

FACILE SYNTHESIS OF CARBON NANOPARTICLES USING AN AQUATIC WEED, EICHHORNIA CRASSIPES

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Abstract

Carbon nanoparticles are widely used as catalysis, sensing to optics, antimicrobial activity, and data storage capacity, thus carbon nanoparticles are synthesized in many different ways. However, limitations in the applications in the biomedical fields have been brought about by issues such as the usage of toxic precursor chemicals, organic solvents, and creation of toxic by-products associated with current synthesis processes. To overcome this, the study related to facile synthesis of carbon nanoparticles using the leaves of an aquatic weed, Eichhornia crassipes was conducted. Eichhornia crassipes is an aquatic plant considered as a weed as they have no potential bioactivity. In this study, a simple burning of the leaves for the formation of biosoot resulted in formation of nanoparticles. The biosoot thus produced were characterized using Dynamic Light Scattering (DLS), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive Spectroscopy (EDS) and X-ray powder Diffraction (XRD). The associated compounds along with the carbon nanoparticles were evaluated using Fourier Transform Infrared (FTIR) Spectroscopy, and Gas Chromatography – Mass Spectroscopy (GC-MS). The DLS study revealed that the particles fall well below the size of 10nm and recorded the second peak in the size range of 200 – 990nm. SEM revealed the size range between 57nm to 194nm and the EDS recorded Carbon as a major material. The 2θ value recorded in XRD corresponds to Carbon and the size range between 40-76nm. FTIR and GC-MS confirmed the presence of very few compounds associated with that of Carbon Nanoparticles. The study revealed the successful facile synthesis of Carbon nanoparticles in the form of Biosoot of the plant Eichhornia crassipes. However they are found to be free of associated chemicals thus their application in pharmaceutical or cosmeceutical industry on its own is not possible. But, their advantage being they may be safe, biocompatible and act as a nanocarrier agent.

Keywords: Biosoot, Eichhornia crassipes, Carbon Nanoparticle, DLS, FESEM, XRD, FT-IR, GC-MS

INTRODUCTION

Nanotechnology has gained significant as an interdisciplinary science and been widely used in variety of industries, including electronics, agriculture, cosmetics, and medicines. They are also used as a biosensor and for bioimaging, In a general sense, it depends on the development and control of materials with nanoscale dimensions by altering the properties of precursors¹. The distinctive physicochemical and biological features of a wide range of inorganic and organic nanostructures, such as their morphological diversity, high surface area to volume ratio, stability, dispersity, and toxicity to microbes or tumour cells, have been studied and employed. Due to these exceptional qualities, numerous applications outside the field of those currently being used still need to be investigated.

One among the element, carbon is most preferable which includes carbon quantum dots, carbon dots, and carbon nanotubes². Carbon nanoparticles are particularly important among many because they are easy to produce and have the most promising applications³. As a result, extensive study has been conducted in recent years on carbon nanoparticles and their capabilities, such as catalysis, sensing to optics, antimicrobial activity, and data storage capacity⁴. They exhibit a wide range of applications including catalysts, lubricant additives, heat transfer nanofluids, electrical and optical device manufacturing, conductive inks, solar energy conversion materials,

biosensors and cancer cell treatments. Furthermore, carbon nanoparticles have the potential to replace more expensive noble nanoparticles like silver and gold^{5,6}. This nanoparticle plays a major role in current electronic circuits because of its low cost. Carbon nanoparticles have piqued scientists' interest as a potential component for future nano-devices⁷.

