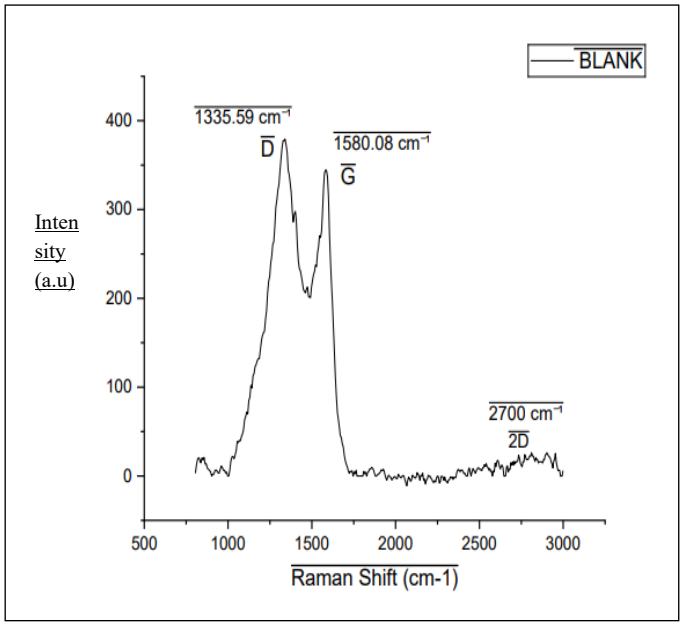

Figure 5. Result of spectrum blank.

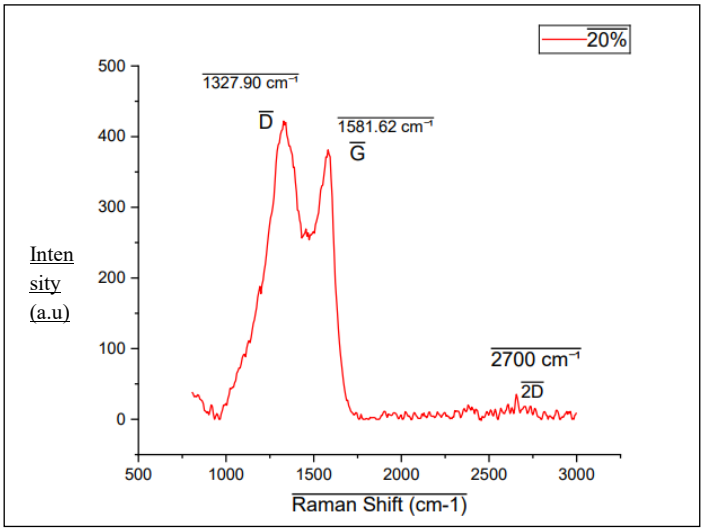

Figure 6. Result of spectrum Iron (III) nitrate.

4. CONCLUSION

In this research, rGO was produced from bamboo at a laboratory scale using iron nitrate as a catalyst. This supports the idea that bamboo is a sustainable source of carbon. The catalyst concentration was systematically varied, with 20% catalyst (20% $Fe(NO3)_3$ producing the optimum structure, which demonstrates the highest level of graphitization and crystallinity as determined by XRD, SEM, and Raman analysis. The Fe nanoparticles catalyze the formation of a graphitic structure while providing useful surface defects and porosity, creating a material with excellent electrical and catalytic characteristics.

These results provide an environmentally friendly method for manufacturing rGO from biomass while reducing reliance on traditional graphite. The bamboo rGO–Fe composite offers significant promise for use in catalytic reactions, energy storage devices, and advanced functional materials. Future work will optimize reaction conditions, explore alternate metal catalysts, and evaluate electrochemical performance to continue advancing the use of biomass-derived nanocarbon materials in large scale industrial applications.

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