

Experimental Support for the Characterization of Carbon Allotropes and Nanocomposite Materials for Easy Analysis

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Abstract

The rapid advancement of nanotechnology necessitates precise and comprehensive characterization of synthesized nanomaterials to correlate their physicochemical properties with functional performance. In the present study, several nanomaterials were synthesized via chemical routes and systematically characterized using a suite of complementary analytical techniques. The structural properties, crystallinity, phase purity, and crystallite size were investigated by Powder X-ray Diffraction (PXRD). The morphological features, particle size, shape, and surface topography were examined by Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDX) and Transmission Electron Microscopy (TEM) / High-Resolution TEM (HRTEM). To determine the 3D surface topography, surface roughness, and precise height profile of the nanomaterials at the nanoscale using Atomic Force Microscopy (AFM). The elemental composition and distribution were confirmed by EDX. Functional groups, chemical bonding, and surface chemistry were analyzed by Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy. Optical properties, including band gap energy, with characteristic D and G bands confirming phase purity and defect density, and the percentage of S and P character of materials, absorption behavior, and surface plasmon resonance were evaluated using UV-Visible Spectroscopy (UV-Vis). Colloidal stability, hydrodynamic size, and surface charge were determined by Dynamic Light Scattering (DLS) measurements. Thermal stability and composition were further assessed by Thermogravimetric Analysis (TGA/DTA). The combined results confirm the successful synthesis of nanoscale materials with controlled size, uniform morphology, and desirable structural and optical characteristics, validating their potential for applications in catalysis, sensing, biomedical, and energy storage devices.

Keywords: Carbon Allotropes; DLS; FTIR; Nanomaterials; SEM; TEM; UV-Vis; XRD

Introduction

The application of a suitable analytical technique to inquire into the internal properties, like structure and elemental composition, of a prepared sample is essential in the field of nanomaterials. Characterization technique is imperative for understanding and investigating features, structure, mechanism, composition etc. of the prepared samples (Hannay, 1967). In the present work, several extensive analytical techniques were used and are discussed here. The structure of the prepared materials was determined by techniques such as powder X-ray diffraction (PXRD), Raman spectroscopy, Nuclear Magnetic Resonance (NMR) spectrometry, Fourier Transform Infrared (FT-IR) spectroscopy, Energy-dispersive X-ray analysis (EDX) and Thermogravimetric analysis (TGA). The dimension and morphology of the prepared samples were analyzed by Scanning Electron microscopy (SEM), Transmission Electron microscopy (TEM), Atomic Force microscopy (AFM) and Field Emission Scanning Electron microscopy (FESEM). Photophysical features of the prepared samples were analyzed by Ultraviolet-visible (UV-Vis) spectroscopy. Gas chromatography (GC) and High Resolution Mass spectrometry (HRMS) were used for the determination of the yield and nature of products of chemical reactions. Composition of materials was also studied by Atomic Absorption spectrometry (AAS). This is the fundamental working principle of several characterization techniques used

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for analysis of carbon allotrope and nanomaterials-based nanocomposites that come under the scope of the various research works. Protocols of the various reactions and studies undertaken have also been described and also depicted in Figure 1.

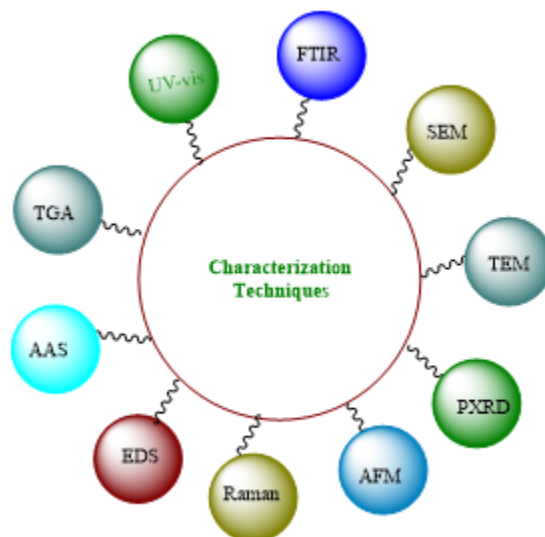


Figure 1: Different Characterization Techniques for Carbon Allotropes and Nanocomposite Materials

Characterization techniques

The following characterization techniques are discussed here in details.

