PROJECT REPORT

On

VALORISATION OF WASTE COTTON CLOTH INTO CELLULOSE NANOCRYSTALS AND CARBON DOTS: A WASTE TO WEALTH APPROACH

Submitted by

ANN LIYA JUDE
(AB23CHE017)

In partial fulfillment for the award of the

Bachelor's Degree in Chemistry

DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

ST. TERESA'S COLLEGE (AUTONOMOUS)
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ST. TERESA'S COLLEGE (AUTONOMOUS)

ERNAKULAM

B.Sc. CHEMISTRY PROJECT REPORT

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Register Number : **AB23CHE017**
Year of Work : **2025-2026**

This is to certify that the project "**VALORISATION OF WASTE COTTON CLOTH INTO CELLULOSE NANOCRYSTALS AND CARBON DOTS: A WASTE TO WEALTH APPROACH**" is the work done by ANN LIYA JUDE.

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Submitted to the Examination of Bachelor's Degree in Chemistry

Date: 10/04/2026

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DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

ST. TERESA'S COLLEGE (AUTONOMOUS)

ERNAKULAM

CERTIFICATE

This is to certify that the project work entitled "**VALORISATION OF WASTE COTTON CLOTH INTO CELLULOSE NANOCRYSTALS AND CARBON DOTS: A WASTE TO WEALTH APPROACH**" is the work done by **ANN LIYA JUDE,** under our guidance in the partial fulfilment of the award of the Degree of Bachelor of Science in Chemistry at St. Teresa's College (Autonomous), Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

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DECLARATION

I hereby declare that the project work entitled "**VALORISATION OF WASTE COTTON CLOTH INTO CELLULOSE NANOCRYSTALS AND CARBON DOTS: A WASTE TO WEALTH APPROACH**" submitted to Department of Chemistry and Centre for Research, St. Teresa's College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by me under the guidance of **Dr. USHAMANI M, PROFESSOR; Ms. SICILY RILU JOSEPH, RESEARCH SCHOLAR,** Department of Chemistry and Centre for Research, St. Teresa's College (Autonomous), Ernakulam and this project work is submitted in the partial fulfilment of the requirements for the award of the Degree of Bachelor of Science in Chemistry.

ANN LIYA JUDE

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Contents

Chapter 1

Introduction

1.1 Waste to wealth

One of the major issues that our world face today is waste management which is essential for a sustainable living. In such a scenario, the 'Reduce, Reuse, Recycle (R-R-R)' principle (Fig.1.1) becomes relevant. 'Reduce' aims to mitigate the amount of waste generated every year by choosing renewable and sustainable products. The environmental impact of wastes can be lessened by making public aware of the subsequent consequences and thereby making them to rethink their excessive purchasing habits and adopt a sustainable living. 'Reuse' emphasizes the utilization of waste and unwanted substances in day-to-day life and use it as an alternative to the existing materials instead of discarding it as a waste. 'Recycle' is a means of converting already used materials to valuable products, so that they can be used for future needs(1). Cotton (*Gossypium spp.*) is a plant species widely cultivated in tropical and subtropical regions around the world. It belongs to the *Malvaceae* family (2). Cotton is mainly processed to make textiles and a large quantity of cotton waste—such as linters, short fibers, and damaged fabric—is typically thrown away because it cannot be used directly. Reusing waste cotton textiles will conserve resources, promotes sustainability and circular economy, reduces pollution, environmental burden and creates value added products like cellulose nanocrystals, carbon dots and more. The key concept of our work is 'Waste to Wealth'. It refers

to the process of transformation of waste materials into valuable products or resources. This initiative plays a crucial role in promoting sustainable waste management, minimizing environmental pollution and driving economic growth.

Fig.1.1: The 3R hierarchy – 'Reduce, Reuse, Recycle'

1.2 CELLULOSE

Cellulose stands out as an ideal choice for sustainable material development. It is a complex carbohydrate with molecular formula $(C_6H_{10}O_5)_n$ shown in Fig1.2. It is the most abundant organic compound on Earth. It is present in the cell walls of plants and algae. Cellulose is made of D- glucopyranosyl units linked by β-1, 4 -glycosidic bonds (3). They are highly crystalline, insoluble in water, renewable and biodegradable. Through photosynthesis, plants synthesize cellulose. It is comprised of plasma membrane, hemicellulose, pectin etc. as shown in Fig.1.3

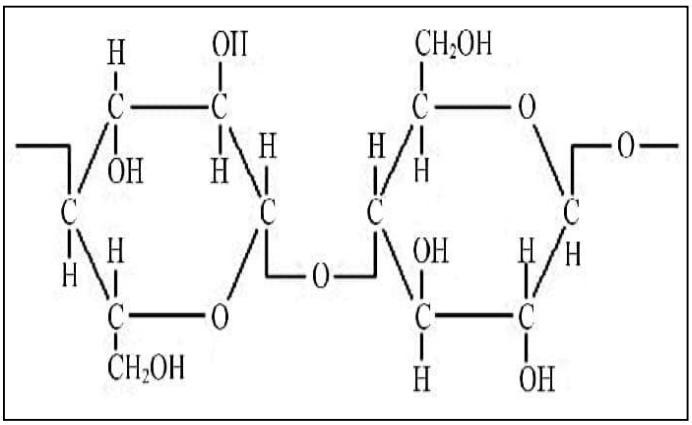

Fig.1.2: Structure of Cellulose

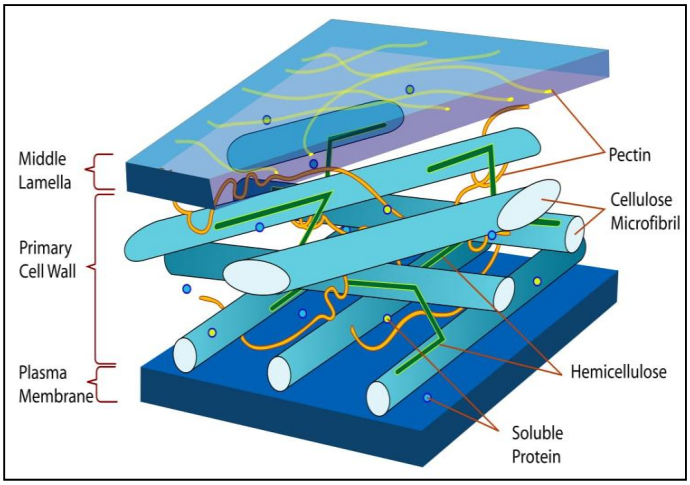

Fig.1.3: Plant cell wall

1.2.1. Sources of Cellulose

There is natural as well as synthetic sources of cellulose. The primary source of cellulose is the plant cell wall. Natural fibers are extracted from cotton, jute, flax, ramie, sisal, hemp etc. Synthetic fibers are derived from fabrics like Lyocell, modal, rayon etc. Fig.1.4. shows the different sources of cellulose.

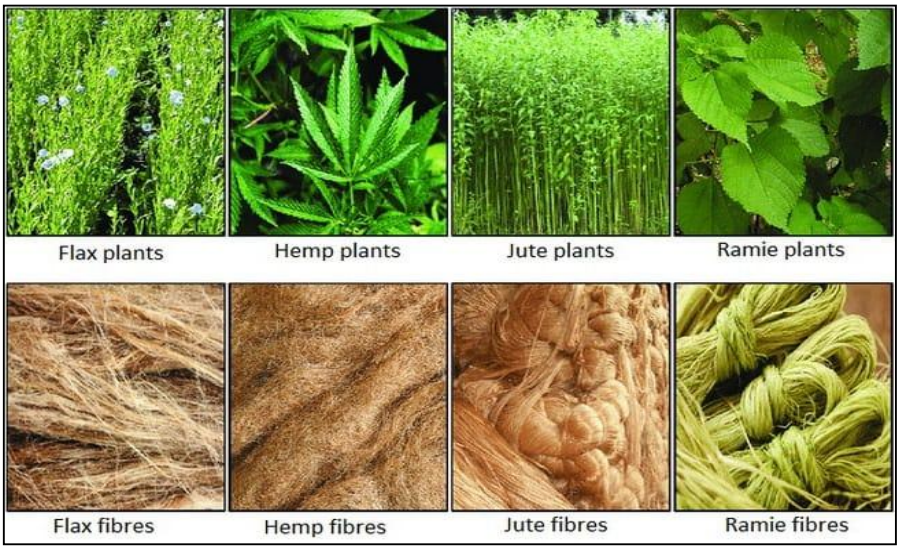

Fig.1.4: Sources of Cellulose

There are lignocellulosic sources which include animal, algae and bacterial sources as shown in Fig.1.5.

i. The lignocellulosic sources can be divided into two:

a. Woody residues: Hardwood, Softwood (4) and Sawdust wastes (5)

b. Non-woody plants and Agricultural residues: Kenaf (6), Corn husk (7), Bamboo (8), Watermelon rind and Water hyacinth, Sugarcane bagasse (9) etc.

ii. Animal sources: Tunicates like *Halocynthia papillosa* (10)*, Halocynthia roretzi* (11), *Styela plicata* (12) etc.

iii. Algae (red, green, grey, yellow-green etc)*: Cladophorales* and *Siphonocladales* are the orders of algae that produce cellulose. *Cladophorales* include *Cladophora* (13) *Chaetomorpha* (14), *Rhizoclonium* (15) etc. and *Siphonocladales* include *Valonia*, *Dictyosphaeria* (16) etc. *Gelidium* red algae is another example.

iv. Bacteria: *Acetobacter xylinus*, *Agrobacterium*, *Seudomonas*, *Rhizobium and Sarcina* are examples of cellulose producing bacteria. *Acetobacter*

xylinum which is a Gram-negative acetic acid bacterium is the most dominant among these to produce cellulose.