As one of the most significant types of nanomaterials with a wide range of uses, several carbon-based nanoparticles have been produced in recent years. Nanoparticle production can be accomplished in a variety of ways⁸. The most common ways for producing nanoparticles are chemical procedures. Some chemical procedures, on the other hand, are unable to prevent the usage of harmful substances in the synthesis protocol⁹. Because nanoparticles are commonly used in places where people come in contact with them, there is a rising need to develop environmentally acceptable procedures that do not employ harmful chemicals¹⁰. However, limitations in the applications of these nanomaterials in the field of medicine have been brought about by issues such as the usage of toxic precursor chemicals, organic solvents, and creation of toxic by-products associated with current synthesis processes¹¹. Sustainable production of nanoparticles has been proposed as an alternative to chemical approaches to tackle these problems¹². It has been demonstrated that effective cost and speed of synthesis can replace physical approaches. Resources that are naturally occurring and economically viable have been suggested for the environmentally friendly production of carbon nanoparticles¹³.

To overcome this, the study related to facile synthesis of carbon nanoparticles using the leaves of an aquatic weed, *Eichhornia crassipes* was conducted. *Eichhornia crassipes* is an aquatic plant considered as a weed as they have no potential bioactivity. In this study, a simple open burning of the leaves for the formation of biosoot resulted in formation of nanoparticles. The biosoot thus produced are characterized using Dynamic Light Scattering (DLS), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive Spectroscopy (EDS) and X-ray powder Diffraction (XRD). Fourier Transform Infrared (FTIR) Spectroscopy, and Gas Chromatography – Mass Spectroscopy (GC-MS) studies were also conducted to find out the associated compounds where they may be a better agent which can be utilized by the pharmaceutical and cosmeceutical industries.

2. MATERIALS AND METHODS

2.1 Collection of Leaves (*Eichhornia crassipes*)

The leaves were collected from the Palkeni tank located in Pallavaram, Chennai, India. The leaves were identified and authenticated by Late Prof P. Jayaraman, Plant Anatomy Research Centre (PARC), Tambaram, Chennai, India.

2.2 Preparation of Biosoot

The surface of the collected leaves of *Eichhornia crassipes* were cleaned in tap water, cut into small pieces and kept under shade to air dry. The air dried plants were directly burnt for the formation of soot. The developed soot were collected by deposition of the same on a sterile porcelain tile. The deposited soot were scraped using sterile scalpel and stored in Eppendorf tubes for further characterization. The methodology of collection of biosoot are represented in Fig. 1.



Fig 1: Habitat of *Eichhornia crassipes* – (a) plant habit (b) leaves kept for shade dry (c) soot sample

2.3 Characterization of soot sample (*Eichhornia crassipes*) from leaves:

2.3.1 DLS (Dynamic Light Scattering) Analysis:

The particle size of the biosoot was determined using Dynamic Light Scattering (Model: Nanotracs Wave II, Microtrac, Inc. USA).

2.3.2 FESEM (Field Emission Scanning electron microscopy) and EDS (Energy Dispersive X-ray spectroscopy) Analysis:

The morphological features of the carbon nanoparticles were studied using Field Emission Scanning Electron Microscope (FESEM) (Model: JSM-6610 LV, Jeol Asia PTE Ltd, at CIL, VIT, Vellore, India) and the elemental composition using Energy Dispersive X-ray spectroscopy (EDS).

2.3.3 XRD (X-ray Diffraction) Analysis:

The soot sample of carbon nanoparticles were measured using X-ray diffraction (XRD) utilising a Cu-K radiation source in the scattering range 2θ of 20-90 on an apparatus running at 45 kV and 40 mA of current. By using X-ray diffraction spectroscopy.

2.3.4 FTIR (Fourier Transform Infrared Spectroscopy) Analysis:

The associated organic compounds with that of carbon nanoparticles of biosoot were examined using FTIR spectrometer (Model: Spectrum Two FT-IR/Sp10 Software Perkin Elmer at CIL, VISTAS, Chennai, India). Using the KBr pellet approach, the soot powder was measured using FTIR spectroscopy in the 4000-400 cm^{-1} region.

2.3.5 3 Gas Chromatography – Mass spectrum study (GC-MS) Analysis:

The biosoot were treated with ethanol and the extract was examined using GC-MS (Model: Perkin elmer, at VIT Vellore, India) to find out the associated compounds in the biosoot.

