Green Synthesis of Reduced Graphene Nanostructure from *Cinnamomum Camphora*

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Abstract: A facile green synthesis for carbon nanoparticle production using *Cinnamomum camphora* (Camphor) is presented. Camphor upon carbonization and chemical oxidation leads to the formation of nano-carbon structures with lateral size 7.33nm to 4.14nm, respectively. The nanomaterial's stacking height is about 2.76nm and 3.10nm, leading to the formation of about 10 layers of carbon. The AFM analysis confirms that the graphene layer formed is wrinkled or folded. Developments of a layered structure with spheroids are observed on the sample's surface, confirming the graphitization of the amorphous carbon. The relative intensity of the defect to the graphite band is found to be 0.98 for the nanostructure indicating a lesser degree of defects. The C1s band of the nanostructure is deconvoluted to components at 284.7, 286.5, 287.3, and 289 eV, which are assigned to non-oxygenated ring carbon (sp² carbon), C in C-O (bound to O either as epoxy or hydroxyl), C in C=O (of alcohols, phenols or ether), and C in C(O)O (carboxylic acid) respectively. The study reveals the formation of few-layer oxygenated carbon layers from the botanical hydrocarbon.

Keywords: green synthesis; carbon nanoparticle; *Cinnamomum camphora*; chemical oxidation

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1. Introduction

*Cinnamomum camphora*, belonging to the plant family Lauraceae [1,2], is generally circulated and developed in southern China as a typical Chinese restorative herb. It is utilized mainly by the Chinese community for medication, including rheumatic joint inflammation, muscular strains, stomach torment, rheumatism, joint twists, respiratory illness, and bronchitis [3]. A few sorts of mixes, for example, characteristic camphor, essential oil, lignans, flavones, and polyphenols, have been recently segregated from *Cinnamomum camphora* [4], and show traces of bioactivities against harmful microscopic organisms [5], poisonous insects [6,7] and human urothelial carcinoma [8]. Plant-based strategies for the reduction of graphene oxide have specific points of interest over microorganism-based techniques. Plant-based synthesis strategies dispense with the requirement for high maintenance cell societies. They can be promptly exploited for large scale manufacturing of graphene [9]. Notwithstanding these focal points, no examinations on screening plants and improvement of response conditions have been accounted for the reduction of graphene oxide utilizing plant extracts.
There is a need for the growth of naturally amicable graphene synthesis procedures that do not utilize much of harmful and toxic chemicals. Recent reports on the reduction of graphene oxide emphasize on the replacement of traditional reducing agents, with melatonin [10], vitamin C [11], and sugar [12] instead of toxic hydrazine. Organic reduction of graphene oxide utilizing microorganisms [13–15] and plant derivatives [16–18] has been proposed as conceivable eco-friendly options compared to completely chemical and physical synthesis strategies.

Graphene is potentially one of the most energizing materials of the decade. A plain sheet of sp2 hybridized carbon atoms is firmly arranged into a honeycomb lattice [19]. In 2004, graphene’s primary disclosure utilizing a scotch tape stripping technique brought a sensational upheaval, particularly in the realm of materials science research and development [20]. Recently, this single carbon sheet has pulled in serious interest among established researchers attributable to the two-dimensional structure of them [21]. It is demonstrated that a perfect graphene sheet is profoundly arranged and shows remarkable practices, including extraordinary surface areas (2630 m² g⁻¹), high Young’s modulus (1.0 TPa), high thermal conductivity (5000 W m⁻¹ K⁻¹), and strong chemical toughness. The two primary steps in the chemical strategy for making graphene are the oxidation of graphite into graphene oxide, trailed by reducing graphene oxide into graphene. The most commonly utilized technique to reduce graphene oxide is the Hummers’ method [22].

Investigating the unique properties of carbon and graphene-based nanodots has attracted tremendous research interest in recent days due to its unique and extravagant properties. They exhibit fascinating properties like stable fluorescence (FL), chemiluminescence (CL), and electro-chemiluminescence (ECL), taking into consideration their size and edge effect [23,24]. Nano-carbon structure has been fabricated from various carbon-based materials like glucose, graphite or graphene oxides, reduced graphene oxide, and carbon fiber by synthesis methods like hydrothermal or electrochemical routes [25,26]. Even though these are facile approaches, the precursors used are relatively costlier. Studies have been reported on the synthesis of carbon-based dots from agricultural waste, hydrocarbon fuel, plant residues, and humic substances, promising agents of the Nanocarbon domain. Synthesis of nano-carbon derivative in a facile way from an environmentally benign way is a topic of interest [27,28]. In the present investigation, few-layer graphene is prepared from botanical hydrocarbon-Camphor (CS), an environmentally benign source of Nanocarbon, considering its ease of availability, cost efficiency as well as a comparative decrease in toxicity of chemicals.