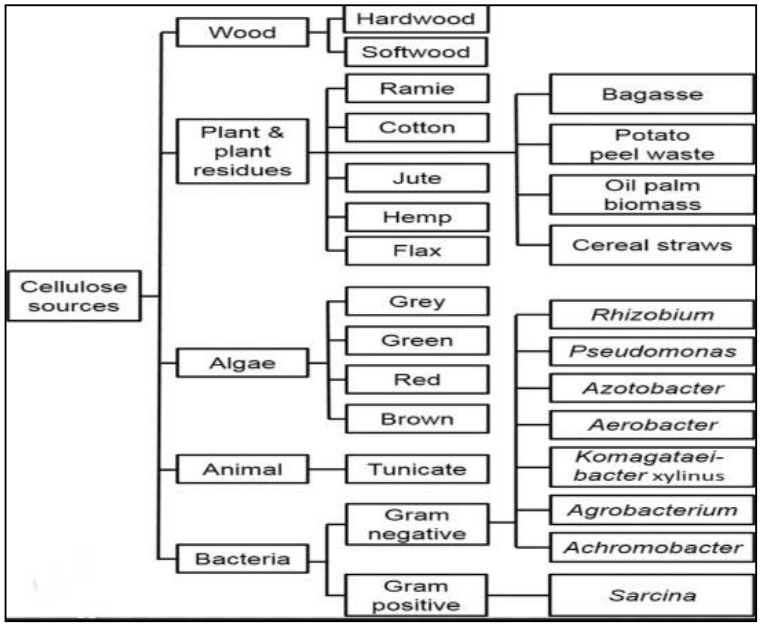

Fig.1.5: Sources of Cellulose (b)

1.3 COMPOSITION OF LIGNOCELLULOSIC BIOMASS

Cellulose Nanocrystals (CNCs) can be effectively segregated and purified from the complex matrix of Lignocellulosic Biomass (LB) using specialized techniques. Lignocellulosic Biomass (LB) is a plant-derived material characterized by its heterogeneous composition, comprising of the polysaccharides like hemicelluloses, cellulose as well as lignin, a complex aromatic biopolymer as in Fig.1.6 (17).

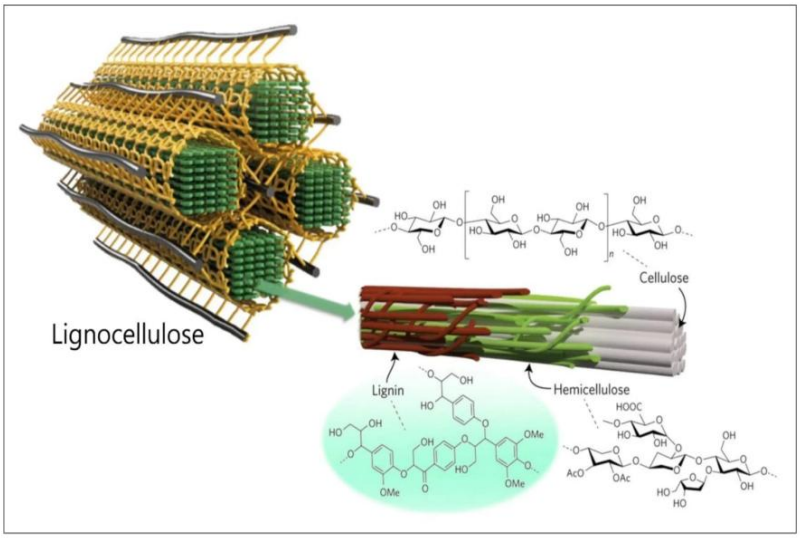

Fig.1.6: Components of Lignocellulosic biomass (LB)

1.3.1 Cellulose

Although the earth generates more than thousand tons of cellulose each year, a mere 5% is currently extracted and utilized in diverse products. In 1838, Anselme Payen successfully extracted cellulose from LB via nitric acid treatment and determined its molecular composition as $C_6H_{10}O_5$. Notably, the term "cellulose" was first formally articulated within the scientific community in 1839. (17)

Cellulose is a homopolymer. It is entirely made up of glucose and no other sugar unit is present. It is the main structural element of plant cell wall. Depending on its source and type of processing, cellulose shows variability in its degree of polymerization. But in general, it exhibits higher degree of polymerization (18).

Native cellulose, produced by plants, exists in two main crystalline forms: cellulose I and cellulose II, with cellulose II commonly found in marine algae and formed by treating cellulose I with sodium hydroxide. Among

cellulose's four crystalline polymorphs - I, II, III, and IV - cellulose II exhibits the highest thermodynamic stability, whereas cellulose I is relatively less stable. Notably, liquid ammonia treatment of cellulose I and II results in the formation of crystalline cellulose III, which subsequently transforms into cellulose IV upon heating (19).

1.3.2 Lignin

Native lignin, also referred to as protolignin, is a complex, three-dimensional, crosslinked macromolecule biosynthesized from the polymerization of three primary phenylpropanoid precursors: Coniferyl alcohol, paracoumaryl alcohol and sinapyl alcohol. The specific proportions of these precursors vary significantly depending on the species, source, and type of lignocellulosic biomass (LB). This variability influences the structural and functional properties of lignin. Lignin acts as a binding agent in cell tissues, holding cellulose and hemicellulose fibers together. As plant cell walls mature, lignin provides stiffness and rigidity. Lignin contributes to the water resistance and impermeability of plant cell structures, protects plants against pathogens and environmental stresses. Lignin's complex structure and functionality make it an essential component of plant cell walls, providing strength, stability, and resistance to degradation (17).

1.3.3 Hemicellulose

The term "hemicellulose" was first coined by Schulze to describe polysaccharides extractable from lignocellulosic biomass (LB) using dilute alkali and readily hydrolysable by dilute acids. Hemicelluloses comprise 15-35% of LB and are the second most abundant constituent after cellulose. These non-crystalline polysaccharides exhibit complex structures comprising various derivatives of pentoses (arabinose and xylose), hexoses

(galactose, fucose, glucose, mannose, and rhamnose), and glycolytic acid (glucose fermentation acid, methyl-glucose and galacturonic acid). The primary sugar units in hemicelluloses are xylans and glucomannans. Hemicelluloses adhere to cellulose through Van der Waals and hydrogen bonding and form strong cross-links with lignin. By integrating between cellulose and lignin, hemicelluloses play a vital role in maintaining cell wall structure. Hemicelluloses can be removed from lignocellulosic biomass (LB) through pretreatment methods such as alkali, dilute acid, or steam explosion (17).

1.4 NANOCELLULOSE

Nanocellulose refers to the cellulose material possessing at least one dimension within the nanoscale range of 1-100 nanometers (20). Properties like low density, flexibility, high strength, chemical inertness etc. contribute towards its outstanding applications (17).

Low density of nanocellulose makes it lightweight. High aspect ratio, strength etc contributes to its potential as a reinforcing agent. The surface of nanocellulose is ample with hydroxyl bonds which in turn promote hydrogen bonding. CNC has the ability to self-assemble into chiral nematic liquid crystalline phase. Their potential in packaging applications is liable to their low-porosity (21). Fig.1.7 represents the schematic representation of nanocellulose present in plant cell wall.