1. Powder X-ray Diffraction (PXRD)
2. Raman Spectroscopy
3. Thermo Gravimetric Analysis (TGA)
4. Fourier Transform Infrared Spectroscopy (FT-IR)
5. Scanning Electron Microscopy (SEM), Field Emission SEM (FESEM)
6. Energy- Dispersive X-ray Analysis (EDX)
7. High Resolution Transmission Electron Microscopy (HRTEM)
8. Ultraviolet-Visible Spectroscopy (UV-Vis)
9. Atomic-force Microscopy (AFM)
10. Nuclear Magnetic Resonance Spectrometry (NMR)
11. Atomic Absorption Spectrometry (AAS)
12. High Resolution Mass Spectrometry (HRMS)
13. Gas Chromatography (GC)

Powder X-ray Diffraction (PXRD)

Powder X-ray Diffraction is a non-destructive analytical technique to examine crystal phase composition and crystallographic structures of prepared materials (Moore & Reynolds, 1997). The basic use of XRD in the present work was to examine the actual phases and crystallographic structures of prepared

compositions, comparing the resulting PXRD pattern with the standard diffraction data in the database of the Joint Committee on Powder Diffraction Standards (JCPDS). In order to do this, solid powder samples were analyzed by monochromatic X-ray from Cu K α radiation and short wavelength, $\lambda = 1.5418 \text{ \AA}$. The scanning rate and 2θ range of the operating system were typically 0.02° per second and 10° to 80° . Sharp peaks indicated that the prepared materials were pure crystallites, while broad peaks implied that the prepared materials had an amorphous structure. JCPDS is a non-profit scientific organization that collects, edit, publish, and distribute powder X-ray diffraction data of several materials with known crystallographic structures. In the present work, powder X-ray diffraction technique was carried out using a Rigaku MiniFlex 600 instrument in most of the cases. This is shown in Figure 2.

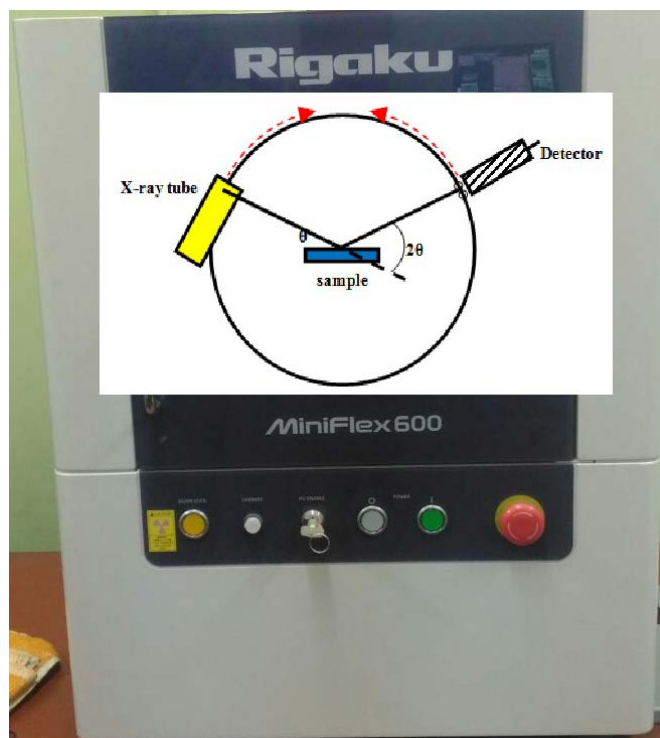
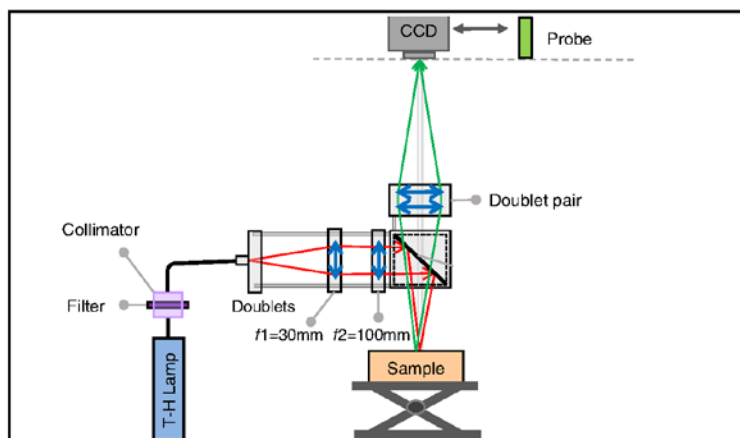


