

GREEN SYNTHESIS, CHARACTERIZATION AND APPLICATION OF CARBON BASED NANOMATERIALS

Submitted in partial fulfillment of the requirements for the award of

Master of Science Degree in Physics

By

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DEPARTMENT OF PHYSICS

SCHOOL OF SCIENCE AND HUMANITIES

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

**Accredited with "A" grade by NAAC | 12B Status by
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of S. Vaishali (41590025) who carried out the project entitled "GREEN SYNTHESIS, CHARACTERIZATION AND APPLICATION OF CARBON BASED NANOMATERIALS" under my supervision from November 2022 to May 2023.

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ABSTRACT

Carbon-based nanomaterials play significant role in the development of material science. From the traditional industrial carbon (e.g., activated carbon, carbon black) to new industrial carbon (e.g., carbon fibers, graphite) and new carbon nanomaterials such as graphene and carbon nanotubes (CNTs), fundamental research and applications of carbon based materials are always popular in the fields of chemistry, materials, and other interdisciplines due to their environmental friendliness and various potential application. However, macroscopic carbon material lacks the appropriate band gap, making it difficult to act as an effective fluorescent material. Carbon nanodots (CDs) and Carbon nanospheres are a new rising stars in the carbon family, have attracted considerable attention due to their excellent and tunable photoluminescence (PL), high quantum yield (QY), low toxicity, small size, appreciable biocompatibility and abundant low-cost sources, providing important applications in many fields including biomedicine, catalysis and optoelectronic devices. In the present study, carbon based nanostructured materials was prepared using halimeda opuntia seaweed as precursor through a one- step thermal pyrolysis method, a green route which is environment benign and low cost. The structural and microscopic characterization was done using X-ray diffraction (XRD), Fourier transform Infrared (FTIR) analysis, Raman spectral analysis and high resolution transmission electron microscope (HR TEM). The antibacterial activity of prepared carbon based nanostructured material was studied using the bacterial species, *pseudomonas aeruginosa* and *staphylococcus aureus*.

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LIST OF ABBREVIATIONS

NPs : Nanoparticles

MWCNTs : Multi-walled carbon nanotubes

SWCNTs : Single-walled carbon nanotubes

XRD : X-Ray Diffraction

FTIR : Fourier transform infrared

TEM : Transmission electron microscopy

HR-TEM : High- resolution transmission electron microscopy

CNTs : Carbon Nanotubes

CNS : Carbon Nanospheres

CDs : Carbon Dots

CBNMs : Carbon based Nanomaterials

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CHAPTER-1

INTRODUCTION

1.1 INTRODUCTION TO NANOSCIENCE AND NANOTECHNOLOGY

Nanomaterials are cornerstones of nanoscience and nanotechnology. Nanostructure science and technology is a wide and interdisciplinary as well as interesting area of research and developmental activities that has been growing worldwide in past few years (Alagarasi *et al.* 2011). Nanoparticles can be defined as the objects ranging in the size from 1- 100 nm since their size may differ from bulk materials (Saba Hasan *et al.* 2014).These materials are invisible to the naked eye. These materials can occur as result of product of combustion reactions or it can be obtained naturally. They can have different physical and chemical properties to their bulk-form counterparts. Nanotechnology is a promising field of research which has the potential to offer sustainable solutions to ever pressing challenges confronting our modern livelihood be it intensive agriculture or any health care. Nanotechnology with nanoscale inputs for production of nano agri-inputs (NAIPs) has emerged as an innovative solution for addressing issue of low or declining nutrient use efficiency (NUE) with minimal environment footprint (Yogendra Kumar *et al.* 2021).

Nanomaterials is emerging as new in recent years and creating interest in researchers in the field of nanoscience and nanotechnology and much efforts are being taken on synthesis and applications of various nanomaterials by considering and understanding its potential applications in science and industry . For example, natural materials are replaced by or directly applied on as biocompatible nanomaterials to function or to be with the living system (Ajay Soni *et al.* 2014). The electrical, magnetic, optical and mechanical properties of the nanomaterials have provided many fascinating applications. These properties are fascinating and researchers are still in progress to learn and discover more in this feild .

Nanomaterials is being used in healthcare field, drug delivery and many more . One example of this is nanomaterials being developed for assisting the transportation of

chemotherapy drugs directly into cancerous growths cells, as well as to deliver drugs to areas of arteries that can damage in order to fight cardiovascular diseases.

The Nanoparticles size, their distributions, number of interfaces or graining boundaries, the constituent phases and its chemical compositions, and their interactions are the governing basic factors the unique properties of nanomaterials. These particles occur in nature or structured particles of the 21st century is termed as "magic bullets" because they can be targeted to deliver in a specific manner and thereby have high potential in various applications, including the manufacture of drug, textile and food (Bazila Naseer *et al.* 2018).

In material science, " green " synthesis has become quite popular and gained extensive attention as a reliable, sustainable, and environmental friendly protocol for synthesizing a wide range of materials/ nanomaterials including metal / metal oxides nanomaterials , hybrid materials and bioinspired materials (Jagpreet Singh *et al.* . 2018). Green synthesis or green methods simply meant synthesis of Nanoparticles using plants or plant parts or using their extracts, an alternative to chemical and physical methods. Green synthesis is an emerging area in the combined fields of biotechnology and nanotechnology and delivers lots of economic and environmental benefits. Green synthesis of NPs aims at minimizing waste generation and implementing sustainable processes.

The goal of green synthesis is to reduce the environmental impact of chemical synthesis while maintaining or improving the efficiency of synthesis process. Examples of green synthesis methods include the use of microbes and biocatalysts , plants parts or their extract, photochemical synthesis, and the use of renewable energy sources. The NPs have certain advantages such as it is antioxidant, antibacterial, and antimicrobial characteristics, which make them suitable candidates for application in different biomedical applications (Nehil Shreyash *et al.* . 2021). The synthesis of NPs can be carried out following two different approaches , viz., top-down approach and bottom-up approach.

In the early stages, traditional synthesis methods were utilized and they relied on both carcinogenic chemicals and high energy input for production of nano-sized material. The pollution produced by the traditional synthesis method induces a need for environmentally safer synthesis method that is a method which does not cause hazardous effects. Green methods for nanomaterial synthesis apply natural biological systems to nanomaterial production. Green synthesis is a method just as effective, if not more so, than traditional synthesis; it provides a wider perspective as well as sustainable approach to nanomaterial manufacturing by using naturally sourced starting materials and relying on low energy processes. Thus, the investigation of innovative green synthesis in scientific research and mass production provides a potential solution to limitations of traditional synthesis methods by promoting green processing and low wastage (Matthew Huston *et al.* 2021).

1.2 CARBON BASED NANOMATERIALS

Carbon nanomaterials are a class or type of nanomaterials that are composed of carbon atoms. They showcase unique physical, chemical and electronic properties due to their small size and highly organized structures. Carbon based nanomaterials (CBNMs) have attracted significant interest and curiosity due to the affluent science underpinning their implementations associated with novel mixture of high aspect proportions, greater thermal and electrical performance, outstanding optical features and high interior area. CBNMs not only bear importance in the range of implementations in medication, nano and microelectronics and ecological remedies but may also can be utilized in practical laboratory determinations.

Recent studies and observed investigation display an expansion within the application conducted with CBNMs on wastewater processing analysis and highly positive results are being accumulated (Karamveer Sheoran *et al.* 2022). Carbon-based materials play significant roles in the field of material science and its development. The fundamental research and application of carbon-based materials are popular in chemistry, materials and other interdisciplinary fields like some of the traditional industrial carbon (e.g., activated carbon, carbon black) to new industrial carbon (e.g., carbon

fibers, graphite) and new carbon nanomaterials such as graphene and carbon nanotubes (CNTs), (Junjun Liu *et al.* 2020).

From the early half 19th century, carbon nanotubes (CNTs) and nanostructures were one of the blooming fields and have been in constant limelight since then (Anu.N.Mohan *et al.* 2012). In aerospace, carbon nanotubes has been used in the morphing of aircraft wings. The nanotubes are used in composite form to bend in response to the application of an electric voltage. Carbon nanotubes are the most exciting areas of research in recent decades. Carbon nanotubes are tube shaped material and made up of carbon. The diameter is too small and it is measured by nanoscale. Carbon nanotubes are graphene rolled up into cylinder (Rajwant Kaur *et al.* 2018).

In recent years carbon-Dots (C-Dots) have drawn much attention owing to their remarkable properties such as high compatibility, low toxicity, nano-scale size and ease of modification with good tuneable photoluminescence performance (Nurul Kamilah Khairol Anuar *et al.* 2020). CNDs and CNDs- containing composite materials are attractive potential materials for UV protection (Kyung Woo Kim *et al.* 2021). C dots have drawn tremendous attention because of their potential use in the fields of biology, sensing, catalysis and energy storage and conversion (Ayan Pal *et al.* 2020).

Furthermore, carbon is abundantly available in nature at low cost, thus increasing the potential of CDs to replace semiconductor quantum dots in photonic devices and sensor applications (Manasa Perikala *et al.* 2019). Carbon nanomaterials is one of the most extensively studied materials because of their unique properties spanning from the high specific surface area, high carrier mobility, high electrical conductivity, flexibility and optical transparency fostering their use in sensing applications. (Giorgio *et al.* 2021).