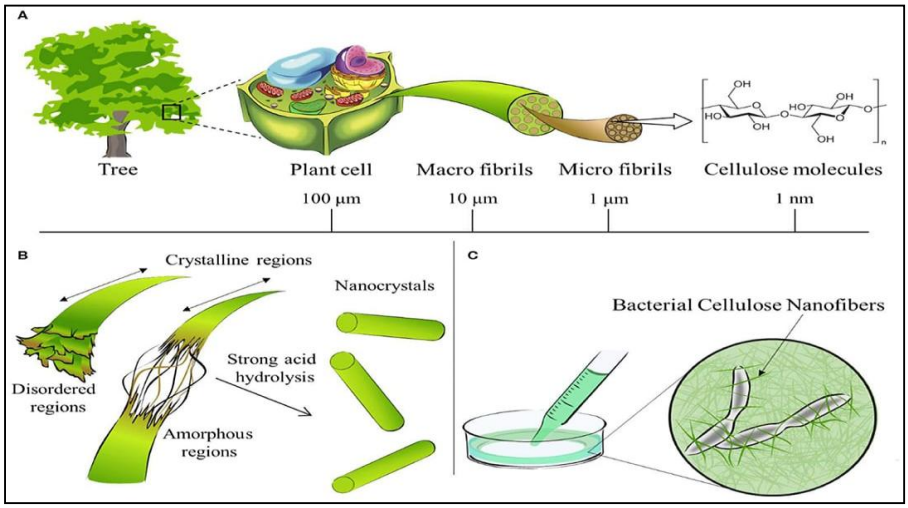

Fig.1.7: Schematic representation of Nanocellulose present in plant cell wall (22)

1.4.1 Characteristics of Nanocellulose

Cellulose researchers have noted that plant and crop cellulose is made of millimeter-sized fibers, which in turn are made of increasingly denser microfibers. These microfibers further consist of nanometer-sized microfibrils that form the basic building blocks of cellulose from different sources. Scientists realized the potential of these nanosized microfibrils, which have vast surface areas and strong bonding capacities, to create innovative products with significantly enhanced strength and functionality (23). Nanocellulose holds a remarkable number of properties and, thus, is quite versatile in diverse applications. Its mechanical strength, large specific surface area, and stiff structure provide great potential in reinforcing composite applications and making it chemically modifiable. In addition to these beneficial properties, nanocellulose's biocompatibility and biodegradability coupled with its very good barrier properties as well as its ability to form stable gels and emulsions open wide windows for even more

extensive uses in the biomedical, environmental, packaging, and drug delivery sectors. Fig.1.8 shows the important characteristics of nanocellulose.

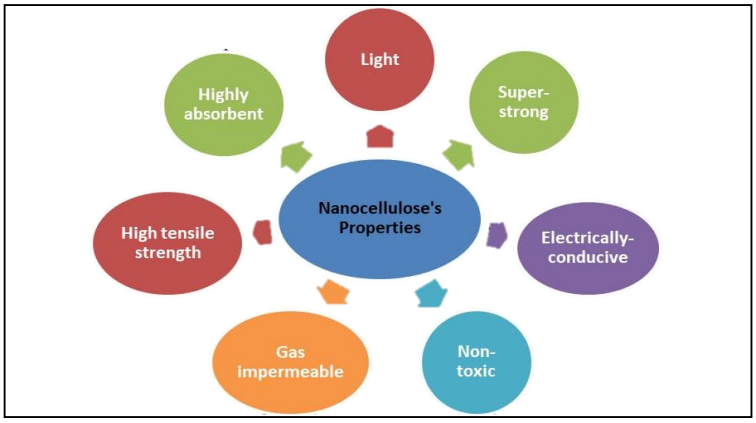

Fig.1.8: Characteristics of Nanocellulose

1.4.2 Classification of Nanocellulose

Cellulose nanostructured materials are comprised of four main classes: Cellulose nanofibrils (CNF), cellulose nanocrystals (CNC)/ cellulose whiskers, bacterial nanocellulose (BNC) and Electrospun cellulose Nanofiber (ECN) (20) as shown in Fig.1.9. Cellulose nanocrystals (CNC) can be prepared using acid hydrolysis whereas Cellulose nanofibrils (CNF) production usually involves chemical pretreatment and subsequent mechanical disintegration through homogenization, micro fluidization, or super-grinding. The dimensions of cellulose nanocrystals (CNC) typically range a few nanometers in diameter and 10-500 nanometers in length, whereas cellulose nanofibrils (CNFs) have diameters ranging from 3-50 nanometers and lengths up to several micrometers(17). Rice husk, wheat straw, sugarcane bagasse pulp, wood pulp, cotton linters, bacterial cellulose, algal cellulose etc. are the various sources of nanocellulose (24).

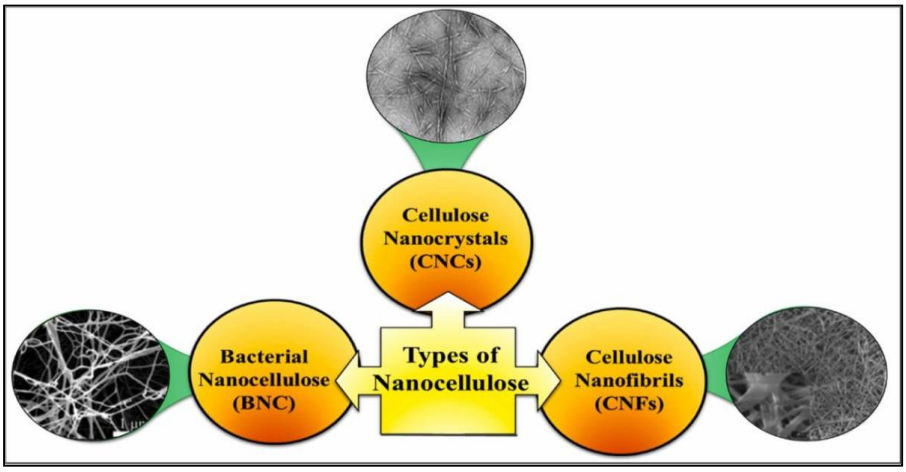

Fig.1.9: Classification of Nanocellulose

1.4.2.1 Cellulose Nanocrystals (CNC)

Cellulose nanocrystals (CNCs) are rod- or needle-shaped materials with high crystallinity. During acid hydrolysis Fig. 1.10, the amorphous regions are removed, leaving behind CNCs with diameters of about 5 nm and lengths ranging from 20 to 100 nm. (20)

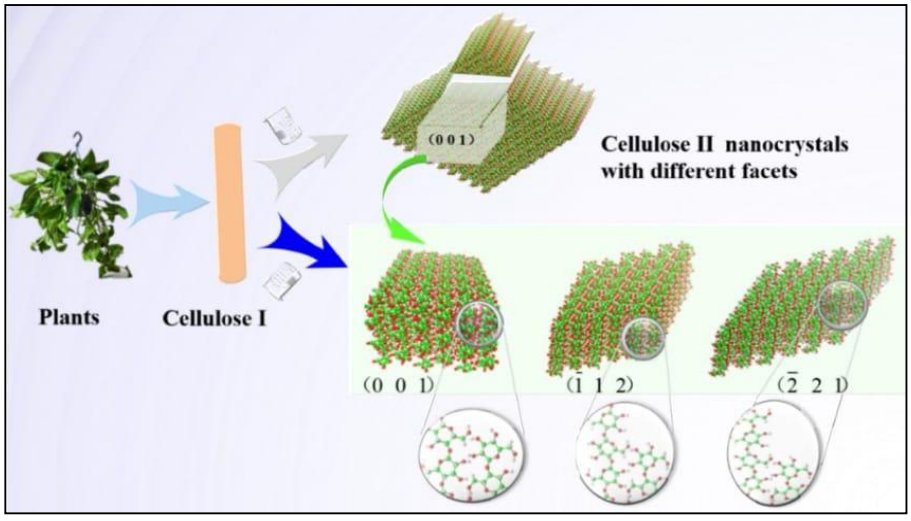

Fig.1.10: Synthesis of Cellulose nanocrystals (CNC) through acid hydrolysis

1.4.2.2 Cellulose Nanofibres (CNF)

Cellulose nanofibrils are nanoscale cellulose fibers. They exhibit diameters between 5-60 nm and lengths up to several microns. These fibrils comprised of both crystalline and amorphous regions. They are typically extracted from wood pulp using the TEMPO-mediated oxidation method (20). Fig.1.11. shows the synthesis of cellulose nanofibres (CNF) from wood pulp**.**

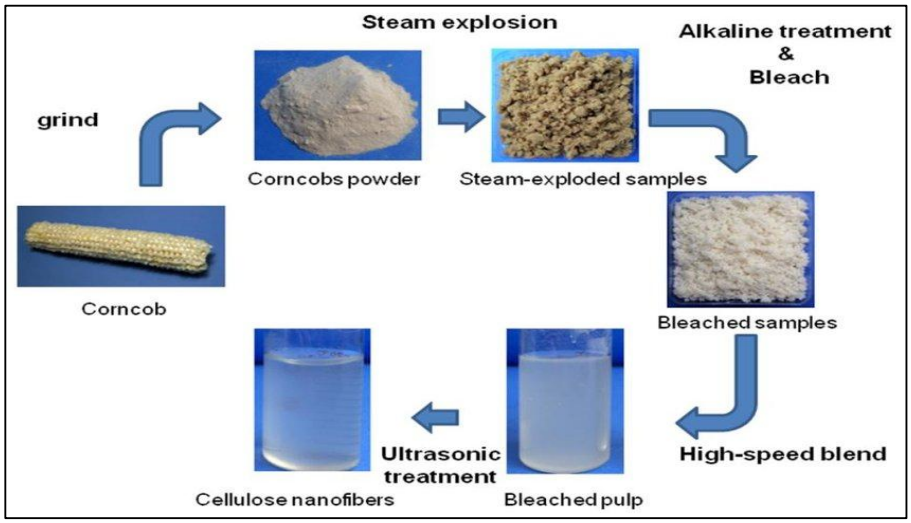

Fig.1.11: Synthesis of Cellulose nanofibre (CNF)