Figure 2: Instrumental Setup of Powder XRD Analysis, Rigaku MiniFlex 600.

Raman Spectroscopy

Raman spectroscopy is a contactless and non-destructive analytical technique to ascertain molecules under investigation (Raman & Krishnan, 1928). It is a very powerful method and is also broadly used to analyze purity and the crystalline nature of carbon-based nanomaterials. Thus, Raman spectroscopy is a very important tool to analyze graphene or graphene oxide, for example, to find out the number of layers of such materials. This procedure yields information about rotational, vibrational and other such transitions in molecules. Raman spectroscopy involves interaction of photon and matter in which the photon is inelastically scattered and excited to higher rotational or vibrational energy levels, the process being known as Raman effect. In 1928, the Raman effect was discovered by C. V. Raman (Ferraro *et al.*, 2003; Smith & Dent, 2019). The main components of commercially available Raman spectrometers are: sample holder, illumination setup, monochromator, scattered light collection system and detection system. In the present work, Raman spectroscopic analysis was carried out using a WITEC CRM200 Raman system with a 532 nm laser (2.33 eV) source and 100x objective lens as shown in Figure 3



<https://www.witec.de>

Figure 3: Instrumental Setup of Raman Spectroscopy, WITEC CRM200 Confocal Raman System

Thermogravimetric Analyses (TGA)

Thermogravimetric analysis was first explored more than 50 years ago. Nowadays, it is a widely used method for studying the nature of materials and their thermal stability. Generally, it is used to find out particulate characteristics of prepared materials that exhibit certain rate of change in the amount of mass loss and/or gain with temperature. This information also reveals any dehydration or oxidation occurring in thermal treatment of the sample under a controlled atmosphere (Li & Rees, 1986). Thermogravimetric analysis is also used to investigate the chemical stability of the prepared materials (Valodkar *et al.*, 2003; Mercuri *et al.*, 2006). In the case of graphene-based nanocomposite materials, TGA is regularly conducted in air as a quantitative method to find out the composition of graphene or graphene oxide and other implanted nanoparticles. In the present work, thermogravimetric analysis was carried out mostly using a NETZSCH TG-209-F3 instrument, as shown in Figure 4.

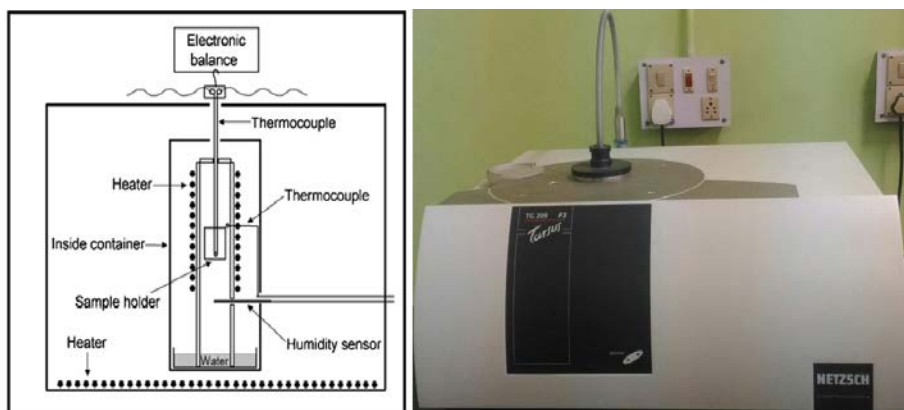
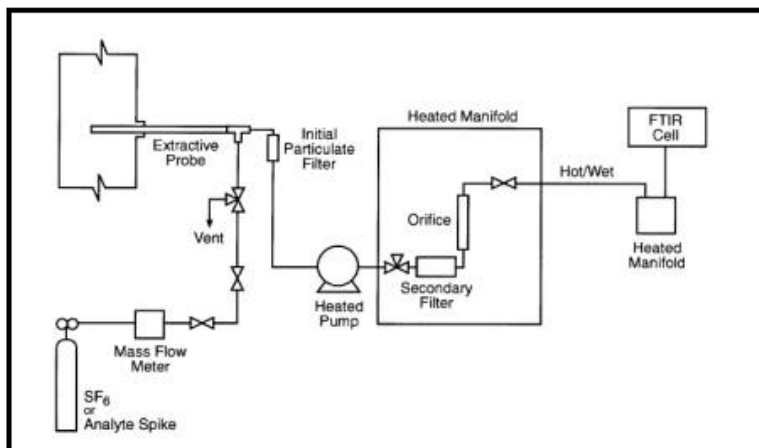