3. RESULTS

3.1 DLS Analysis:

The DLS study revealed that the particles fall well below 10nm and recorded the second peak in the size range of 200 – 990nm. The DLS pattern recorded for the biosoot is presented in Fig.2

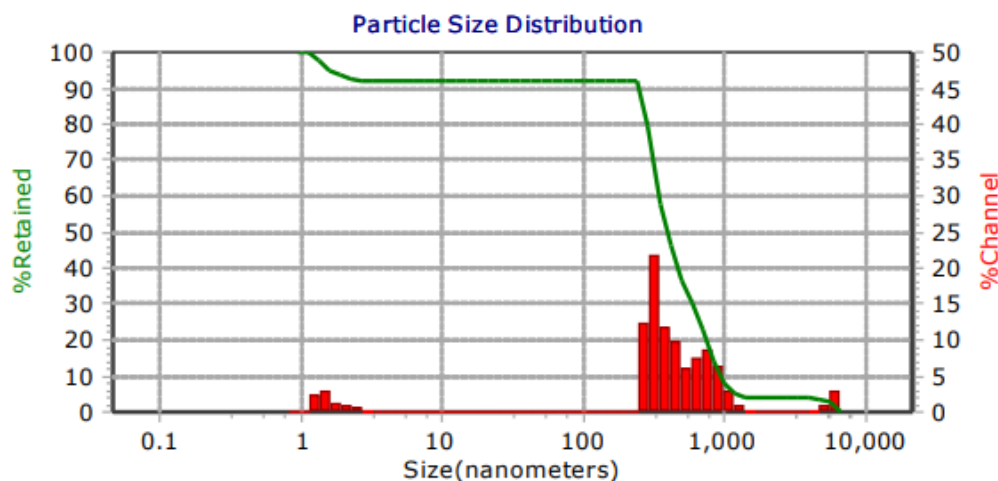


Fig 2: DLS pattern of carbon nanoparticles of the biosoot

3.2 FESEM (Field emission scanning electron microscope) and EDS (Energy Dispersive X-ray spectroscopy):

A scanning electron microscope was used to examine the surface morphology of carbon nanoparticles, size and form. The SEM image of carbon nanoparticles from an *Eichhornia crassipes* leaf is shown in Fig.3. The SEM images depicts a variety of aggregates with inadequate morphologies as well as individual carbon nanoparticles,

majority of which are spherical in shape. SEM revealed the size range between 57nm to 194nm (Fig. 3) and the EDS recorded Carbon as a major material (Fig 4).