1.4.2.3 Bacterial Nanocellulose (BNC)

Bacterial Nanocellulose (BNC) exhibits a ribbon-shaped morphology with dimensions ranging from 20-100 nm and many micrometers in length. Fermentation by specific bacterial genera like *Rhizobium*, *Agrobacterium*, *Gluconacetobacter*, and *Sarcina* generates Bacterial nanocellulose (BNC) as an extracellular product. The purity of BNC makes it stand apart, lacking unwanted plant-derived polymers that are commonly present in traditional

plant based cellulose sources (20). Fig.1.12. shows the synthesis of bacterial nanocellulose (BNC).

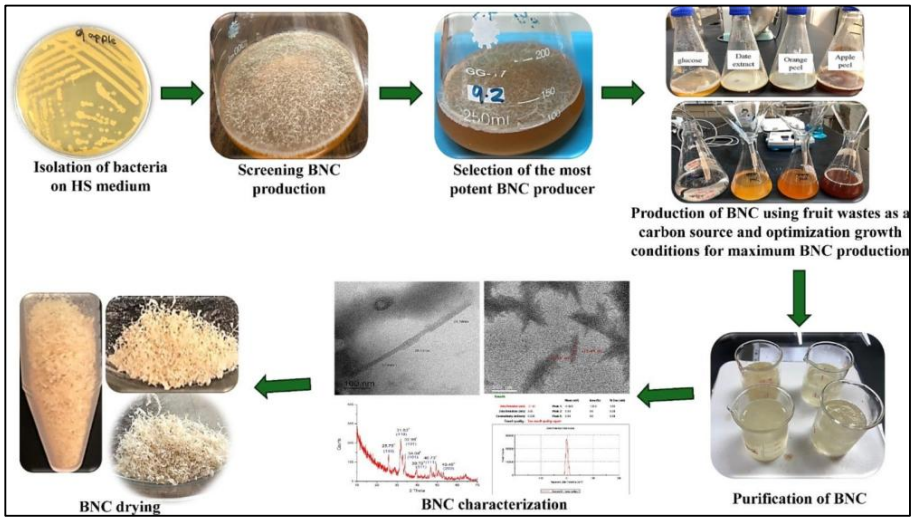

Fig.1.12: Synthesis of Bacterial nanocellulose (BNC)

1.4.2.4 Electrospun Cellulose Nanofiber (ECN)

Ultrathin fibers having diameters in the nanometer ranges can be produced using electrospinning. Electrospun fibers can be engineered according to our interest so that it can be used in many applications. Electrospinning is a method of electro hydrodynamics whereby an electrified liquid droplet experiences elongation into a filament via jet formation, followed by later stretching and elongation. This technique utilizes high voltage on a solution or a melt of polymer; the resultant charged jet elongates and solidifies during the travel toward a collector. The fiber has large surface area-to-volume ratios with directional porosity and better mechanical properties(25). Fig.1.13. shows the synthesis of electro spun cellulose nanofibers (ECN).

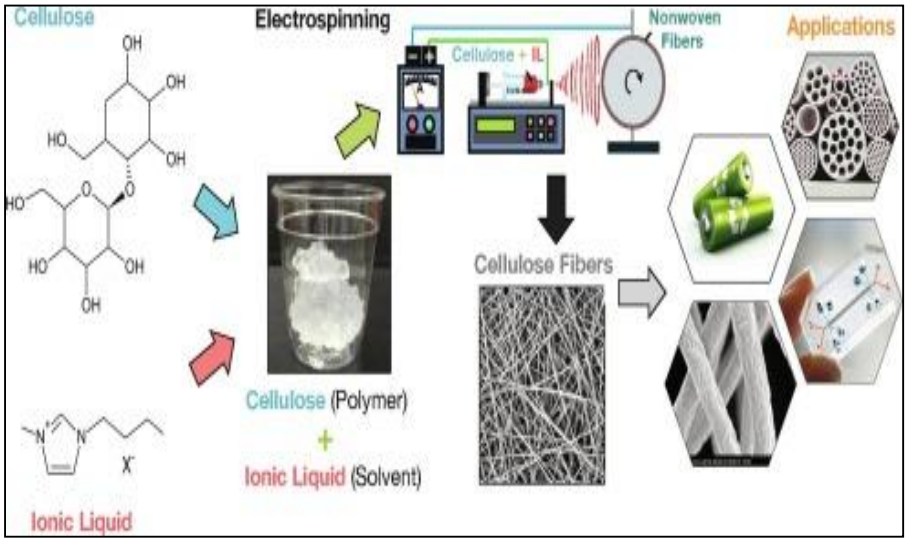

Fig.1.13: Synthesis of Electrospun Cellulose nanofibres (ECN)(26)

1.5 THE SOURCE - COTTON CLOTH

Cotton (*Gossypium spp*.) is a smooth, fine textured, renewable natural fiber obtained from seed hairs of the cotton plant Fig.1.14 (a), which is a member of the *Malvaceae family*. Cotton plants are cultivated on a large scale in tropical and subtropical regions around the world, including China, United States, India, Pakistan, Uzbekistan, Brazil, and Turkey(27). It is known as *Paruthi* (Tamil), *Paruthi Panju* (Malayalam), *Kapus* (Marathi) and *Kapda* (Hindi). On an average about 250-300 grams of cotton fibres are obtained from a single cotton plant. It also contains cottonseed which is further utilised for oil extraction and cattle feed, which in turn reduces agricultural waste. In India, which is known as the largest cotton growing country, yields about 26% of the world's total cotton. Its yield per acre is very low even though India has the largest cotton cultivation area in the world. Countries like the USA and China have higher yields in comparison to India.

The highly exceptional qualities of cotton such as high moisture absorbency, biodegradability, soft and comfortable texture, breathability, high tensile strength and high cellulose content have increased its utility over the years (28). Being a biodegradable and sustainable fiber, it is preferred over synthetic alternatives with respect to environmental conservation. It is widely used in textile industries for producing garments, fabrics, home textiles like bed sheets, towels, curtains, blended with other fibres to enhance strength, durability and cost effectiveness. Its hydrophilic nature makes it comfortable to wear as it absorbs sweat from human body and can release in the surface (29). It is a main component in medical and hygiene products such as bandages, sanitary napkins, cotton swabs, gauze and surgical dressings due to its hypoallergenic nature and high absorbency. It has other industrial applications as well, like its use in ropes, nets, tents, tarpaulins, canvas materials, paper and cardboard. Cotton derived nanocellulose find applications in the fields of material science, biomedical engineering, pharmaceutical, cosmetics, food and packaging(30). Cellulose nanocrystals and nanofibers, which have applications in biomedical fields, composites, food packaging and wastewater treatment, use cotton as their raw material. The textile sector worldwide generates large amounts of post-consumer cotton garments and industrial textile waste, out of which a large portion is either incinerated or dumped into landfills (31), thereby creating serious environmental concerns such as soil contamination, greenhouse gas emissions and growing waste management burdens. The main composition of cotton fibres is cellulose, with minor amounts of other non-cellulosic materials such as hemicellulose, pectin, proteins, waxes, and inorganic materials. Waste cotton clothes Fig.1.14 (b) contain cellulose over 90%, which shows their ability to serve as a promising raw material for synthesising CNCs. Cotton textile waste is treated as an agro-industrial

residue. It can be recycled and repurposed efficiently through many innovative approaches to enhance sustainability.