2. Materials and Methods

For the sample preparation, about 2g of camphor soot and sodium nitrate (NaNO₃) was treated with concentrated sulphuric acid (H₂SO₄) in an ice bath. Under continuous stirring, about 6g of KMnO₄ was added slowly. The mixture was then allowed to cool down for 30 minutes. After removing from the ice bath, the solution was stirred continuously for 48 hours using a Teflon coated magnetic stirrer. This was followed by the addition of 184 mL of distilled water, 560 mL of warm water along with 40 mL of hydrogen peroxide one at a time, and the mixture was left undisturbed for 12 hours. The GO particles (CS1) were separated from the solution by centrifugation and washed repeatedly with water and acetone, followed by sonication for 20 minutes.
2.1. Characterization techniques.

The X-ray profiles of the samples were obtained using Bruker AXS D8 Advance X-ray diffractometer. The FTIR spectra were recorded using a Shimadzu FT-IR-8400 spectrometer. Raman measurements were performed at a wavelength of 514.5 nm using the Horiba LABAM-HR spectrometer. The samples' compositional analysis was carried out using an X-ray photoelectron spectroscope (XPS-Omicron ESCA probe). The XPS spectra were deconvoluted by XPS Peak 4.1 program using the Gaussian-Lorentzian mixed function and Shirley background subtraction. The AFM images were obtained using a Nano Surf Easy Scan2 AFM machine. The samples' surface morphology was studied by scanning electron microscopy (SEM-EDX -Joel model JSM-6390 LV).

3. Results and Discussion

The XRD profiles of the CS sample are presented in Fig.1. The sample shows a distinct, highly intense peaks at ~24° and a broadened peak around 42° owing to (002) and (110) planes of graphite [27-31]. Comparing the XRD spectra of the samples with that of pure graphite (which shows only a single prominent peak at ~26°), revealed a broadening of the peak. This is due to the effect of disorder in the sp² hybridized carbon. The sp² sites in the carbon will have both σ and Π states. The medium-range order due to Π-bonding distinguishes disordered carbons from the σ-bonded amorphous carbon. Π -bonding is maximized if the Π states form pairs of aligned, or six-fold aromatic rings or graphitic clusters. This occurs in nano-crystalline graphitic carbon [31,33]. The presence of a strong and broadened Π band in all the samples suggested that the carbon Nanospheres were composed of crystalline graphitic carbon with some defects.

The γ-band at ~2Θ degree originates from disorder in the sp² hybridized carbon and indicates lattice imperfection in the curve graphene sheets, spheres, and tube ends, etc. [32-37]. The comparatively low intensity of the γ-band in the XRD profile of the soot indicates that all the sources have a very low percentage of amorphous carbon. It is also noticed that there is a reduction in lateral dimension (La) with the modified Hummers method treatment (7.33 nm to 4.14 nm for CS1). The stacking height is found to be increased from 2.76 nm to 3.10 nm, indicating 10 layers of carbon.

![Figure 1. XRD analysis of camphor samples exhibiting formation nanostructure.](https://nanobioletters.com/)
3.1. Atomic force microscopy of graphene layers.

A typical AFM image of the obtained graphene is presented in Fig. 2. The cross-section height profile analysis indicates that CS1 samples consist of more than 10 layers.

![AFM analysis of CS1 sample exhibiting sheet-like structure.](image)

**Figure 2.** AFM analysis of CS1 sample exhibiting sheet-like structure.

The height profile confirms that the graphene layer formed is wrinkled or folded. The topography exhibited periodicity, confirming the development of a sheet-like structure. The forward scan fit displayed the height of the synthesized structure in the nanometer range.

3.2. Scanning electron microscopy.

The untreated samples' SEM image appears as agglomerated spheroids with irregularities with their diameter of about 100nm and is presented in Fig.3. With Hummers' treatment, the development of layered structure with spheroids is observed on the surface. In the case of the CS1 sample, the nanosphere is open up, and a layered structure is formed, confirming the amorphous carbon's graphitization.