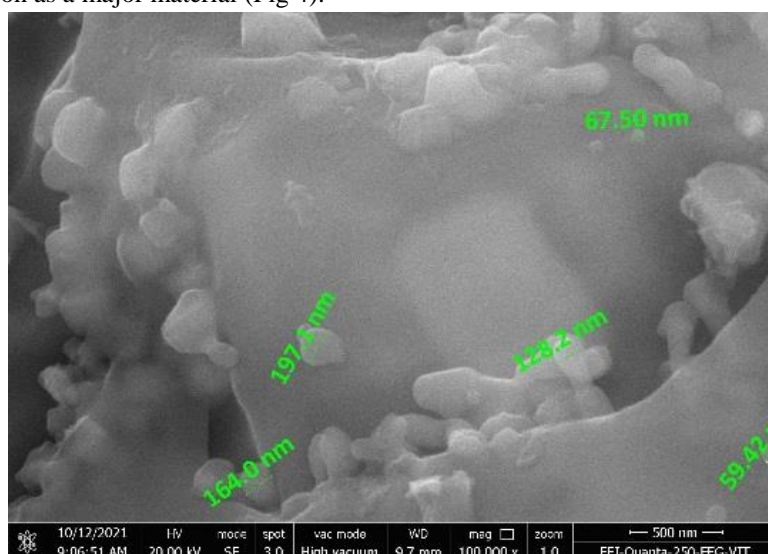


Fig 3: SEM images of carbon nanoparticle of soot sample

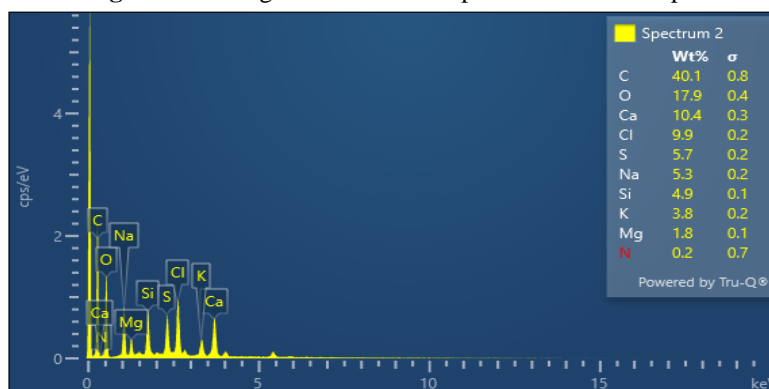


Fig 4: EDS spectrum of the Biosoot of the leaves of *Eichhornia crassipes*

3.3 X-Ray Diffraction Analysis:

The X-Ray Diffraction analysis of the biosoot revealed the crystalline size and structural content of the formed carbon nanoparticles. The diffraction peaks were detected at $m2\theta = 24.7^\circ, 27.1^\circ, 28.7^\circ, 32.0^\circ$ and $40.9^\circ, 50.5^\circ, 58.9^\circ, 66.7^\circ$ and $74.07^\circ, 75.06^\circ$. The 2θ value recorded in XRD corresponds to Carbon and the size range between 40-76nm. The XRD spectrum of the biosoot is presented in Fig 5.

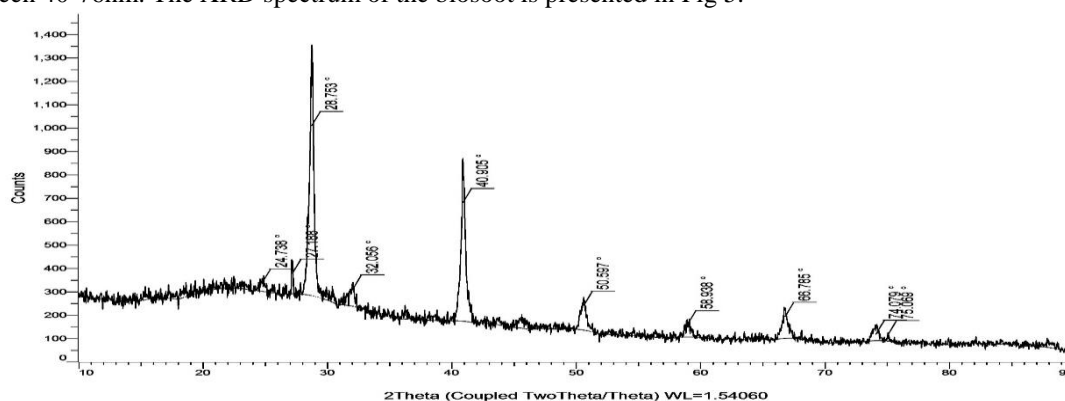


Fig 5: XRD pattern of carbon nanoparticles

3.4 FTIR Analysis:

The FTIR spectrum of Carbon Nanoparticles is shown in Fig 6. The IR spectrum of carbon nanoparticles shows few spectral peak at the band of 3333.88cm^{-1} , 2915.26cm^{-1} , 1952.53cm^{-1} , 1805cm^{-1} , 1557.14cm^{-1} corresponds to O-H stretching alcohols, C=O stretching carboxylic acids, C-H stretching alkyl and aryl halides and C=C stretching alkenes, NO_2 stretching nitrocompounds (Table 1). FTIR spectrum of carbon nanoparticles suggested that nanoparticles are mostly free of associated organic compounds.