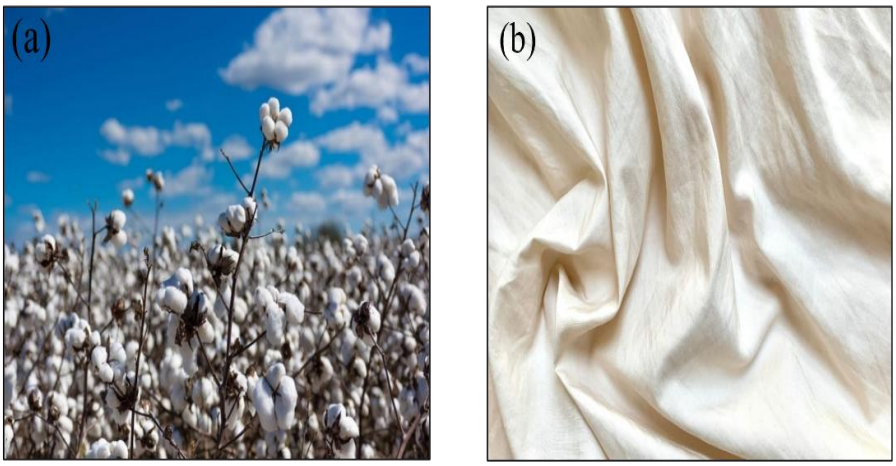

Fig.1.14: (a) Cotton plant (b) cotton cloth

1.6 DIFFERENT METHODS USED FOR THE EXTRACTION OF NANOCELLULOSE FROM CELLULOSE

1.6.1 Acid Hydrolysis

The acid-hydrolysis process of cellulose transforms it into smaller sugars, mainly glucose. It involves the cleavage of β-1, 4-glucosidic bonds of cellulose chains so that shorter chain fragments are created. Hydrolysis is generally influenced by the presence of other factors such as acid concentration, temperature, and the kinds of structural forms of cellulose. Acid concentration is the most influential because it constitutes a very crucial factor for the kinetics and mode of hydrolysis. For example, the sulfuric acid amounts are varied considerably, depending on the procedure for hydrolysis.

There is another factor, namely temperature, which brings about different rates of occurrences and yield during hydrolysis. Cellulose degrades into oligosaccharides and is further hydrolysed to glucose at higher

temperatures, about 30°C, in 40% hydrochloric acid. An important aspect is the cellulose acid complex formation during acid hydrolysis, caused by the partial destruction of the crystalline structure of cellulose when concentrated acid reaches the level of effective hydrolysis. Kinetics of acid hydrolysis are complex and have been widely studied. Many kinetic equations have been proposed to describe hydrolysis of cellulose and oligosaccharides in different conditions. Chemical modifications include acid hydrolysis decreasing the average degree of polymerization of cellulose and introducing reactive groups in the polymer to improve the reducing power of cellulose. Acid hydrolysis of cellulose is a complex process. It will help in several applications, such as biofuel production (32). Fig 1.15 represents the acid hydrolysis of cellulose.

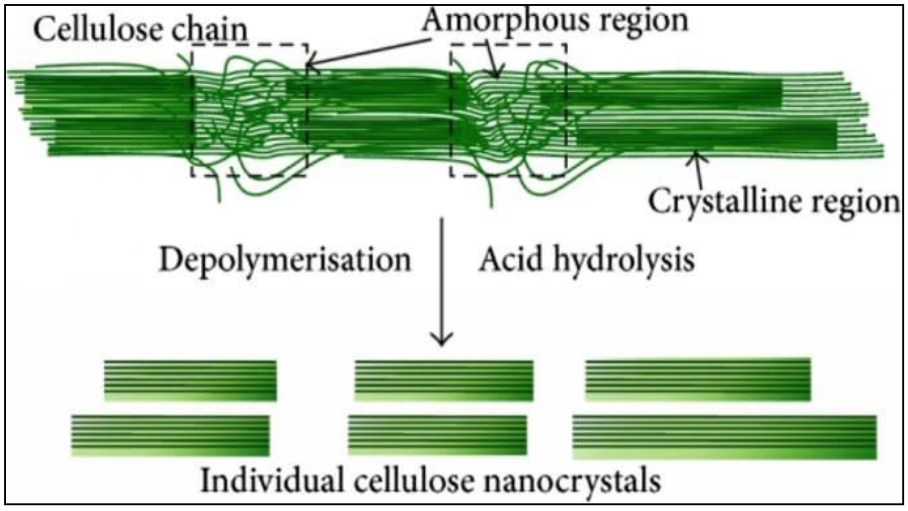

Fig.1.15: Acid hydrolysis of Cellulose yielding Cellulose nanocrystals (CNCs) (33)

1.6.2 Enzymatic Hydrolysis

Enzymatic hydrolysis breaks down cellulose at the solid-liquid interface through a series of steps. The process is largely carried out by enzymes known as glycosyl hydrolases, of which endoglucanases and exoglucanases /cellobiohydrolases are primarily included. These enzymes, collectively, degrade cellulose to smaller soluble products such as cellobiose and cellulooligosaccharides, which are further hydrolysed by β-glucosidase to glucose. The physical properties of cellulose and the unique modes of action of enzymes influence enzymatic hydrolysis efficiency. Increasing enzyme access and activity involve factors such as biomass pretreatment, which alters the physical and chemical properties of cellulose. The physical properties of cellulose and the unique manner in which enzyme acts, influence the efficiency of enzymatic hydrolysis. Increasing enzyme accessibility and activity necessitates conditions such as biomass pretreatment, which alters the physical and chemical properties of cellulose (34). Schematic representation of enzymatic hydrolysis is as shown in Fig.1.16

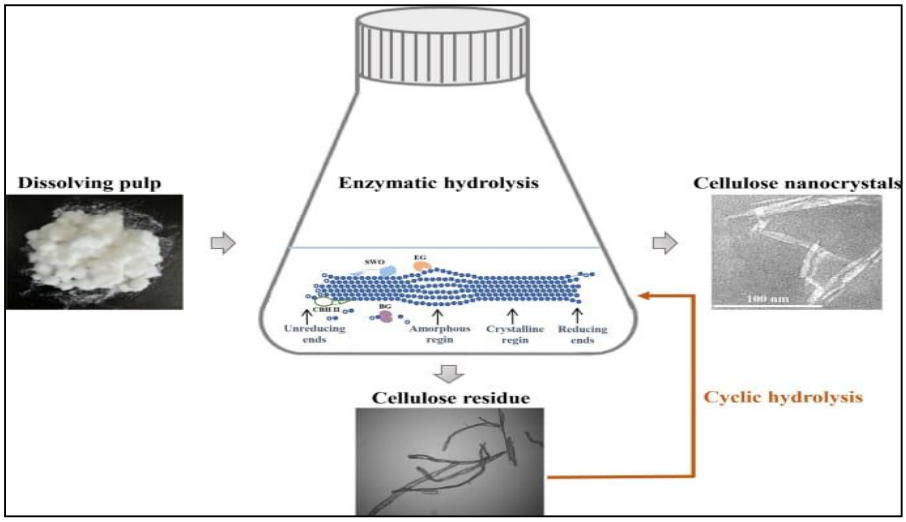

Fig.1.16: Enzymatic Hydrolysis(35)

1.6.3 Mechanical Process

Mechanical pretreatments, as shown in Fig.1.17. as a step in cellulose modification before acid hydrolysis in the synthesis route, are therefore of great significance as this leads to effective and efficient strategies for CNCs production. Researchers have evaluated various mechanical pretreatment techniques (magnetic agitation, milling, and blending). Magnetic agitation simplifies the procedure by agitating cellulose in solution to promote its surface area. Milling enhances the interaction between fibers and the acidic solution by processing the cellulose fibers. Grinding cellulose by a colloidal mill is a process allowing, by varying the intensity of the grinding, to produce the desired fiber size reductions and surface areas.

Mechanical pretreatment affects the final characteristics of CNCs. These are the crystallinity index, total mass yield and particle size distribution. Crystallinity index determines CNC stability and properties. Total mass yield reflects how much CNC is made by individual pretreatments. Particle size distribution is also dependent on the pretreatment method. Mechanical pretreatments can decrease production costs due to ability for large-scale production and comparison with intensive approaches. Alternative simpler approaches, like magnetic agitation or homogenization, are able to generate CNC with acceptable properties. Additional studies are needed on the investigation of more severe mechanical pretreatment that may help decrease the hydrolysis time, temperature, or acid concentration. Mechanical pretreatments are critical for CNC production, as they control hydrolysis and modify cellulose properties to allow efficient and low-cost production processes (36).