The pairing of pentagonal and heptagonal carbon rings can result in the formation of carbon nanospheres (CNS) and carbon can bond in different ways to create structures with dissimilar properties (Amaya Romero *et al.* 2011). These nanospheres are dispersible in water and constantly show high efficiency to breach nuclear membrane while transporting drug molecules into the cell. Carbon nanospheres show potential

applications in optoelectronics, medicines, gas storage and sensors among all the shapes and sizes (Priya Karna *et al.* 2017). CNS are nanostructure materials whose morphology has attracted interest due to fact they present structural, textural and chemical properties suitable for different environmental and technological applications. CNSs can also exhibit semiconducting properties by inducing photo excitation along with considerable charge transfer ability (Aayush Gupta *et al.* 2019).

1.3 SYNTHESIS OF CARBON BASED NANOMATERIALS

In recent decades, numerous synthetic and traditional approaches for the preparation of C-Dots have been investigated and explored and reported to improve the fluorescent by increasing the quantum yield and narrowing the bandgap hence producing longer wavelengths (Zhang and Yu , 2016). These methods can be largely classified into two main routes: top-down and bottom-up methods (Sagbas and Sahiner,2019). C dots were first discovered and were described as fluorescent carbon impurities at the time of purification of single-walled carbon nanotubes (Xu *et al.* 2004). The synthesis of fluorescent and "carbon dots " (size < 2nm) can be carried out using a combustion oxidation method (Liu *et al.* 2007). Since then several physical and chemical methods have been developed to prepare easy-to-synthesize and cost effective Cdots, starting with different precursors. In general , a top-down approach of synthesis refers to breaking down or decomposition of a larger entity into smaller components with novel characteristics, whereas a bottom-up approach is described as building-up complex system starting with small molecular precursors.

The modern advancement and the uprising of new technology have led to a new era. The nano-revolution opens up with many natural reducing agents based on their environment friendly nanoparticles synthesis protocol for agricultural uses (Acharya *et al.*, 2019; Samy *et al.*, 2019; Malandrakis *et al.*, 2019). With a view to synthesizing biocompatible, ecofriendly nanoparticles for agricultural practices many natural materials have attracted unambiguous attention (Kavitha *et al.*, 2013). While for increasing crop productivity in modern agriculture many advanced technologies are being developed for overcoming the production barriers (Singh *et al.*, 2016); their sustainability for utilization of nanoparticles in crops management are still under debate

(Parveen *et al.*, 2016). A variety of procedures like chemical, physical, biological and hybrid techniques should be chosen by Nanoparticles synthesizing strategies by considering the desired functionality as well as biosafety of the yielded nanomaterials (Parveen *et al.*, 2016). Several positively increased seed germination rates, root and shoot elongation, and plant biomass and plant yield of metal and metal-oxide nanoparticles can be observed within certain concentration levels (Chhipa *et al.*, 2019). It is possible to obtain thin films of various materials, such as ceramics, metal, or semiconductors on the surface of different substrates using chemical vapor deposition (CVD). Pyrolysis of precursor compounds, such as hydrocarbons, occurs on the surface of metal catalysts at high temperatures for the preparation of graphene by this method (Son *et al.*, 2017). For the synthesis of CNTs and fullerenes ablation method was employed (Terrones, 2003 ; De Bonis *et al.*, 2015 ; Sajid *et al.*, 2016).

1.4 APPLICATIONS OF CARBON BASED NANOMATERIALS

Nanoparticles (NPs) are increasingly used to target bacteria as an alternative to antibiotics. Nanotechnology have particularly advantageous in treating bacterial infections. For implantable devices and medicinal materials, the utilization of NPs in antibacterial coatings to prevent infection and promote wound healing, in antibiotic delivery systems to treat disease, in bacterial detection systems to generate microbial diagnostics and in antibacterial vaccines to control bacterial infections are some of the examples (Linlin Wang *et al.* , 2017). It is basically known that carbon-based nanoparticles exhibit high antimicrobial activity as well. Carbon-based nanomaterials such as fullerenes, carbon nanotubes (CNTs) (especially single-walled carbon nanotubes (SWCNTs) and graphene oxide (GO) nanoparticles show potent antimicrobial properties according to some recent reports. The antimicrobial activity of carbon-based nanoparticles together with their mechanism of action has been recently reported (Solmaz Maleki Dizaj *et al.*, 2015). In science and technology CNTs have been applied in many fields.

SWCNTs against both *S. enterica* and *E. coli* which was improved with the increase of nanotube concentrations exhibited antibacterial activities (Gurunathan S *et al.*, 2012). The soccer ball-shaped molecules composed of carbon atoms is called Fullerenes

(Tegos GP *et al.*, 2005). Among three different classes and variety of fullerene compounds (positively charged, neutral, and negatively charged), cationic derivatives shows the maximum antibacterial effect on *E. coli* and *Shewanella oneidensis*; while the anionic derivatives were almost ineffective (Mizuno K *et al.*, 2011). Graphene is a monolayer of carbon atoms which are tightly packed into a two-dimensional crystal (Lu Z *et al.*, 2010).

In 2007, for the first time it was reported that strong antimicrobial activity were showed by single-walled CNTs which caused cell membrane destruction through direct contact, Therefore reducing the cell viability by 80% (Kang *et al.*). due to the unique physicochemical properties and antimicrobial potential of MWCNTs it is widely studied and used in many sectors. To produce effective antimicrobial surfaces, MWCNTs functionalized with various materials are extensively studied and implemented (Rabea EI *et al.*, 2003).

SWCNTs with antimicrobial activity is related to a number of factors. Yang *et al.* also demonstrated that SWCNTs with surface groups of –OH and –COOH shows extremely strong antimicrobial activity to both Gram-positive and Gram-negative bacteria cells (Yang *et al.*, 2009).

Graphene-based nanomaterials is widely analysed for their antibacterial properties. Antibacterial activity of graphene oxide nano sheets against many Gram-positive and Gram-negative bacteria which were made effective and the results were made commercially available (Aunkor *et al.*., 2020).

CHAPTER-2

LITERATURE SURVEY

In 2009, Ray et al. analyzed that the carbon nano particles of 2-6 nm in size with a quantum yield of about ~ 3% were synthesized using nitric acid oxidation of carbon soot and concluded that this approach can be used for milligram-scale.

In 2011, Dieter Bimberg et al. reported that the single quantum dots square facilitating the development of electrically operated emitters of entangled photons or single polarized on demand which acts as an essential component for quantum communication system.

In 2013, Lingling Li et al. analyzed that Graphene based fluorescence material which is one of the effective approach to convert 1 D graphene to 0 D graphene dots for yielding an emerging nano light.

In 2014, Volker Strauss et al. reported the synthesis of mild and controlled conditions, that is , in a microwave reactor. The CND' s thus synthesized exhibit homogeneous and narrowly dispersed optical properties.

In 2017, Xiaofeng Sun et al reported that Carbon Quantum Dots that appeared as attractive Photo luminescent materials which can overcome the poor photo stability materials with respect to poor photo stability of organic dyes and the environmental hazards of heavy metal containing quantum dots .

In 2018, Nagaraj Murugan et al. studied for the first time, a new and green method to synthesis fluorescent carbon dots using a flower obtained from a *Borossus flabellifer* (male tree) by thermal pyrolysis .

In 2019, Mamasa Perikala et al. reported on synthesis of white-light emitting, highly stable carbon dots using a colloidal synthesis technique and marked the role of successful fabrication of highly stable CDs using non-coordinating solvent.

In 2019, Yibo Yan et al. reported that graphene quantum dots that are flat zero dimensional nanomaterials that employed increased interest because of their exceptional chemical and physical properties and possess novel applications in energy storage and conversion.

In 2019, Amra Bratovic et al. studied the classifications and applications of nanomaterials including catalysis, water treatment, sensors storage of energy and nanomedicine.

In 2019, Bui Thi Hoan et al. investigated highly luminescent carbon dots synthesized from lemon juice by one-pot simple hydrothermal method based on different temperatures, aging of precursors, and diluted solvents to control the luminescence of Cdots.

In 2019, Sheena Raina et al. reported for the first time on the synthesis of carbon dots using the acetone leaf extract of cannabis sativa, an abundant weed plant as a sole carbon precursor. The result suggested weed abundance as an excellent natural carbon reserve and the biosynthesized Ag@CDs carry great potential for the antibacterial application.

In 2019, Xiaoyou Xa ,et al. reported the preparation of crude nanotube suspension and the procedures involved.

In 2020, Junjun Liu et al. reported the carbon dots and its properties, because of its diverse physicochemical properties and favorable attributes like good biocompatibility, unique optical properties.

In 2020, Sharmistha Mathur et al. discussed that involvement of green nanotechnology has reduced the use of chemical hazardous fertilizers in crop production and soil development has led to increase uptake of minerals by plants. And analysed the advantages as well limitations of nanofertilizers.

In 2020, Ayan Pal et al. reported the green synthesis of C dots and their photoluminescence properties. Also studied the findings on long range structures in C-dots.

In 2020, Narul Kamilah Khairul Anuar et al. used C dots as a platform for bioimaging, metal ion sensing and as an antibacterial agent, prepared using top bottom or bottom up approach for using biomass waste to produce high quality c dots.

In 2021, Kyung Woo Kim et al. developed a valuable nanomaterials that can block total UV rays using undervalued and profitable marine organism. Successfully developed carbon nanodots (CNDs) and a CNDs- polyvinyl alcohol (PVA) composite film that protect against UV-A, UV-B and UV-C radiation.