Figure 4: Instrumental Setup of Thermogravimetric Analysis (TGA) NETZSCH TG-209-F3

Fourier Transform Infrared Spectroscopy (FT-IR)

With fast Fourier transform in operation, Cooley and Tukey introduced Fourier Transform Infrared (FT-IR) spectroscopy in 1965. Infrared spectrum of a sample gives information about various vibrations that may occur in the sample. Identifying each vibrational band with standard functional groups provides an idea of the chemical compound or compounds that may exist in the sample. An FTIR spectrometer concurrently collects high-resolution spectral data over a range of wavelengths, typically 400 to 4000 cm^{-1} . This analysis is extensively used in polymer science, organic synthesis, petrochemical engineering, food and

pharmaceutical industries (Sing, 1985). The different phases of the synthesized sample may be completely characterized by this instrument (Skoog *et al.*, 2007; Islam *et al.*, 2011). In the present work, FT-IR spectroscopy was performed with an EPA Method 320 - Vapor phase organic and inorganic emissions by extractive FTIR, as shown in Figure 5.



<https://www.epa.gov> , <https://www.epa.gov>

Figure 5: Instrumental Setup of Fourier Transform Infrared Spectroscopy (FT-IR)

Electron Microscopy (FESEM)

Scanning electron microscope (SEM) usually performed by electron diffraction method to study the morphology of a sample. The sample shape or size of particles and the properties of surface were studied by a high resolution of FE-SEM (field emission scanning electron microscope). It generated high-resolution images of high magnification due to the use of electrons instead of light in electron microscopy (Che *et al.*, 2003). All the tested samples are used in powder from this characterization technique. The beam of electrons is normally scanned in raster scan pattern, electrons beam is focused on the sample by scanning to detect the signal to generate an image of the sample (Miyata & Kuroda, 1999). In my research work, the images were taken using FEI Quanta 400F FESEM, as shown in Figure 6.



Figure 6: Instrumental Setup of Electron Microscopy (FESEM), FEI Quanta 400F FESEM

Energy- Dispersive X-ray Spectroscopy (EDX)

EDX is a nondestructive analytical tool used for the analysis of chemical and elemental composition of a sample. The EDX instrument is comprised of three parts, namely the processing electronics, the detector, and display and MCA. After the beam of electrons hits the sample, the X-rays are collected by the detector.

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Therefore, usually standard electron microscopic instruments (e.g. SEM, TEM) can also perform EDX analysis. Small areas in the sample are scanned by the beam of electrons. In the present work, EDX analysis was carried out using FEI Quanta-400F FESEM instrument as shown in Figure 7