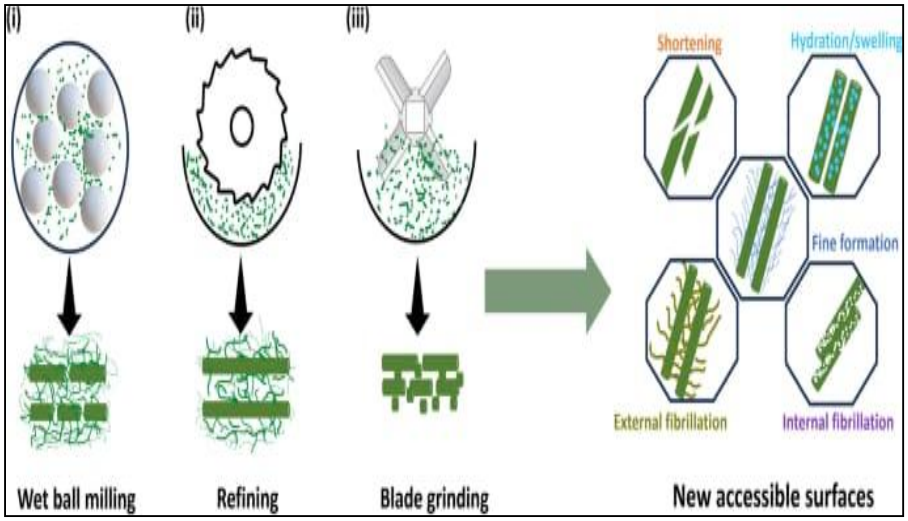

Fig.1.17: Mechanical Treatment of Cellulose

1.7 PRETREATMENTS FOR THE EXTRACTION OF NANOCELLULOSE

Lignocellulosic biomass converted into cellulose is carried out using several pre-treatments. Breakdown of cell walls and internal tissues of the lignocellulosic biomass can be carried out using pre-treatments as shown in Fig.1.18

The pretreatments can be divided into three types:

i. Physical pre-treatments: Physical pre-treatments for cellulose extraction includes sonication, pyrolysis, mechanical etc

ii. Biological pre-treatments: Non-cellulosic components like lignin and hemicellulose can be removed by biological pretreatments like enzymatic hydrolysis, bacterial treatment etc.

iii. Chemical pre-treatments: Cellulose can be extracted from raw material using two important chemical pre-treatments which are alkali pulping and bleaching. It helps in removing non-cellulosic components like pectin, lignin, hemicellulose etc., which are present along with cellulose (37).

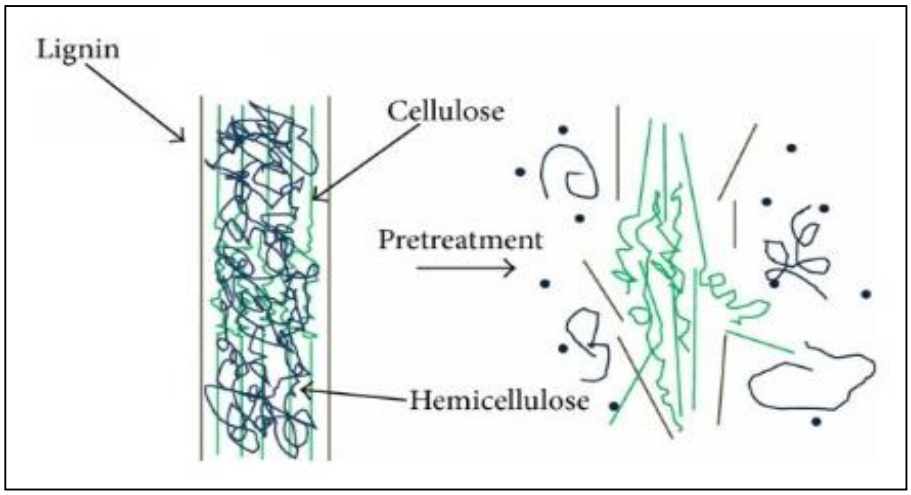

Fig.1.18: Effect of Pre-treatments

1.7.1 Alkali Pulping

Alkali pulping dissolves and disintegrates non-cellulosic components by encroaching into the crystallites to devastate the inter and intra molecular hydrogen bonds in the cellulose molecules and the nearby crystalline area. The non-cellulosic component hemicellulose is an amorphous entity and it is less densely packed when compared to the crystalline region. So, the removal of such amorphous entities increases the degree of crystallinity and the crystallinity index. In most cases sodium hydroxide is used to impregnate the fiber matrix during the alkali pulping process. This process causes the solvation and depolymerization of lignocellulosic materials by breaking the hydrogen bonds that keep cellulose macromolecules together and in combination with other constituents (37).

1.7.2 Bleaching

Bleaching is carried out in the alkali treated sample to remove the noncellulosic component lignin. Delignification is performed to increase the wettability between the polymer matrix and the natural fiber. Important bleaching agents are hydrogen peroxide (H_2O_2), sodium chlorite ($NaClO_2$), sodium hypochlorite (NaClO) etc. Due to the presence of lignin, the alkali pulped sample appears to be brown in colour. After bleaching, the cellulose appears to be white in colour whereas the bleaching solution appears to be bright yellow colour(37). Nagarajan et al. (38) carried out bleaching using 0.7 – 2 wt.% of sodium chlorite as bleaching agent and acetic acid (CH_3COOH) as buffer solution. Mueller et.al (39) used H_2O_2 (1.3% w/w) and acetic acid (0.1% v/v) for bleaching the alkali pulped sample.

1.8 ACID HYDROLYSIS

The distortions in the amorphous structure makes it more prone to acid hydrolysis when compared to crystalline region. The temperature of the reaction, concentration and type of acid used etc., are the important parameters that influence acid hydrolysis(37). Fig.1.19. shows the schematic diagram for the isolation of cellulose nanocrystals from waste biomass. There are two types of acid hydrolysis:

1.8.1 Mineral Acid Hydrolysis

Sulphuric acid, hydrochloric acid etc. are the important mineral acids used for acid hydrolysis. It is used for the isolation of nanocellulose from cellulose. Small size, high crystallinity, high thermal stability etc. makes it stand apart. Sulphuric acid is commonly used for acid hydrolysis (37). Morán et al. (40) used sulphuric acid to isolate nanocellulose from sisal

fibres. Camarero Espinosa et al. (41) used phosphoric acid to isolate nanocellulose from cotton pulp.

1.8.2 Organic Acid Hydrolysis

Use of organic acid in hydrolysis render less toxicity, high thermal stability, high crystallinity etc. (37). Sun et al. (42) carried out the hydrolysis of cellulose using 78.22 wt.% of formic acid, 17.78 wt.% of water and 4 wt.% of hydrochloric acid. Mosier et al.(43) carried out acid hydrolysis using 200 mL of 50 mM of maleic acid and sulphuric acid for hydrolysing cellobiose and microcrystalline cellulose (Avicel).

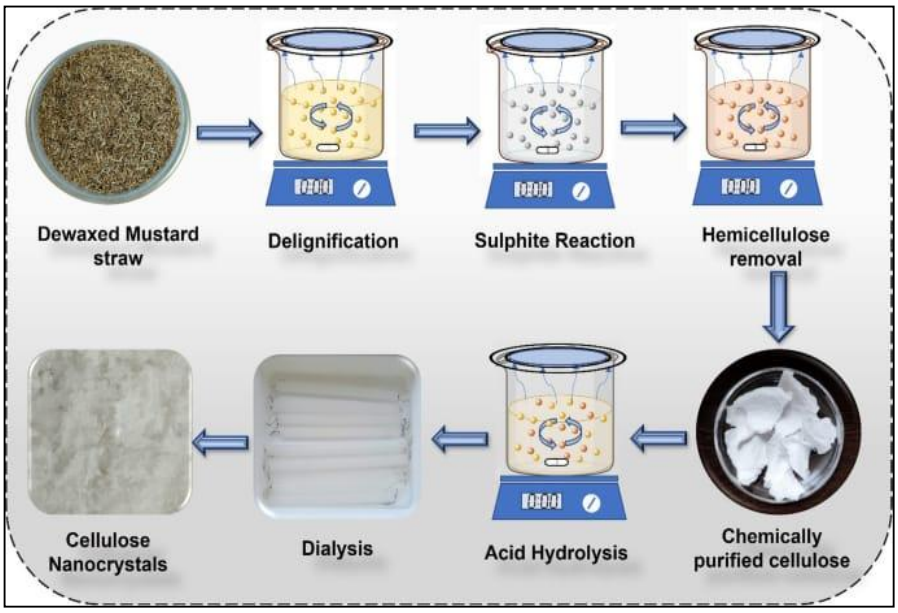

Fig.1.19: Isolation of Cellulose nanocrystals (CNCs) from waste biomass

1.9 APPLICATIONS OF NANOCELLULOSE

Nanocellulose is typically obtained from waste materials, thereby making it a valuable asset in sustainable waste treatment (23). Fig.1.20. shows the different applications of nanocellulose.