In 2021, Giorgio Speranza et al. identified that Carbon enables the development of new biosensors for the detection of biological, inorganic and organic analyte with sensitivity,

low detection and good stability.

In 2021, Nikita rao et al. investigated applications of carbon nanomaterials and properties like catalytic activity and capacitance enhanced through modification. By optimization and controlled structural properties have created multidimensional carbon nanomaterials.

In 2021, Yogendra Kumar et al. showed that Nanotechnology as a promising field with potential to offer sustainable solutions to challenges confronting in agriculture, applications of nano fertilizers, imparting sustainability to crop production.

In 2022, Harun et al. analysed that agro-nanotech innovations offer low input but maximum output based agro farming with effective crop production. Also summarized the current technical innovations and future perspective of nanotechnology as improved soil condition and disease resistance.

CHAPTER-3

AIM AND SCOPE

3.1 AIM OF THE PRESENT INVESTIGATION

- To synthesis Carbon nanostructured materials using *halimeda opuntia* seaweed as precursor.
- To characterize the synthesized Carbon nanostructured materials by various structural and microscopy analysis such as X-Ray diffraction (XRD), Fourier Transform-Infrared (FT-IR), Raman analysis, Transmission Electron Microscopy (TEM)/High Resolution TEM (HRTEM) with Energy Dispersive X-ray analysis (EDAX).

3.2 SCOPE OF THE PRESENT INVESTIGATION

- Carbon nanostructured materials is synthesized via Thermal pyrolysis method, green route which is environment benign and low cost.
- In this method, sea weed (*halimeda opuntia*) was used as carbon source materials for the preparation of Carbon nanostructured materials for the first time.
- The prepared Carbon nanostructured materials was used as antibacterial agent for various Biomedical applications.

CHAPTER-4

EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES

4.1 MATERIALS AND METHODS

4.1.1 Materials

Materials used for synthesis of carbon nanostructured materials are Seaweed (*Halimeda Opuntia*) , Distilled water and Acetone. Seaweed is an algae. It is widespread in all warm water. The polysaccharides which are present in the seaweed extract have been shown to be an excellent source for the preparation of Carbon nanostructured materials, available functional groups and enzymes in the algal cell will be responsible for the formation of Carbon nanostructured materials .

4.1.2 Preparation Of Carbon Nanostructured Materials

Raw seaweed (*Halimeda Opuntia*) was collected, soaked / dipped in castor oil. It was then burned and the carbon materials were collected in powder form. The carbon material was extracted in a petri dish. It was washed with Acetone twice then washed using distilled water. Then centrifuged at 25 °C and finally dried at 100 °C for 24 h to obtain Carbon nanostructured materials. Fig. 4.1 depicts the flow chart for the preparation of Carbon nanostructured materials and Fig 4.2 show the schematic representation for the preparation of Carbon nanostructured materials.

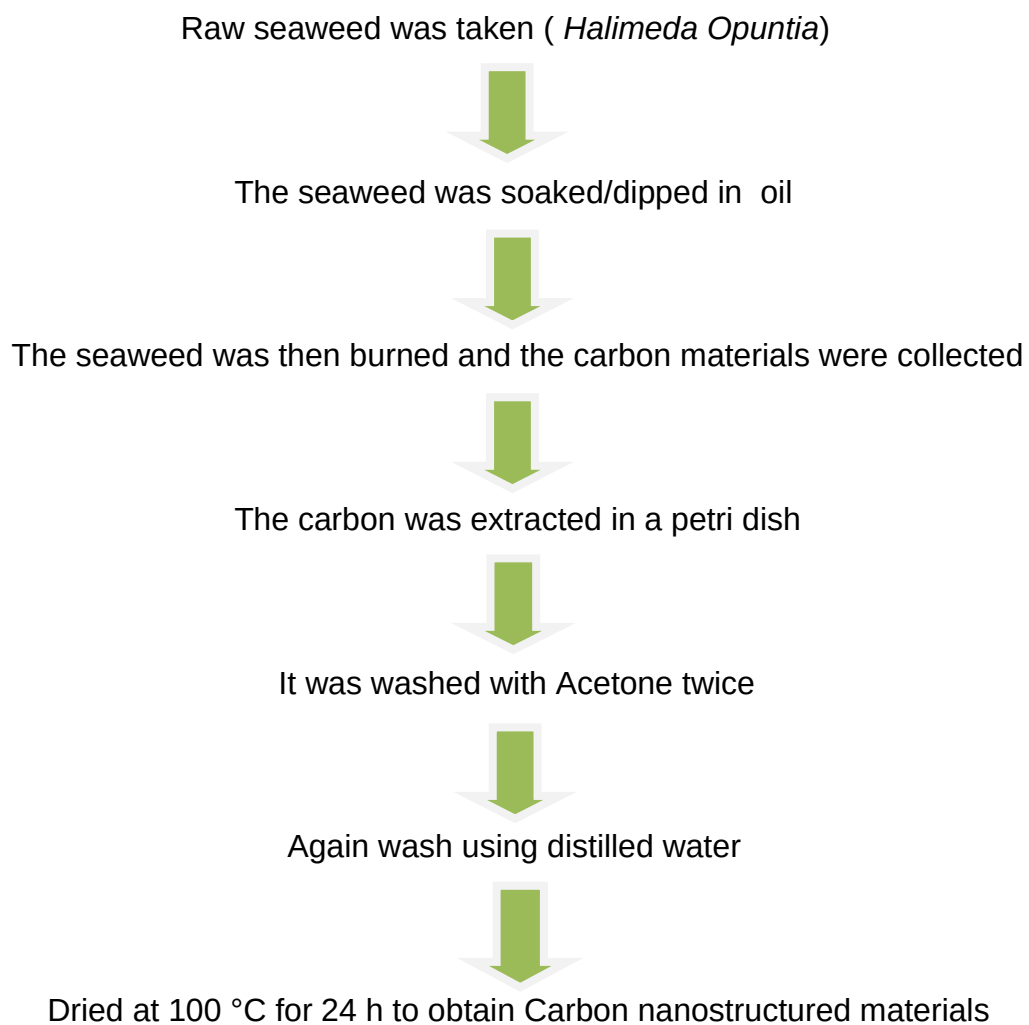


Fig. 4.1 Flow chart for the preparation of Carbon nanostructured materials

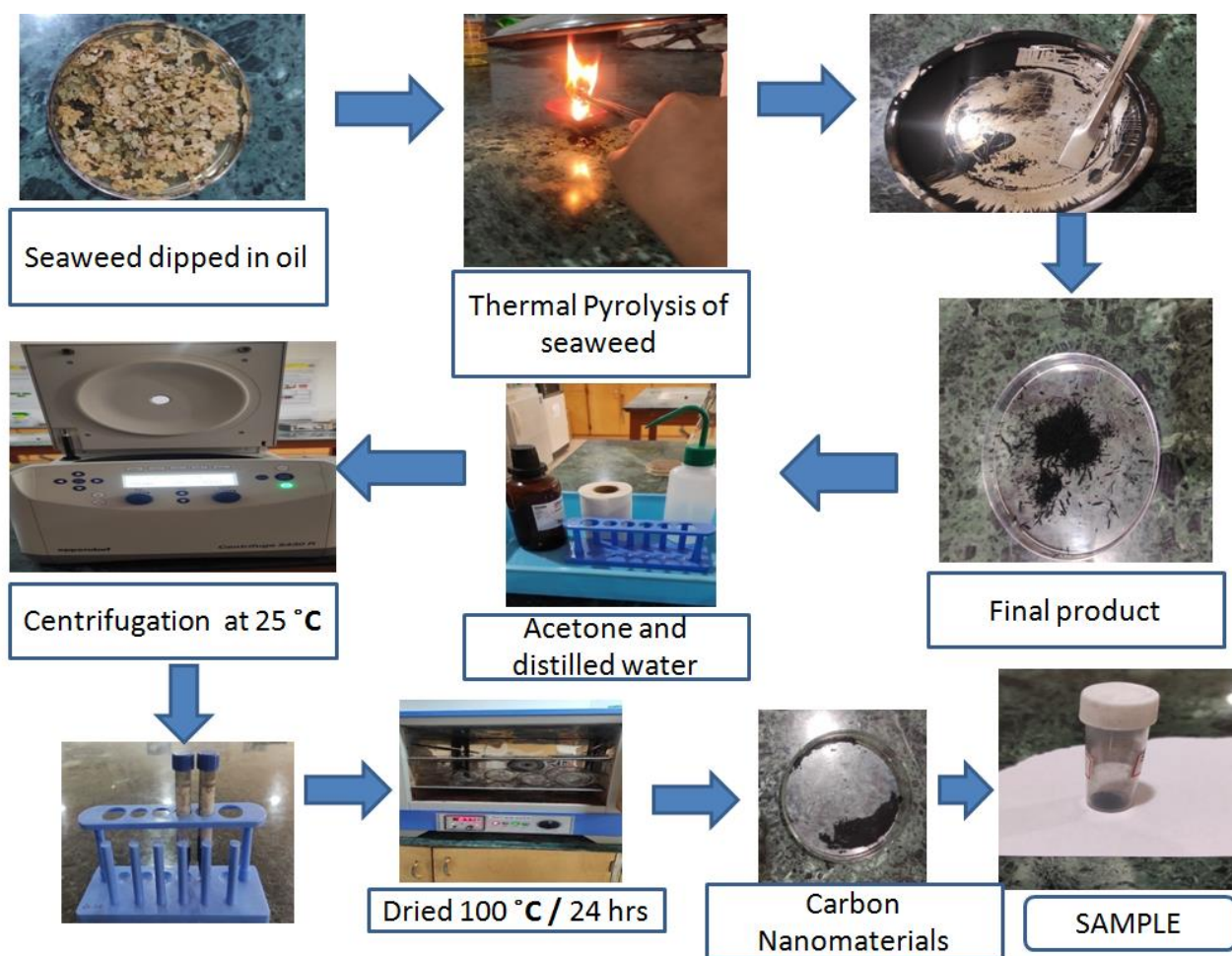


Fig. 4.2 Schematic representation for the preparation of Carbon nanostructured materials

4.2 CHARACTERIZATION TECHNIQUES

4.2.1 X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis is a non-destructive test (NDT) that provides useful information about the structure and crystalline size of synthesized nanoparticles. For characterization of nanoparticles extensive techniques are commonly used (Manoj kumar *et al*, 2022). Both the local and global features can be used to characterize XRD, such as lattice structure with phase morphologies, interlayer distance and so on. A beam (monochromatic X-ray) is focused on a sample material to resolve structural information hidden in the crystal lattice.