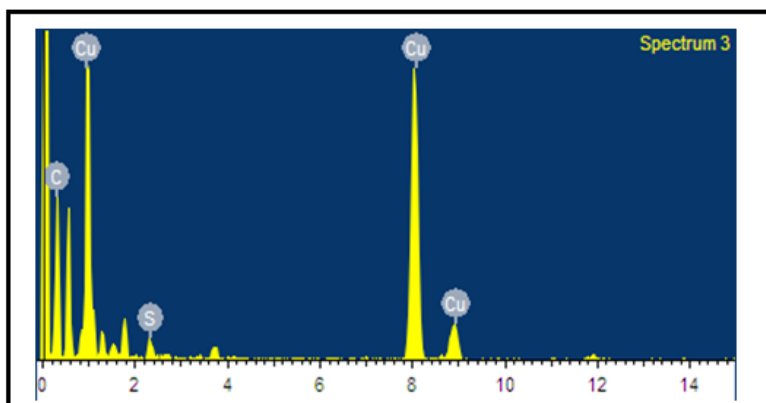
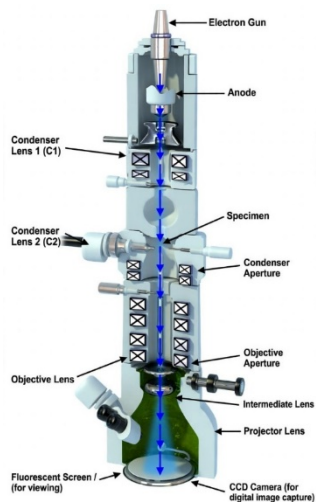


Figure 7: Experimental Data of a Reference Sample in Energy- Dispersive X-ray Spectroscopy (EDX), FEI Quanta-400F FESEM

Transmission Electron Microscopy (TEM)

For material science, Transmission Electron microscopy (TEM) is a very useful technique. The fundamental principles of optical microscopy and TEM are the same. A very high-energy electron beam is used in TEM to generate the specific morphology, crystal structure, shape, size and composition of the prepared sample (Tüysüz *et al.*, 2008). Size distribution of nanoparticles in a sample can be easily carried out using TEM. Selected area electron diffraction (SAED), part of TEM analysis, gives useful information about crystalline or polycrystalline nature of the sample. Very fine crystalline nanostructure and its lattice parameters can also be profoundly determined by high-resolution TEM (HRTEM) analysis (Williams & Carter, 2009; Ernst & Ruhle, 2003). At first, a small amount of the sample is dissolved in ethanol (or any volatile solvent). To homogenize the solution, ultrasonication may be used. After that, a small drop of the solution is placed on a carbon coated copper grid, the solvent evaporated, and the grid mounted in the electron microscopy chamber. In the present work, the nanomaterials were characterized using a JEOL JEM-2010 instrument as shown in Figure 8.

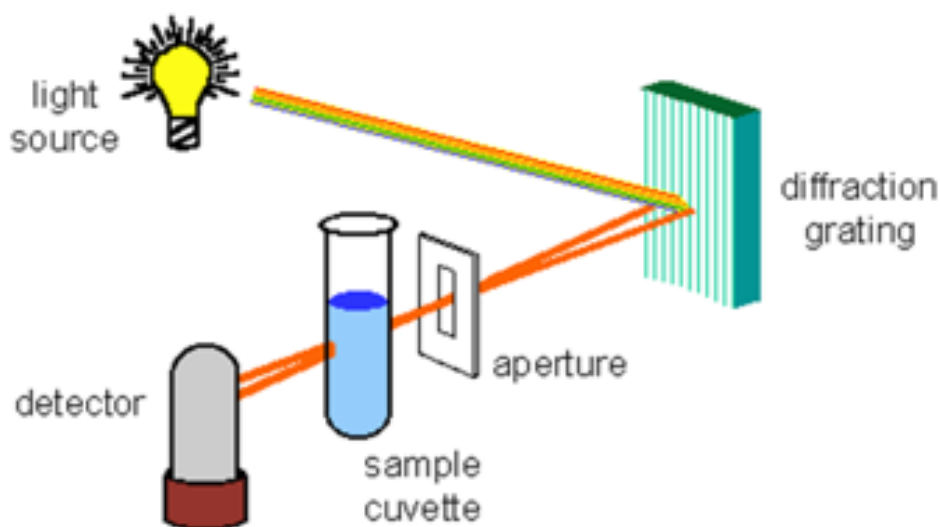


<https://www.jeol.com>

Figure 8: Instrumental Setup of High-Resolution Transmission Electron Microscopy (HRTEM)

Ultraviolet-Visible Spectroscopy (UV-Vis)