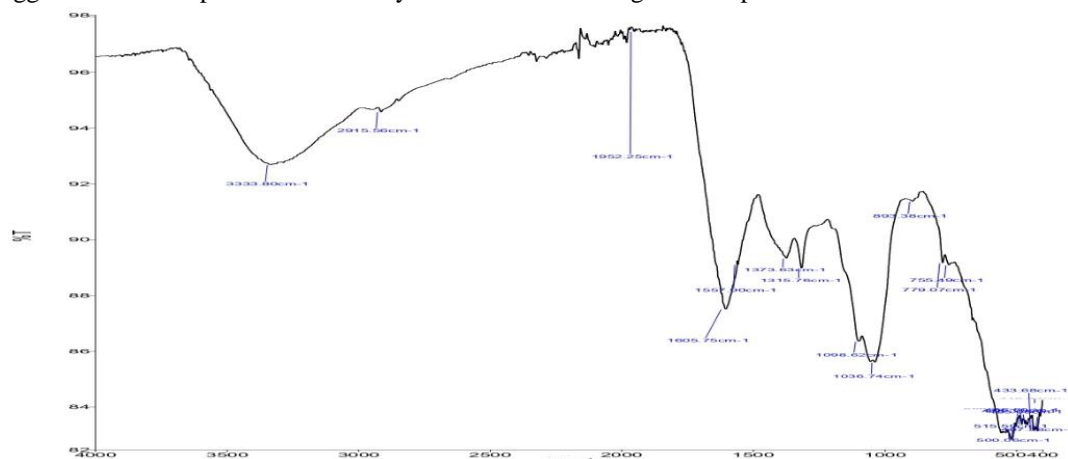


Fig 6: FT-IR spectrum of the Biosoot of *Eichhornia crassipes*

Table 1: List of functional groups recorded through FTIR spectrum for the carbonnanoparticles of *Eichhornia crassipes*

Origin/ Vibration	Exp. (cm^{-1})	Frequency number(cm^{-1})	Intensity	Assignment/Functional group
O-H stretch	3333.29	3550-3200	Medium to strong	Hydrogen bonded (alcohols) NH stretch
C=O stretch	2915.50	3200-2500	Weak to medium	carboxylic acids
C-H stretch	1952.84	1790 – 1600	Medium to strong	Alkyl and aryl halides
C=C stretch	1605.75	1600 – 1650	Medium to strong	Alkenes
NO2 stretch	1557.14	1390 – 1300	Weak	Nitro compounds

3.5 GC-MS analysis:

GC-MS Chromatogram of the biosoot sample of *Eichhornia crassipes* showed only weak peaks. The peaks recorded, their Retention time (RT), molecular formula, molecular weight, peak area of the biosoot sample of *Eichhornia crassipes* are presented in Table 2.

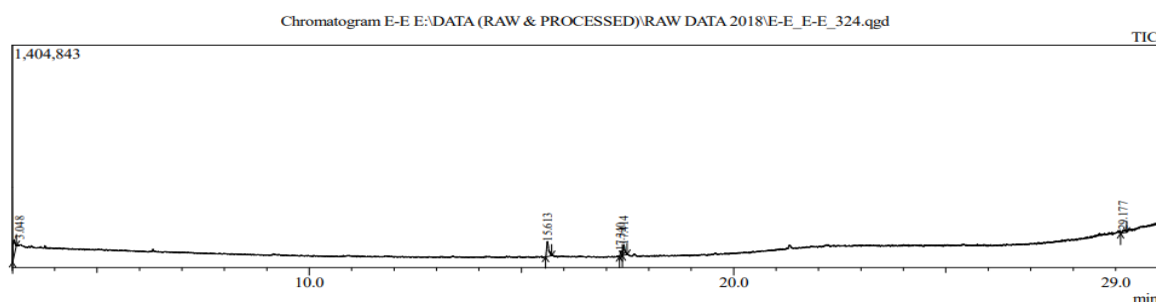


Fig 7: GCMS spectrum of the biosoot of *Eichhornia crassipes*

Table 2: Phytocomponents identified in the biosoot sample of *Eichhornia crassipes* leaves