XRD follows the principle of constructive interference of X-rays and the sample concerned which should be crystalline. The X-rays generated by the CRT are filtered, collimated and then directed towards the sample. The interaction produces constructive interference based on Bragg's law which relates the diffraction angle and lattice spacing wavelength. X-ray powder diffraction (XRD) is rapid analytical technique primarily can provide information on unit cell dimension and atomic spacing also it is used for phase identification of the crystalline material (fig.4.3).

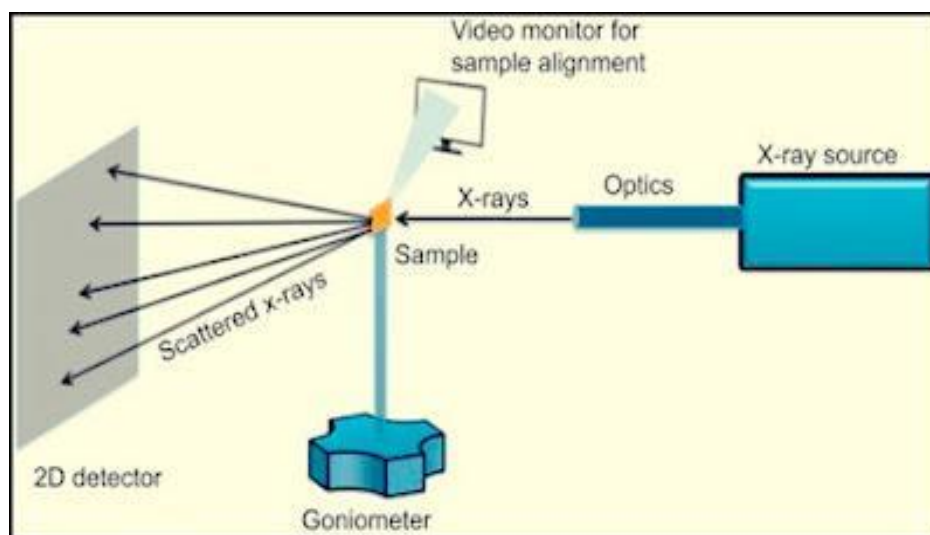


Fig.4.3 Instrumentation of XRD Characterization (Ramani Ummaleti et al.,2020)

The X-ray are generated by cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed towards the sample. The incident monochromatic rays interact and collide with the sample it produces constructive interference (and diffracted ray) when the condition satisfy Bragg's Law $n\lambda = 2d \sin\theta$. This equation can be relates the wavelength (λ) of electro-magnetic radiation to the diffraction angle (θ) and the lattice spacing (d) in a crystalline sample by scanning the sample through arrangement of 2θ angles. From these random orientation of the powdered materials all the possible diffraction directions of the lattice can be attained (Sathish Boddolla et al. 2018).

X-ray powder diffraction (XRD) can be used to further analyze the structural characteristics of the C-Dots. Normally the XRD spectrum of C-Dots exhibits a large diffraction peak of 2θ values around $20-30^\circ$ (Moradi et al., 2018).

4.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is one of the most important analytical techniques available to today's scientists in infrared spectroscopy. This analysis is a type of spectroscopy that can detect changes in the total composition of biomolecules by determining changes in functional groups. The rotation and vibration of molecules influenced by infrared radiation at a particular wavelength is measured using FTIR (Fig 4.4). This structural differences in molecular binding between entities, which can reveal details about the existence of their interactions can be identified using this method. The most popular FTIR- based methods for characterization are Transmittance FTIR, attenuated total reflectance (ATR-FTIR), and micro-spectroscopy FTIR (May M Eid *et al.*,2022).

FTIR are often used to examine the surface properties of the C-Dots. It captures the functional groups in C-Dots prepared from plant-based biomass wastes (Nurul Kamilah Khairul Anuar *et al.*, 2020). The FTIR instrument sends infrared radiation of about range from 10,000 to 100 cm^{-1} through a sample, with some radiation absorbed and some passed through. The absorbed radiation is converted into rotational and/or vibrational energy by the sample molecules. The resulting signal at the detector presents as a spectrum, typically from 4000 cm^{-1} to 400 cm^{-1} , representing a molecular fingerprint of the sample. Each molecule or chemical structure will produce a unique spectral fingerprint, making FTIR analysis a great tool for chemical identification.

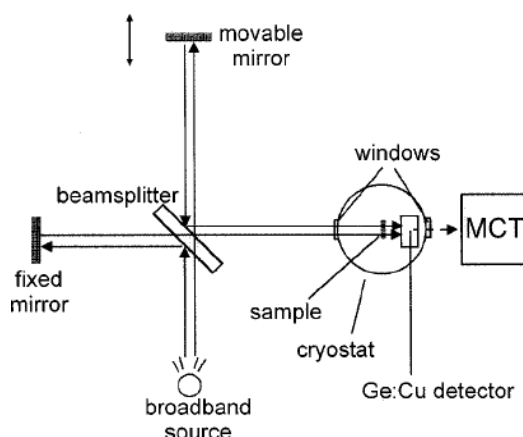


Fig 4.4 Instrumentation of FTIR characterization (D McCluskey *et al.*,2000)

The polar O-H bond usually shows strong and broad absorption bands that are easy to be identified. The broad shape of the absorption band is a result from the hydrogen bonding of the OH groups between molecules. The OH bond of alcohol group usually have absorption in the range of 3200-3600 cm^{-1} , while the OH bond of carboxylic acid group occurs at about 2500-3300 cm^{-1} . The C-H bond stretching of all hydrocarbons occur in the range of between 2800-3300 cm^{-1} . The C=O stretching has strong absorption band in the 1650-1750 cm^{-1} region. Other double bonds like C=C and C=N have absorptions in bit lower frequency regions of about 1550-1650 cm^{-1} . The stretching vibration of triple bonds $\text{C}\equiv\text{C}$ and $\text{C}\equiv\text{N}$ have absorption bands of about 2100~2200 cm^{-1} whereas the polarity of N-H bond is weaker than OH bond, so the absorption band of N-H is not as strong or intense, nor that broad as O-H, and the position is in 3300-3500 cm^{-1} region (Xin Liu *et al.*, 2021).

4.2.3 Raman Spectroscopy

Raman spectroscopy is commonly used in the recognition of metal and special characteristics of molecular structures (Hu *et al.*, 2017). Raman spectroscopy is the powerful non-destructive tool for nanomaterials characterization and to study the phases and phase transitions of various nanostructured materials (Debmalya roy *et al.*, 2020). The defects distributions, identification of amorphous or crystalline regions, doping, shape and size of nanomaterials have been routinely investigated by Raman scattering phenomenon (Drescher D *et al.*, 2012). Raman spectroscopy has played a important role to study the lattice dynamics and vibrational characteristics of sp^2 - hybridized carbons (Reich S *et al.*, 2004). The sp^2 microdomain diameters in nanoscale materials are typically in the order of 5–20 Å and are very sensitive to Raman scattering. The spectrum showed presence of D band and G band in the range 1300-1400 cm^{-1} and 1500-1650 cm^{-1} (Nurul Kamilah Khairol Anuar *et al.*, 2021).

The basic principle behind Raman spectroscopy is that the monochromatic radiation is allowed to pass through the sample such that the radiation may get reflected, absorbed, or scattered (Fig.4.5). The vibration and rotational property vary as the scattered photons have a different frequency from the incident photon. This results in the change of wavelength, which is studied in the IR spectra. The difference between the incident photon and the scattered photon is known as the Raman shift. When the energy associated with the scattered photons is less than the energy of an incident photon, then the scattering is known as Stokes scattering. When the energy of the scattered photons is more than the incident photon, the scattering is known as anti-Stokes scattering. Scattering of electromagnetic waves by any system is related to the heterogeneity of that system. The physics of scattering is the same for every system and independent of the type of heterogeneity. Any particle in the shape of an atom or a molecule of a solid or liquid when illuminated by an electromagnetic wave, the electric charges within them are set to oscillate in an oscillatory motion by the electric field of the incident wave. Accelerated electric charges radiate energy of the same kind in different directions which is called the secondary radiation or radiation scattered by the particle (Sriteja Yamparala *et al.*, 2008).