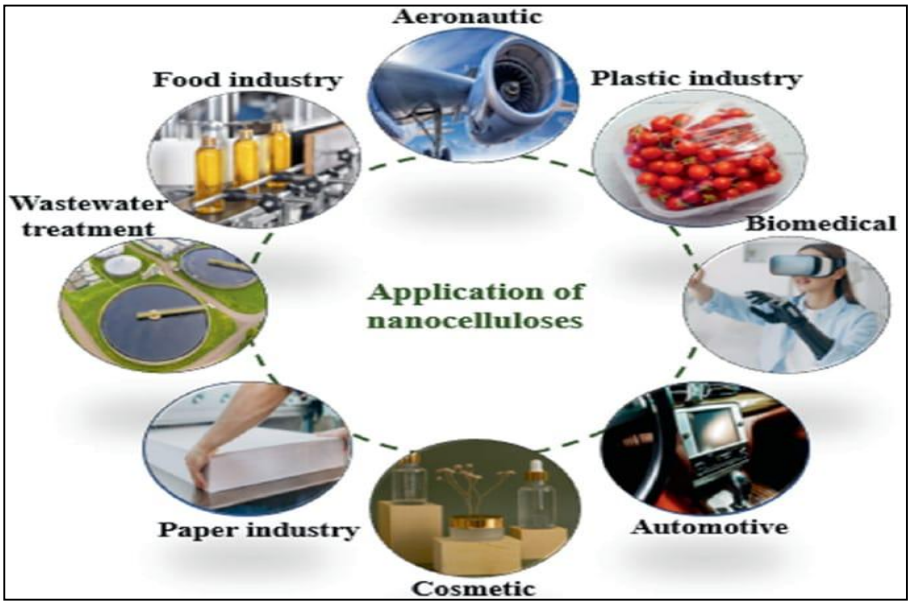

Fig.1.20: Application of Nanocellulose

1.9.1 Food Packaging Industry

Cellulose nanofibers (CNF) exhibit good barrier properties due to its fibril structure. It limits the intrusion as well as dispersion of liquid and gaseous materials into cellulose-derived composite films improving the barrier properties of nanocellulose (23). Not only oxygen barrier properties, but also water vapour barrier properties of bio-based food packaging materials are enhanced by the inclusion of cellulose nanocrystals (CNC) (44).

1.9.2 Biomedical Applications

Nanocellulose is used as a matrix for synthesizing metal nanoparticles like gold (45), silver (46), platinum (47), palladium (48) nanoparticles etc. exhibit efficient antimicrobial activity. Hydrogels based on nanocellulose can be used as drug carriers (23), (49). Membranes made of nanocellulose are used for wound-dressing. The cellulose nanocrystals can be bestowed with sensing properties by incorporating sensing nanomaterials into the hydroxyl groups present on the surface of cellulose nanocrystals (CNC). This highlights their potential to be used as biosensors (44).

1.9.3 Reinforcement in Polymer Matrix

The incorporation of fillers into polymer matrix composites reinforce their thermal as well as mechanical properties. In today's context attempts are made to replace where non-biodegradable artificial fillers with biodegradable natural fillers to achieve sustainable development and the current focus is on cellulose, chitin, starch etc. Cellulose in nano dimension has enhanced properties when compared to cellulose which include elevated thermal, mechanical and augmented hydrogen bonding which makes it ideal to use as a filler in polymer matrix (23), (50), (51). The dispersion of cellulose nanocrystals (CNC) in polylactic acid (PLA) matrix can be increased by adding polyvinyl alcohol (PVA) as a surfactant (44).

1.9.4 Water Purification

The nanocellulose membranes are highly selective. It is coupled with filters and is used to remove harmful contaminants from drinking water like pesticides, dyes etc. Ionic surface-active groups like anionic and cationic groups as well as non-ionic surface-active groups present in nanocellulose

are employed to adsorb and remove natural as well as heavy metal impurities from aqueous solutions (23).

1.9.5 Papermaking Industry

High crystallinity, high surface area, increased amount of hydrogen bonds etc. makes cellulose nanocrystals a promising factor in papermaking industry. But they also have water retention properties restraining its potential as paper coating materials (44). Inclusion of electro-conductive fillers like carbon nanotubes (CNT) can be leveraged to produce electro-conductive cellulosic papers (44).

1.9.6 Food Additives

Cellulose can be used as an emulsifier, stabilizer, thickener etc. because it doesn't affect the food quality. Cellulose nanocrystals (CNC) forms "Pickering emulsions" because the solid particles aggregate at the oil-water interface. Therefore, it can be used as stabilizers (44).

1.9.7 Catalysis

Cellulose nanocrystals (CNC) have high surface area and plenty of electron-rich hydroxyl groups (44). So, they can be used in many sustainable applications like cellulose nanocrystals supported palladium where it can be used as a recyclable catalyst (52), fabrication of cellulose nanocrystals (CNC) supported stable Fe (0) nanoparticles (53) etc.

.1.10 QUANTUM DOTS

Quantum dots are extremely tiny semiconductor particles, usually measuring between one to ten nanometers in diameter. Because of their minute size, quantum dots have special characteristics that result from

quantum confinement effects, in which electron and photon behaviour is controlled by quantum mechanics instead of classical physics. Quantum dots are extremely sought for a variety of applications in photonics, electronics, and nanotechnology because of their size-dependent behaviour, which is essential to their tunable optical and electrical properties. Quantum dots (QDs') properties fall in between those of bulk semiconductors and discrete atoms or molecules. Their size and shape have an impact on their optoelectronic properties. One of the most Important optical properties of colloidal quantum dots is colour (54).

1.10.1 TYPES OF QUANTUM DOTS

Quantum dots are commonly categorized based on their elemental constitution as:

1. II – VI QUANTUM DOTS
2. III – V QUANTUM DOTS
3. IV – VI QUANTUM DOTS
4. SILICON QUANTUM DOTS
5. GRAPHENE QUANTUM DOTS
6. CARBON DOTS

1.10.2 CARBON DOTS

Carbon dots, sometimes referred to as, CD, are semiconductor nanocrystals that possess a number of unique characteristics. Usually, they are very small carbon nanoparticles. CDs are typically described as quasi-0D carbon-based materials with intrinsic fluorescence that are less than 10 nm. When single-walled carbon nanotubes were being purified, these particles were inadvertently created. They have been employed extensively in a growing variety of industries in recent years. Compared to traditional semiconductor

quantum dots and organic dyes, photoluminescent carbon-based quantum dots have better qualities such high solubility, strong chemical inertness, simplicity of modification, and excellent resistance to photobleaching (55). Compared to other carbon-based nanomaterials, CDs are more cost-efficient, have a larger effective surface area, better charge transferability, higher electroconductivity, and less toxicity. A variety of functional groups, including amino, carboxyl, and hydroxyl, can be added to the surface of carbon dots to change their characteristics, increase stability, permit particular interactions, and improve biocompatibility. CDs have excellent electrical conductivity when employed as electrocatalysts in electrocatalytic reactions. Because of their exceptional optical qualities, fluorescent CDs have found extensive usage in a variety of medical applications, especially in the fields of biosensing, bioimaging, and therapeutic development (55).

1.11 ANTIOXIDANT ANALYSIS

Free radicals, also known as ROS, are substances or small particles with unpaired electrons in their atomic or molecular orbitals. They also include a variety of oxygen species, including hydrogen peroxide, a potent oxidizing functional group that cells produce during respiration and cell-mediated immune responses. Free radicals are given electrons by antioxidants, which neutralize them and make them appear innocuous, reducing oxidative damage to biological processes. Oxidative stress is the main factor linked to free radicals. Oxygen reacts with certain chemicals to produce free radicals, which can then interact with vital biological components like proteins and DNA to cause harm as well as damage to the cell membrane. By reacting with free radicals, antioxidants can neutralize them and prevent harm before it begins. Among the different types of secondary metabolites that plants produce are antioxidants. An interaction

with an antioxidant can produce a stable DPPH (2, 2-diphenyl-1-hydrazine) complex along with a colour shift from purple to bright yellow. DPPH (2, 2-diphenyl-1-picrylhydrazyl) radicals are stable free radicals because a single pair of electrons on the N atom is surrounded by three benzene rings. The outcome showed that CDs have scavenging activity against DPPH. It is possible for carbon dots to scavenge free radicals. Additionally, it has been demonstrated that when exposed to visible and/or ultraviolet light, some forms of C-dots act as oxidizing agents. Particularly fascinating is this dual oxidant-antioxidant nature, which is intimately associated with the C-dot characteristics (56).

1.12 OBJECTIVES

•Extraction of cellulose and removal of non-cellulosic components from waste cotton cloth through alkali pulping and bleaching.

•Synthesis of cellulose nanocrystals from cellulose by acid hydrolysis.

•Characterization of raw material, cellulose, and cellulose nanocrystals (CNC) using FTIR and XRD.

• Synthesis of carbon dots using cellulose as precursor via hydrothermal method.

•Characterization of carbon dots using UV, PL and TEM.

•Analysing the anti-oxidant activity of the synthesized carbon dots.

Chapter 2

Materials and Methods

2.1 INTRODUCTION

Waste cotton cloth, an abundant biomass resource, can be transformed into valuable nanomaterials like Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs). This chapter focuses on the sustainable synthesis of CNCs and CDs from waste cotton cloth and the potential application of CDs as a powerful antioxidant. The materials and experimental techniques used to create CDs and CNCs using waste cotton cloth as the raw material are thoroughly described.