![SEM-EDS analysis of nanostructure in the CS1 sample.](image)

**Figure 3.** SEM-EDS analysis of nanostructure in the CS1 sample.
EDS analysis was carried out to identify and quantify the constituents of the surface. The analysis reveals the presence of carbon with a little amount of oxygen in the untreated sample. But with the oxidation-reduction process, oxygen groups are incorporated in the microstructure along with elements like Mn, Si, S, and K, possibly from the chemicals used for the Hummers method.

3.3. Raman analysis of graphene.

Raman spectroscopy plays a vital role in the structural elucidation of graphitic materials, particularly graphene, by providing valuable information about its defects, stacking of layers, and crystallite size [37-42]. Raman fingerprints of single, bi- and few-layer graphene are different and investigated by many research groups [42-47]. The peak was observed at 1583 cm\(^{-1}\), due to the G band revealing high-frequency E\(2_g\) first-order mode of graphite-like structure. The 2D band is very weak, indicating the stacking of layers in the nanostructure synthesized. The relative intensity ratio of the defect to the graphitic band is calculated as 0.95 and 0.98 for CS and CS1 samples, respectively, comparable with other reported results on graphene and graphite [47-50].

![Figure 4. Raman analysis of the nanostructure.](https://nanobioletters.com/)

3.4. XPS analysis of graphene.

The C1s XPS spectrum of graphite oxide (In Fig. 5) indicates a considerable degree of oxidation with four components that correspond to carbon atoms in different functional groups; the non-oxygenated ring C, the C in C-O bonds, the carbonyl C, and the carboxylate carbon [33]. The C1 s band of the GO sample (CS1 after Hummers’ treatment) is fitted to four deconvoluted components, located at 284.7, 286.5, 287.3, and 289 eV. These components are assigned to non-oxygenated ring carbon (sp2 carbon), C in C-O (bound to O either as epoxy or hydroxyl), C in C=O (of alcohols, phenols, or ether), and C in C(O)O (carboxylic acid), respectively which are separated by 1.8, 0.8, 1.7 eV respectively. The components that experienced the highest degree of oxidation is the C in COOH and C-O for the samples CS1 (This can be seen from the high-intensity peak around 289 and 286 eV). These X-ray photoelectron spectrometry results agree with IR, Raman, and XRD results, revealing that oxidation has happened to the graphene plane [47-52]. The observation is also consistent with the elemental analysis by EDS.
3.5. FT-IR analysis of the graphene oxide.

To investigate the thermal evolution and interactions of randomly distributed oxygen functional groups located both at the edges and on the basal plane, Fourier-transform infrared (FT-IR) spectroscopy was carried out on soot and carbonaceous products (spectra not shown). It is observed that modified Hummer’s method leads to strong infrared absorbance associated with the C=O/COOH and C-OH regions of multilayered GO with a sharp peak at 3000-3700 cm\(^{-1}\) due to larger hydroxyl, carboxyl, and water contribution [52-55]. The peak at 3431 cm\(^{-1}\) was assigned to the O-H stretching vibrations and at 1047 cm\(^{-1}\) was due to the C-O stretching vibrations. A sharp band is observed at 1500-1600 cm\(^{-1}\), which arises due to the asymmetric stretch of sp\(^2\)- hybridized C=C. Its intensity depends on the environment, being strongest with C=C bonds and is attributed to the skeletal vibrations from graphitic domains, indicating the presence of crystalline graphitic carbon.

4. Conclusions

Several few-layer graphenes consisting of wrinkles and defects are synthesized from camphor, a botanical hydrocarbon. They are characterized using AFM, SEM, XRD, Raman spectroscopy, XPS, and FT-IR. From the AFM analysis, it is confirmed that the graphene layer formed is wrinkled or folded. The topography exhibited periodicity, confirming the development of a sheet-like structure. XRD analysis indicates the stacking of about 9-10 layers. The Raman analysis shows that the defect to graphitic peak intensity is less than 1, indicating that the graphene layer's purity is better than the reported result using the graphite flake as precursors. The FTIR spectra reveal functional groups like carbonyl, carboxyl, and hydroxyl groups in the carbon matrix. The XPS analysis confirms the presence of carboxylic functional groups in the carbon matrix. In terms of the number of layers, crystallite size, and quality,
reduced graphene oxide prepared from the camphor soot seems to be the best, possessing low defect to graphite band ratio and lesser number of layers.

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**Conflicts of Interest**

The authors declare no conflict of interest.

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