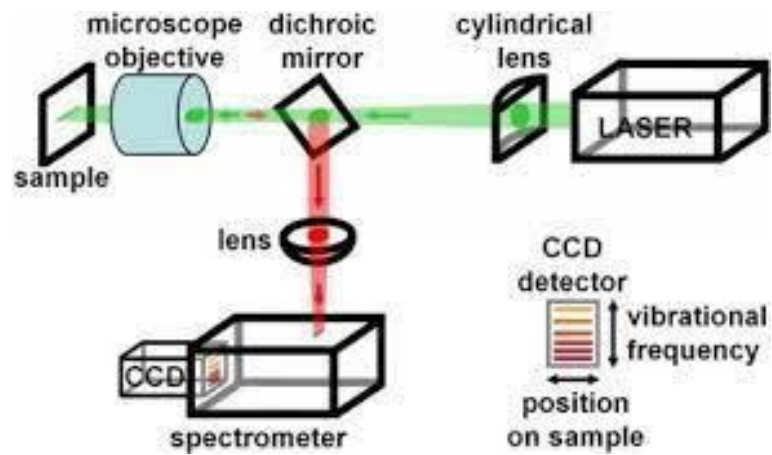


Fig 4.5 Instrumentation of Raman Characterization (Andrew Downes et al.,2016)

4.2.4 Transmission Electron Microscopy (TEM) - EDAX

Transmission Electron Microscope (TEM) transmits the beams of electrons through a biological specimen or sample to produce a high-resolution, magnified image. It is a quantitative method used to determine the particle size, shape and distribution. TEM is also an electronic spectroscopic imaging technique which has higher resolution than SEM and clarity of image with closeness. The advantages of TEM over SEM is its better spatial resolution with the additional capability of analytical measurements. Few limitations of this technique are a requirement of high vacuum, thin sample section and sample preparation is time-consuming. The preparation of sample is extremely important for getting high-quality image for sample information (Hall *et al.*, 2007).

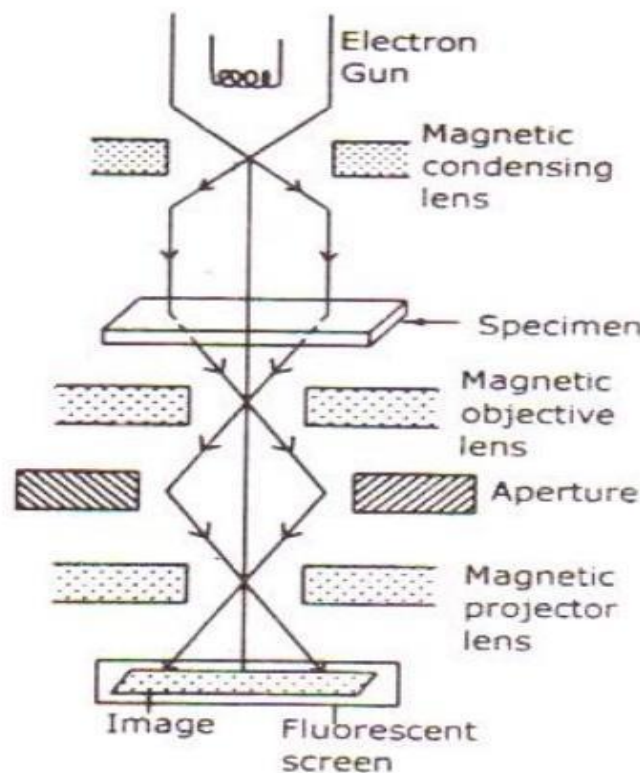


Fig.4.6 Instrumentation of TEM Characterization (Lal Path *et al.*,2016)

A transmission electron microscope is constituted of: (1) two or three condenser lenses to focus the electron beam on the sample, (2) an objective lens to form the image of the

sample in the image plane and the diffraction in the back focal plane, (3) some intermediate lenses to magnify the image or the diffraction pattern on the screen. When the sample is thin (< 200 nm) and constituted of light chemical elements, the image when it is focused presents a very low contrast (Sathish Boddolla *et al.*, 2018). Transmission Electron Microscopy (TEM) is a valuable as well as a frequently used method for the characterization of synthesized nanoparticles. It is used to obtain quantitative measures of particles, size distribution, and morphology.

TEM has mainly two advantages over scanning electron microscopy; it can provide better spatial resolution and provide additional analytical measurements. Different TEM techniques have been used to characterize nanoparticles includes high-resolution TEM (HR-TEM), scanning transmission electron microscopy (STEM) imaging, spectroscopy techniques including X-ray energy dispersive spectroscopy and diffraction techniques including selected area electron diffraction (SAED). These techniques allow the study size distribution, compositional distribution, phase identification and morphology of synthesized nanoparticles. HR-TEM is an imaging mode of TEM, which uses phase-contrast imaging, where both transmitted and scattered electrons are combined to produce the image. HR-TEM is the most commonly used method to determine the internal structure of nanoparticles. It makes use of a larger objective aperture to detect the scattered electrons. The arrays of atoms in crystalline structures can be detected by this method (Su *et al.*, 2017).

TEM offers a significantly enhanced resolution ($0.0001\text{ }\mu\text{m}$), of about one to two orders of magnitude higher than that of the SEM. Due to the complex process of sample however preparation and time-consuming analysis, this technique has been rarely utilized in the size distribution of marine particles can be determined (Kim *et al.* 1995). Ordinary TEM measures the absorbance of the beam. In HRTEM image formation relies or depends on phase contrast, and, as the image is influenced by aberrations of the imaging lenses in the microscope, it is not interpretable. So proper understanding of the sample is required for interpretation of the results.

CHAPTER-5

RESULTS AND DISCUSSION

5.1 XRD ANALYSIS

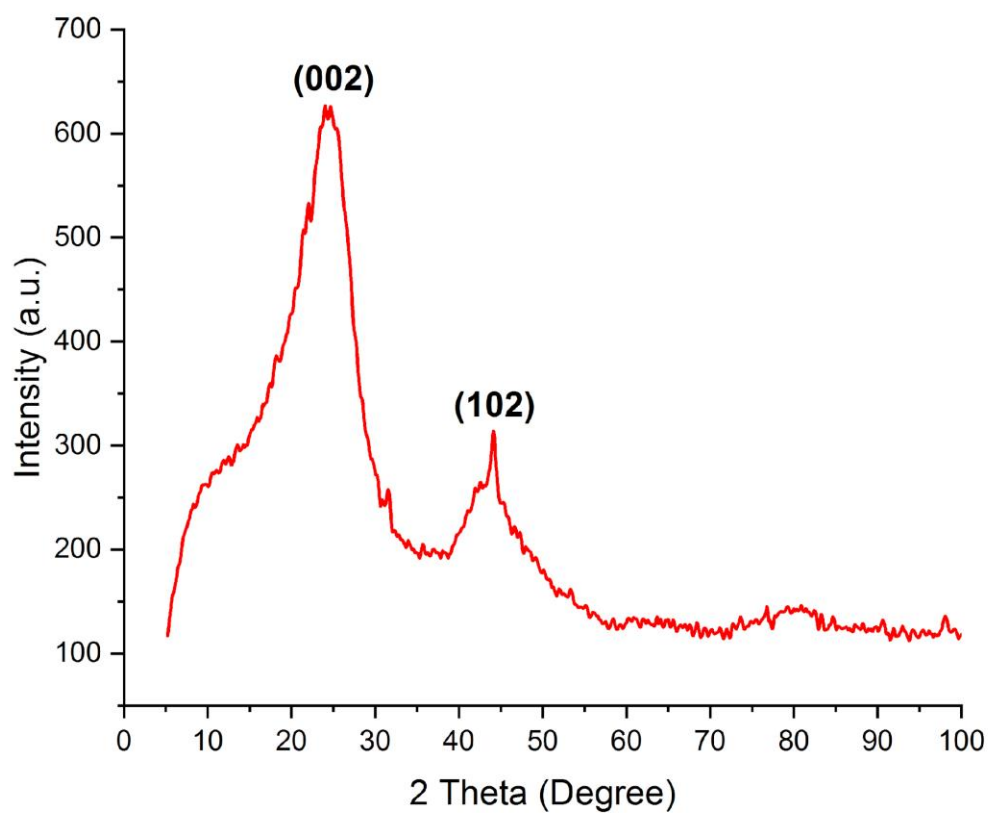


Fig. 5.1 X - ray diffraction (XRD) pattern of carbon nanostructured materials

Fig 5.1. shows the X- ray diffraction (X- Ray) pattern of the carbon nanostructured materials which was green synthesized using seaweed (*Halimeda Opuntia*). The crystal structure and the atomic structure of the prepared sample can be determined using

XRD method. The XRD pattern of carbon nanostructured materials usually exhibits a diffraction or sharp peak. These peaks can have different Miller indices (hkl) to identify different planes of atoms. The observed peaks of diffraction can be related to atoms plane which helps to analyze the atomic structure and microstructure of a samples. By analysing the graph we got peaks at $2\theta = 24^\circ$,the miller indices for which is (002) and another peak at $2\theta = 45^\circ$ its miller indices is (102) . This XRD result is in good agreement with the previous reports (Theivasanthi Thiruganasambanthan *et al.*,2021)