Ultraviolet and visible spectrophotometry is the technique related to absorption or reflectance seen in the visible or ultraviolet part of spectral region (200 to 800 nm) at room temperature. Molecules that contain π electrons or non-bonding electrons can absorb ultraviolet or visible electromagnetic radiation. Fundamentally, electrons need to move from a lower energy to a higher energy orbital. Ultraviolet-visible (UV-Vis) spectroscopy is regularly used in the area of analytical chemistry for determining quantity of several analytes. These can be highly conjugated organic compounds, transition metal ions, and biological macromolecules. It can also be used to analyze materials, using reflectivity measurements by the same instrument (Hawkes & Kasper, 1989). The same method of analysis can also be used in gases. The samples are dispersed ultrasonically in deionized water (or some other solvent) and then poured into quartz cuvettes for the measurement (Forker *et al.*, 2012). In the present work, the prepared samples were characterized using a Specord-200 (Analytik Jena, Germany) spectrophotometer as shown in Figure 9.



<https://www.analytik-jena.com>

Figure 9: The Schematic Representation of the Instrumental Setup of UV-Vis Spectroscopy

Atomic-Force Microscopy (AFM)

Atomic Force microscopy (AFM) is a type of scanning probe microscopy (SPM). It can resolve structures of the order of nanometers. This is a thousand times better than the optical diffraction limit. The microscopy related to this method involves quantum mechanical tunneling. Any type of sample, e.g., composite, ceramic, polymer, biological samples or nanoscaled material, can be examined by atomic force microscopy. This technique can operate both in tapping and non-contact modes. Electric potentials are used for detecting surface features by conducting cantilevers that are equipped with silicon tips of high resolution that engage with the surface of the sample that moves along it in different options (modes) and electric potentials make for returning signals. While the force between the cantilever and the sample, AFM can measure the force as a function of the distance between the cantilever and the sample, the force can be measured by AFM as a function of distance between the two. This can be used to provide force spectroscopy. With the help of relevant height differences upon scanning, the thickness and sizes of the particle can be observed and evaluated. In the present work, AFM was used in tapping mode to analyze the structure of graphene, graphene oxide and graphene-based nanocomposites. In this work, the prepared samples were examined using a 2010 NT-MDT NOVA-1.0.26 AFM instrument, as shown in Figure 10.



Figure 10: Instrumental Setup of Atomic Force microscopy (AFM), 2010 NT-MDT NOVA-1.0.26

Nuclear Magnetic Resonance (NMR)

In the 1940s, Felix Bloch and co-workers, and Edward Purcell and coworkers, developed NMR spectroscopy in condensed media and were awarded the Nobel Prize in Physics in 1952 for their work. NMR spectroscopy is mainly used to determine the chemical structure of an organic molecule. It can also make available detailed information about the molecular reaction state of a particular chemical reaction, the nature of reaction dynamics, phase change, conformational change, diffusion and the nature of the chemical environment of molecules in a solvent. NMR spectra are distinctive, can be well-resolved, are analytically easy to handle, and are often highly predictable for small molecules. NMR technique is often enough to confirm the identity of an organic compound. Apparently indistinguishable functional groups present in a molecule can be separated and distinctly identified by NMR. In the present work, prepared samples or reaction products were identified using a BRUKER Ultrashield 400 MHz NMR instrument, shown in Figure 11.



Figure 11: Instrumental Setup of Nuclear Magnetic Resonance (NMR) - Bruker Ultrashield 400 MHz Spectrometer Used in the Present Work.

Atomic Absorption Spectroscopy (AAS)

Atomic Absorption spectroscopy (AAS) is a procedure used in analytical spectrometry for determining quantitative information of chemical elements present in a sample. It is based on the phenomenon of absorption of electromagnetic radiation of a particular wavelength by atoms excited at high temperature in a flame or in graphite furnace. For atomization, flame and electrothermal vaporizers are the most common techniques. But several other methods are also available for specific tasks. It is useful in analyzing trace metals in water from lakes, rivers, and oceans; in pharmaceuticals; in drinking water; in foodstuffs and beverages; in geological and mineralogical samples; in biological fluids; in petroleum products; in forensic analysis; etc. Spectrometry related to atomic absorption was used as the first analytical method and the fundamental principles were worked out in the second half of the nineteenth century by people like Bunsen and Kirchhoff. The method relies on the relationship between spectrophotometric absorption at a specific wavelength and the concentration of an analyte within a sample. The relation is known as the Beer-Lambert law.