S .no	Retention time (RT)	Compound	Molecular formula	Molecular weight (MW)	Peak area%
1	3.048	Glycidol	C ₃ H ₆ O ₂	74	32.9
2	15.61	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂	270	31.3
3	17.34	Dec-4-enyl isobutyl carbonate	C ₁₅ H ₂₈ O ₃	248	8.53
4	17.41	8,11,14-Docosatrienoic acid, methyl ester	C ₂₃ H ₄₀ O ₂	348	19.16
5	29.17	4-tert-Butylphenol	C ₁₃ H ₂₂ O _{Si}	222	8.25

4. DISCUSSION

The facile synthesis of carbon nanoparticle has several advantages such as economic, efficient and ecofriendly process. The potentially active phytoconstituents involved in the plant mediated facile synthesis of carbon nanoparticle are biocompatible for a wide range of biomedical applications. When it comes to carbon sources used in the creation of carbon nanotubes and carbon nanospheres, hydrocarbons are by far the most commonly used precursors. Previously the soot of candle wax, diesel and kerosene were studied for their synthesis and characterization¹⁶. The major aim of the study is to develop simple, environmental friendly process for making carbon nanoparticles in the form of biosoot. Previously the carbon nanoparticles were prepared as a soot of natural oils¹⁷. The application of biosoot in absorption of dye materials from of industries were highlighted¹⁸⁻¹⁹. The results, related to the size determination confirms that the biosoot fall well under the category as a carbon nanoparticle. The growth in size of nanoparticle recorded in DLS is attributed to the coalescence and Van der Waal's force exhibited between the particles. It is evident that the synthesized nanoparticles grow in size as a particulate matter (third peak recorded in DLS) and carried to the atmosphere with different sizes of soot. The XRD and SEM confirms the size of biosoot in nanometer range and the EDS confirms the presence of elemental carbon.

The absence of associated materials like aromatic hydrocarbons usually present in diesel, kerosene and hydrocarbon based substances are absent in this study. This was evident in the study related to FTIR and GC-

MS which recorded only limited number of associated chemicals. This is attributed to the source being biological in origin. Biosoot was defined as a soot generated by burning any biological material either they may be of plant or animal origin²⁰. The study aptly demonstrate the usage of biological material in synthesis of carbon nanoparticles. Even the preparation method as just burning is easiest way in synthesis of Carbon nanoparticles. As the carbon nanoparticles prepared from an Aquatic weed, Eichhornia crassipes are free from associated compounds, their application in pharmaceutical and cosmeceutical industry can be potentially exploited as they do not possess any bioactivities on their own. They can also be successfully used as a nanocarrier for biomedical application which is biocompatible. However, further studies related to the toxicity of the synthesized carbon nanoparticle is recommended.

5. CONCLUSION

Eichhornia crassipes showed great capability for the synthesis of Carbon Nanoparticles.

The FESEM studies helps at their morphology and distribution. The EDS patterns confirmed the purity, phase composition and nature of the synthesized nanoparticles. The XRD also supported the biosoot as the carbon nanoparticle. The FTIR and GC-MS revealed that the synthesized carbon nanoparticle in the form of biosoot is free of chemicals. Thus, the biosoot in the form of carbon nanoparticle can be potentially used in the field of medicine and cosmetics. They study confirms that the biosoot in the form of carbon nanoparticle can potentially serve as a nanocarrier.

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