5.2 FTIR ANALYSIS

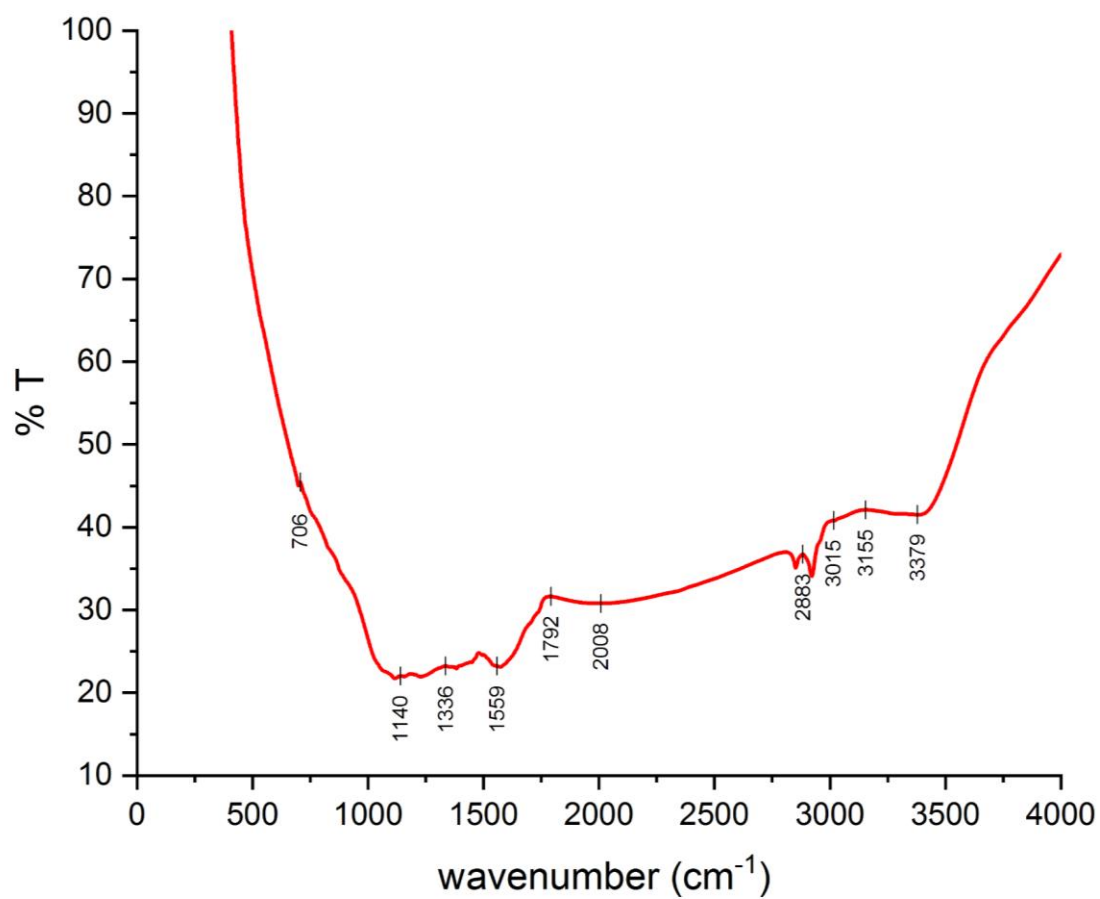


Fig 5.2. FTIR spectrum of carbon nanostructured materials

FTIR spectroscopy is an analytical technique used to identify organic, polymeric and the functional groups which are responsible for the formation of nanostructured materials using the sea weed . The surface properties of carbon nanostructured materials is examined using this unique method. The graph obtained after examining the values gives certain peaks which shows corresponds to some stretching's . Fig.5.2 shows the FTIR spectrum of carbon nanostructured materials . The peaks at points 2883 cm⁻¹, 3379 cm⁻¹, 3015 cm⁻¹, 3155 cm⁻¹, 2008 cm⁻¹, 1792 cm⁻¹, 1559 cm⁻¹, 1336 cm⁻¹, 1140 cm⁻¹ and 706 cm⁻¹. The bands at 2883 cm⁻¹ and 3015 cm⁻¹ corresponds to C-H stretching. The OH stretching mode is observed at peak 3379 cm⁻¹ . The peak at 1559 cm⁻¹ shows C=O bending whereas peaks at 1140 cm⁻¹, 1336 cm⁻¹ and 1559 cm⁻¹ shows C-H/C-O bonding (Kyung Woo Kim eal., 2022).

5.3 RAMAN ANALYSIS

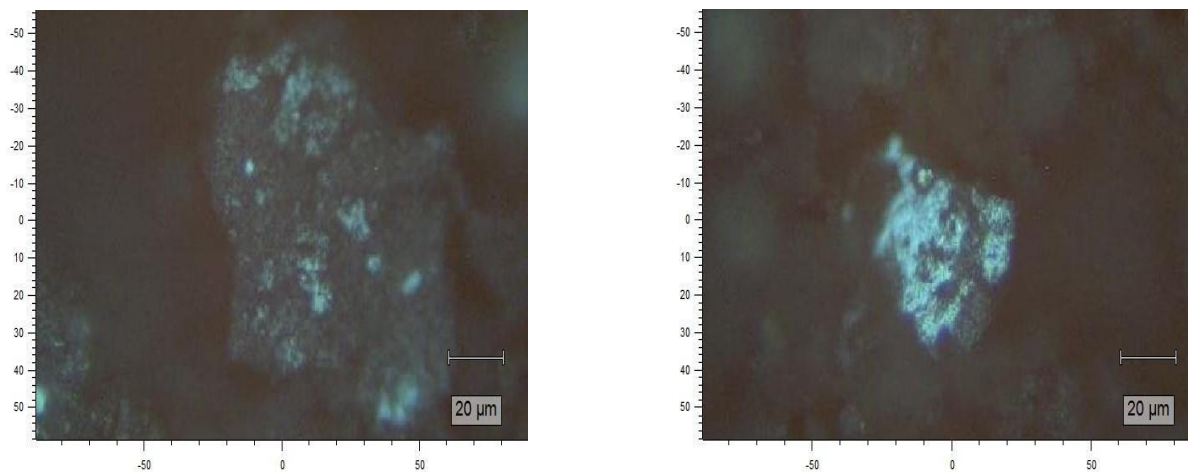


Fig. 5.3(a) Dimension of carbon nanostructured materials

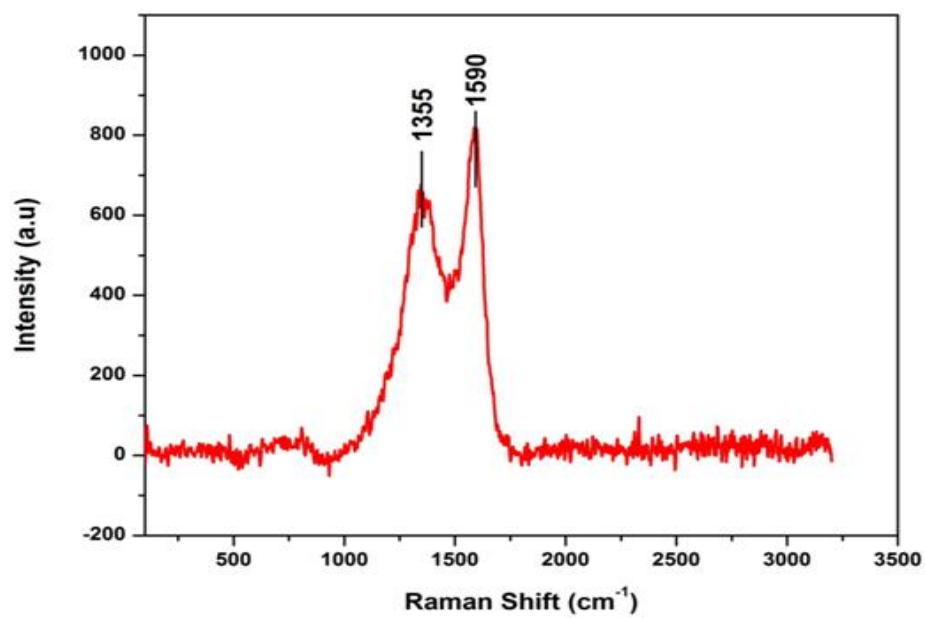
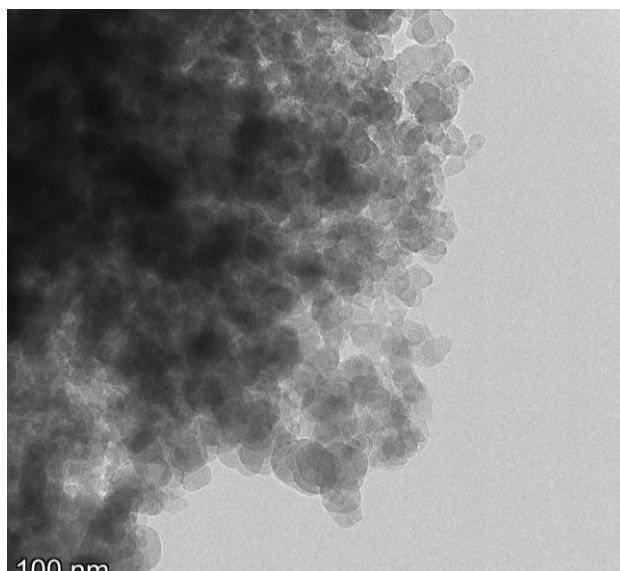