In brief, atomic electrons can move to a higher energy orbital by absorbing light of definite wavelength. This wavelength is specific for a definite transition of an electron in a particular atom. The radiation flux in the atomizer is measured with and without a sample using a detector (Walsh, 1955). It shows the ratio between the two absorbance values, from which the concentration of analyte can be obtained from the Beer-Lambert law. In the present work, the prepared samples were analyzed using an AA-240 Agilent Atomic Absorption spectrometer, as shown in Figure 12.



Figure 12: Instrumental setup of Atomic Absorption Spectroscopy (AAS)

High Resolution Mass Spectroscopy (HRMS)

High-resolution mass spectrometry is the basic technique for identifying or confirming the molecular formula of a new compound through accurate measurement of the mass of the molecular ion and various charged fragments obtained by interaction of the molecule with a beam (Biemann, 1990). Mass spectrometers have evolved over the years through many stages. The birth of high-resolution mass spectrometers having double focus was possible due to modified and improved electronics and proper understanding of ion optics during the 1960s. The structural application was thought of mainly for organic compounds, but later it was applied to other fields of chemistry like characterization of organometallic and inorganic compounds.

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Nowadays, it is also used to study much larger compounds such as biopolymers (Burlingame *et al.*, 1996; Bowers *et al.*, 1996). In the present work, the prepared compounds were analyzed using a Waters XEVOG2-S-QTOF mass spectrometer as shown in Figure 13.



Figure 13: Instrumental Setup of High-Resolution Mass Spectrometry (HRMS) - Waters XEVO G2-S QTOF System

Gas Chromatography (GC)

Gas chromatography (GC) is a very common analytical tool, used in analyzing and sorting out various volatile compounds in a reaction mixture. The compounds also have to be thermally stable over the operating temperature range of GC. GC is a specific gas-liquid chromatographic technique, which involves getting a sample vaporized and injected onto the head of a chromatographic column without decomposition. The gaseous mobile phase of GC is usually an inert gas that can transport the sample through the column by its flow. The column is present in the liquid stationary phase of GC, which is an inert solid that adsorbs the gases. GC can be used for testing the purity of a particular substance or to analyze and sort out several compounds in a mixture. It also determines the relative amounts of such components in the mixture.

In gas chromatography, normally an inert (e.g., helium) or unreactive (e.g., nitrogen) gas acts as the mobile or carrier phase. The stationary phase comprises a microscopic layer of liquid or polymer that is on a solid (inert) support present inside a block of glass tube known as a column. The injected mixture of compounds vaporizes and then comes in contact with various stationary phases present in column walls. The scope of interaction between the column and injected compounds is not the same for each compound. Hence, every single compound elutes at different times, known to be the retention time of the compound. Comparison of these retention times gives an important analytical value to identify volatile compounds. The essential parts of GC include the mobile phase of a carrier gas that must be inert like nitrogen, an inlet to deliver the sample to a column, the column where separations occur, an oven as a thermostat for the column, a detector to register the presence of a chemical in the column effluent, and a data system to record and display the chromatogram. In the present work, the reaction products were determined or quantified (GC data) using a Varian 430 gas chromatograph equipped with a 30 m CP-SIL8CB capillary column and a flame ionization detector as shown in Figure 14.



Figure 14: Instrumental Setup of Gas Chromatography (GC)- Varian 430, 30m CP-SIL8CB Capillary Column System

Conclusion

The present work provides comprehensive experimental support for the efficient characterization of carbon allotropes and their nanocomposite materials. The integrated application of techniques such as XRD, Raman, FTIR, SEM, TEM, AFM, UV-Vis, and TGA enables a clear, reliable, and easy analysis of structural, morphological, optical, and thermal properties. This multi-technique approach not only confirms the successful synthesis and quality of the materials but also establishes a simplified analytical framework to develop new kinds of carbon allotropes and nanocomposite materials. This work will be highly beneficial for future research and application-oriented development of the advanced research field of graphene-based nanomaterials.

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