Fig. 5.3(b) Raman spectrum of carbon nanostructured materials

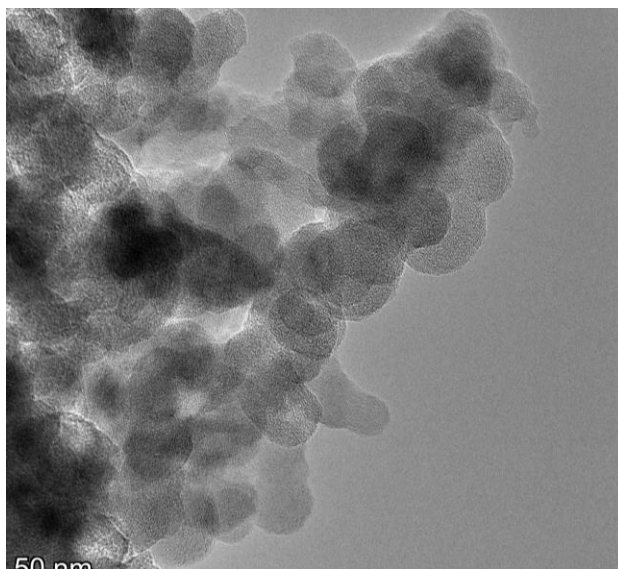
Fig.5.3 (a) shows the dimension or size of carbon nanoparticles which were analyzed in this characterization. Raman characterization is basically used to study the morphology and defect density of sample. The spectroscopy is a tool to determine the region which is amorphous or crystalline, the lateral dimensions or diameter, and the defects present in the various types of nanomaterial (G Katumba *et al.*, 2008). Nanoscale materials are very sensitive to Raman scattering and the sp^2 microdomain diameter typically ranges in the order 5-20 Å (Debmalya Roy *et al.*, 2020).

Fig.5.3 (b) show the raman spectrum of in carbon nanostructured materials which shows two unique peaks. These peaks represents formation of two bands, the D band and G band will be respectively. Usually the D band will be in the range of 1300-1400 cm^{-1} and G band 1500-1650 cm^{-1} (Nurul Kamilah Khairul Anuar *et al.*, 2021). From Fig.5.3 (b), it is clear the D band formed at 1355 cm^{-1} and G band at 1590 cm^{-1} , which is in consistent with the literature reports.

5.4 TEM-EDAX ANALYSIS



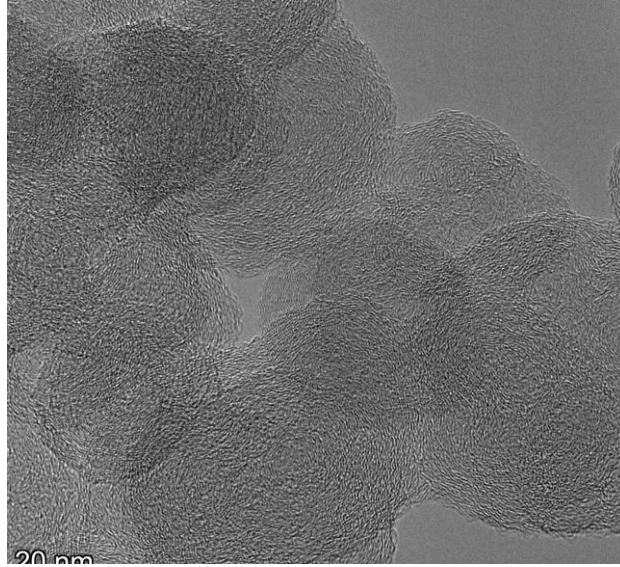
(a)



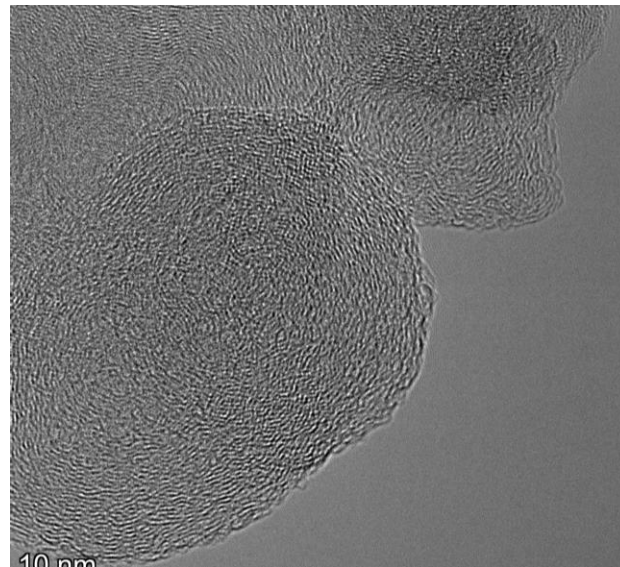
(b)

Fig. 5.4(a-b) TEM images of carbon nanospheres

5.4.1 HR-TEM ANALYSIS



(c)



(d)

Fig. 5.4 (c-d) HRTEM images of carbon nanospheres

Transmission electron microscopy (TEM) is a microscopic technique in which electron beams are passed , transmitted through specimen and resolution image is

obtained. The images obtained by examining carbon nanostructured materials is shown in Fig 5.4 (a,b) TEM images shows that the prepared carbon nanostructured materials has spherical morphology and uniform. A type of TEM with highest resolution is HR-TEM in which a imaging of up to 0.050nm resolution can also be obtained. TEMs are capable of imaging at significantly higher resolution than light microscope. Usually TEM is used to determine the shape and size of nanomaterials. Fig 5.4 (c,d) shows HR-TEM images of carbon nanostructured materials characterization which ranges from 10-20 nm. HR-TEM gives more wide perspective about the size of particle with much higher resolution and closer imaging. It can also be observed that the images obtain shows a spherical pattern of carbon nanospheres. The EDAX patterns revealed the presence of carbon, copper and nitrogen elements present in the specimens.

5.4.2 EDAX PATTERN OF CARBON NANOSTRUCTURED

MATERIALS

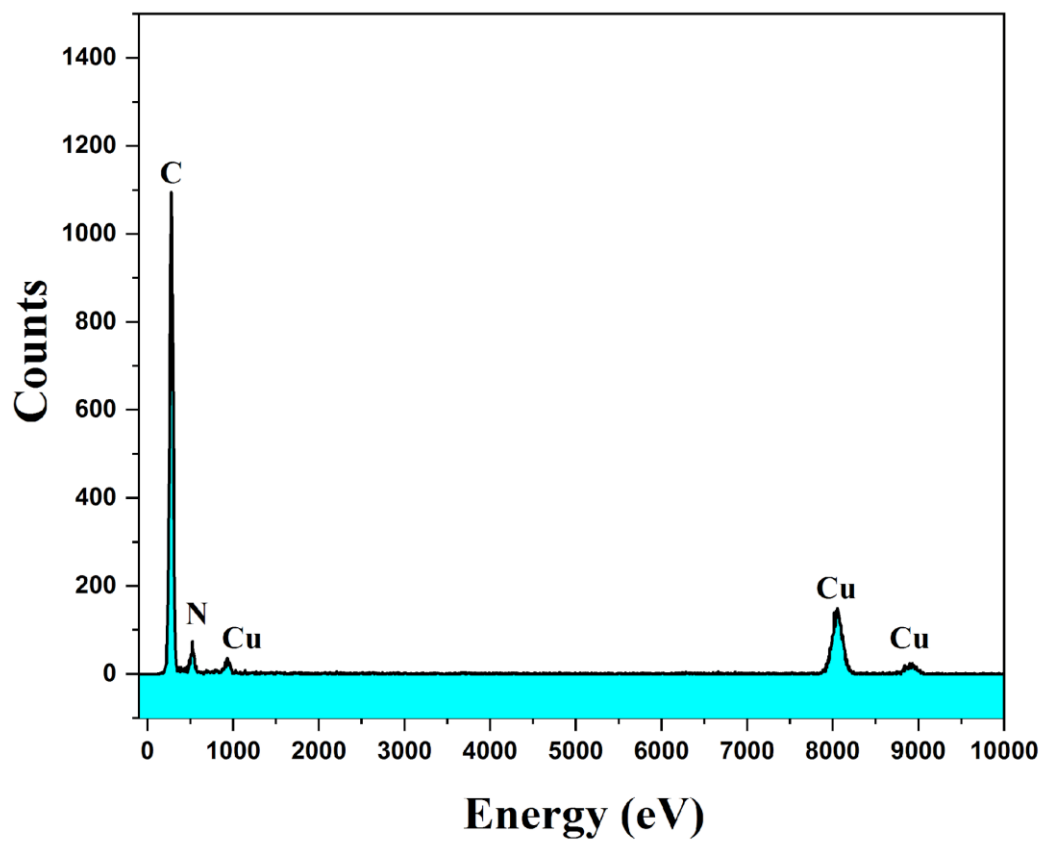


Fig.5.4(e) EDAX pattern of carbon nanostructured materials

CHAPTER-6

APPLICATION

6.1 PREPARATION OF BACTERIAL CELLS

Materials required

1. Muller Hinton agar (MHA) plates
2. Muller Hinton broth (MHB)
3. Micropipette
4. Micropipette tips
5. Microbial loop
6. Sterile cotton swab
7. 0.5 McFarland standard

Test pathogens: *Pseudomonas aeruginosa* and *Staphylococcus aureus*

Sample preparation:

- Given sample was dissolved in 5 % DMSO and vortexed well.

6.2 PREPARATION OF BACTERIAL INOCULUM

- A loopful of test pathogens were inoculated in MHB respectively and adjusted to 0.5 McFarland standard.

6.3 DETERMINATION OF GROWTH KINETICS IN THE PRESENCE OF CARBON NANOMATERIAL

The bacterial growth kinetics was monitored to check the effect of carbon nanomaterial in different concentrations (10, 50, 100, 150 and 200 µg/ mL) with 10^6 cells of each bacterial isolates grown in LB broth medium. A control was maintained under same conditions in the absence of carbon nanomaterial. They were incubated at 37 °C and the optical density (OD) was measured at 660 nm at an interval of 3 h for 18 h.

6.4 RESULTS

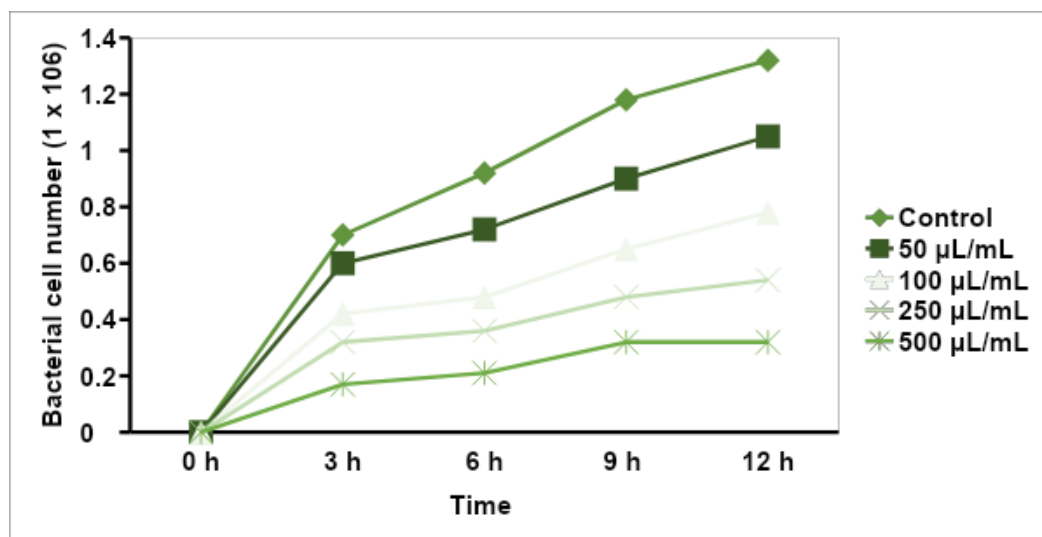


Fig 6.1 Growth kinetics study of bacterial species *P. aeruginosa* when subjected to different concentrations (10, 50, 100, 150 and 500 µg/mL) of carbon nanoparticles.

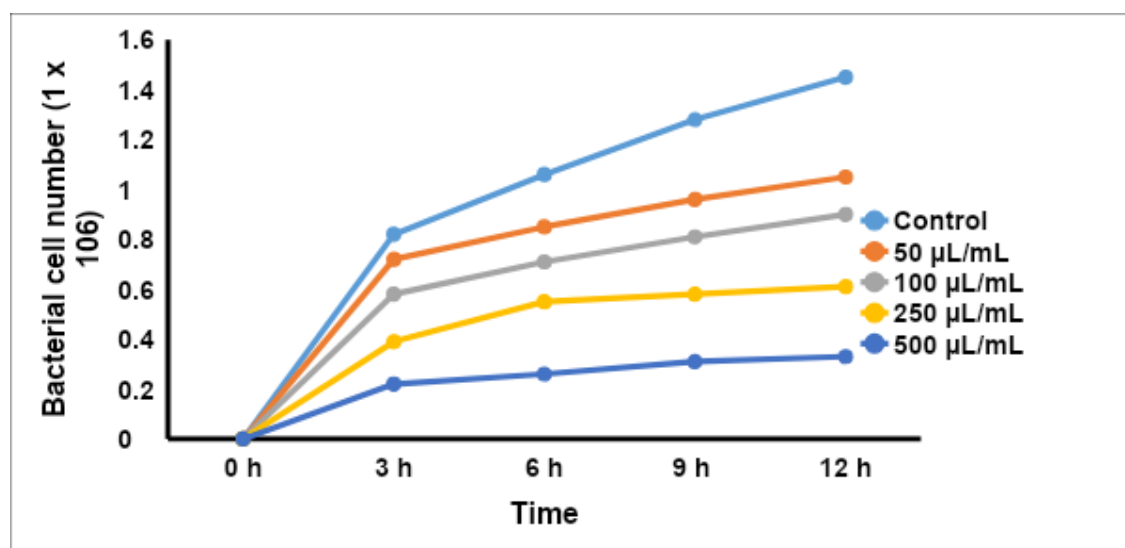


Fig 6.2 Growth kinetics study of bacterial species *S. aureus* when subjected to different concentrations (10, 50, 100, 150 and 500 µg/mL) of carbon nanoparticles.

The bacterial growth dynamics were analysed with 1×10^6 cells treated with different concentrations (50, 100, 250 and 500 µL/mL) of carbon nanoparticles. The

bacterial growth kinetics (*Pseudomonas aeruginosa* and *Staphylococcus aureus*) were monitored at a time interval of 3 hours. It is apparent from the graph (Fig 6.1, 6.2) that the number of bacterial cells decreased with increasing concentration of carbon nanomaterial and with different times of exposure. Moreover, there is no growth inhibition in cultures free of polymers solutions and the bacterial cells survived till they reached the static phase. The field of nanotechnology is constantly expanding in an effort to produce novel materials with practical applications. To keep up with the pace of changes in science and technology, new nanomaterials with modern and supreme features are needed (Yang et al., 2019; Alvial-Palavicino et al., 2019). Among the different nanostructures, carbon nanomaterials and their derivatives have unique characteristics and applications.

The antimicrobial activity of carbon nanomaterials depends on their composition, surface modification, target microorganisms, and reaction environment (Alizadeh et al., 2019). The antimicrobial mechanisms of carbon nanomaterials are based on the invasion of the microbial cell wall/membrane and induction of structural damages, besides the physical mechanism of biological isolation of microbial cells from their supportive environment (Alizadeh et al., 2019; Zhou et al., 2019; Le et al., 2008). Chemical antimicrobial actions comprise the third group of mechanisms that depend on the interaction of carbon nanomaterials with the microorganisms and the establishment of oxidative stress conditions via the production of toxic materials like reactive oxygen species (ROS). The carbon nanomaterial-microorganism interactions lead to electron transfer; ROS-independent oxidative stress is induced as the electrons are evacuated from the microbial surface, leading to biological death (Zhou et al., 2019; Tang et al., 2012).

This study has been defined to cover the significant developments in the use of CNMs individually or in nanocomposites as antimicrobial agents in recent years; it will critically focus on the antimicrobial studies (*in vitro/in situ*) and applications of these nanostructures against pathogens. Carbon nanomaterials can be considered as a novel class of antibiotics/ antimicrobial agents for various fields including biomedical and food applications.

CHAPTER-7

SUMMARY AND CONCLUSION

- A simple, cost-effective and environmental benign route is formulated for the preparation of Carbon nanostructured material using green synthesis of seaweed (*Halimeda Opuntia*) by thermal Pyrolysis method.
- From XRD studies, pattern of carbon nanostructured materials were obtained at two peaks that is 24 ° and 45 ° with their respective Miller indices .
- From FTIR studies, peaks were obtained at 2883 cm⁻¹, 3379 cm⁻¹, 3015 cm⁻¹, 3155 cm⁻¹, 2008 cm⁻¹, 1792 cm⁻¹, 1559 cm⁻¹, 1336 cm⁻¹, 1140 cm⁻¹ and 706 cm⁻¹. The peak at 2883 cm⁻¹ and 3015 cm⁻¹ belonged to the C-H stretching, OH bond at peak 3379 cm⁻¹, C=O bonding at 1559 cm⁻¹, 1336 cm⁻¹, 1140 cm⁻¹ peaks respectively.
- From TEM, the dimension were obtained as 100 nm and 50nm, while for HR-TEM it was 10 nm and 20 nm respectively. The EDAX patterns revealed the presence of carbon, copper and nitrogen elements present in the specimens.
- From Raman analysis, the two band D and G was achieved at peaks ranging from 1300-1400 cm⁻¹ and 1500-1650 cm⁻¹.
- In this study, the application of carbon based nanospheres on antibacterial activities were done by studying the growth kinetics of gram- positive as well as gram-negative bacterial species. The bacterial growth kinetics (*Pseudomonas aeruginosa* and *Staphylococcus aureus*) were monitored for few intervals.
- This study has been defined to cover significant developments in the use of CNMs individually or in nanocomposites as antimicrobial agents and application of these nanostructures against pathogens.
- Carbon nanomaterials can be considered as a novel class of antibiotics/ antimicrobial agents for various fields including biomedical and food applications.

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