2.2 PREPARATION OF CELLULOSE NANOCRYSTALS (CNCs) AND CARBON DOTS (CDs) FROM WASTE COTTON CLOTH

2.2.1 Chemicals Required

1. 10% NaOH.
2. 1:1 solution of NaOH (5%) and H_2O_2 (30%)
3. 10 ml of 45% H_2SO_4

2.2.2 Materials Required

1. Waste cotton cloth

2.2.3 Apparatus Required

1. Magnetic stirrer
2. Mechanical stirrer
3. Hot air oven
4. Sonicator

2.2.4 Synthesis of Cellulose Nanocrystals (CNC)

The procedure involves a sequential three stage methodology (57)

1) Alkali Pulping
2) Bleaching
3) Acid Hydrolysis

Waste cotton cloth was collected from household to maintain uniformity in the study. The cloth was thoroughly washed with distilled water and air dried before the procedure (Fig.2.1(a)). It was then cut into very small pieces as shown in Fig.2.1 (b). 10g of the waste cotton cloth was used for the synthesis of CNCs.

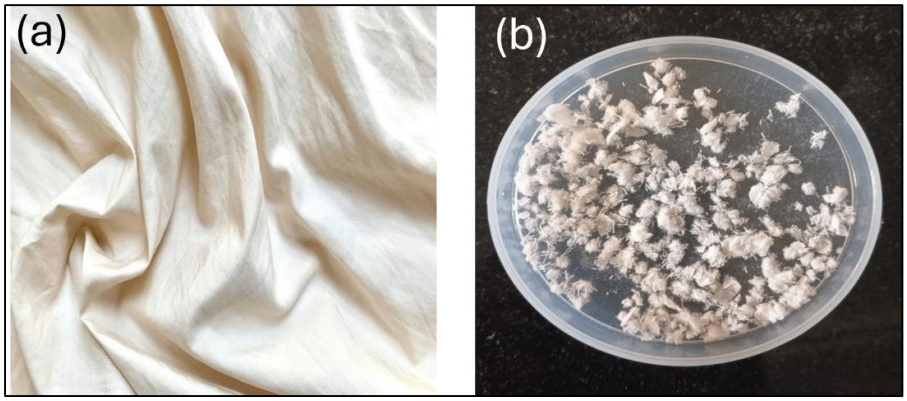

Fig.2.1:(a) Cotton Cloth (b) Cotton Pieces

2.2.4.1 ALKALI PULPING

About 50 g of NaOH was used for preparing 10 % NaOH solution. 10g of cotton pieces was treated with NaOH for 5 hours at 80°C with constant stirring using a mechanical stirrer. To maintain the pH, the alkali pulped fibers were washed with distilled water. It was then dried in an oven at 60ºC (Fig.2.2). After alkali pulping, hemicellulose, waxes, pectin, proteins, dyes, and dirt were removed.

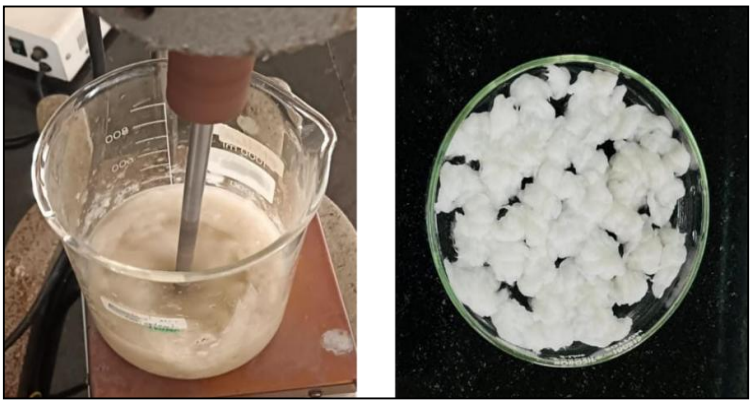

Fig.2.2: Alkali pulping

2.2.4.2 BLEACHING

The dried alkali pulped sample was treated with 400 ml 1:1 mixture of 5% NaOH and 30% Hydrogen peroxide at 50°C for 3 hours. The bleached sample was washed and dried in the oven at 60°C. (Fig.2.3). This step removes residual lignin and oxidizable impurities. Fig.2.4 represents the flowchart for the synthesis of cellulose from waste cotton cloth.

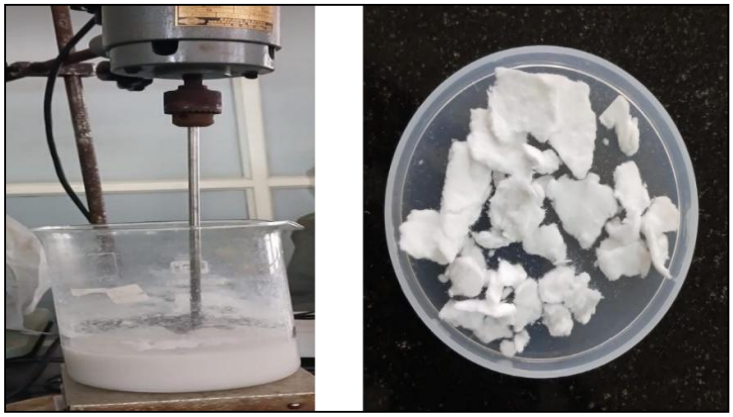

Fig.2.3: Bleaching

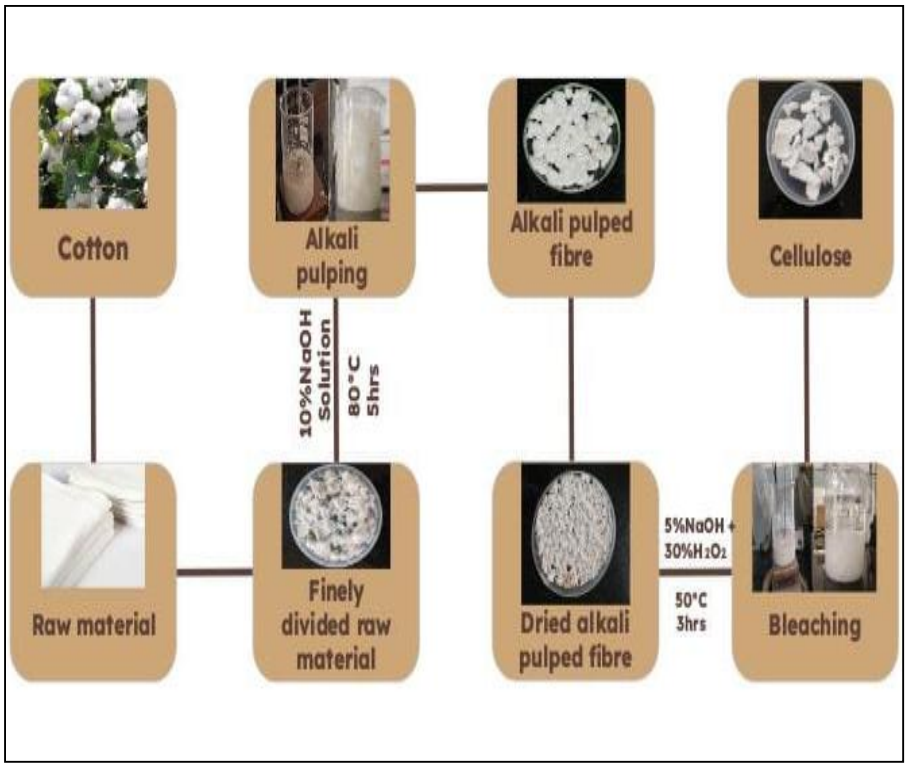

Fig.2.4: Flowchart representing the synthesis of cellulose from waste cotton cloth

2.2.4.3 ACID HYDROLYSIS

6 g of bleached alkali pulped fibres were treated with 120 ml of 45% sulphuric acid at 50°C for 3 hours. The fibers were then washed with distilled water until a neutral pH is obtained. The nanocrystals were obtained by centrifugation as shown in the Fig.2.5. This procedure eliminates amorphous cellulose and minor residual impurities.

Fig.2.5: Cellulose Nanocrystals

2.3 PREPARATION OF CARBON DOTS

Cellulose is a great source for synthesis of carbon dots as it is abundant, renewable, and eco-friendly. Its inherent qualities enable adjustable optical characteristics such as fluorescence, by altering the conditions of synthesis, making it an environmentally friendly substitute for conventional carbon-based sources in the production of CDs. Furthermore, the carbon dots can be altered by using the functional groups on the surface of cellulose. Properties of carbon dots like tunable emission, water solubility, biocompatibility, high photostability and low toxicity make it a sustainable and adaptable material for various applications.

2.3.1 Hydrothermal Method

Hydrothermal synthesis is a common method to make carbon dots (CDs) from cellulose, using different solvents and reaction conditions. In a closed system, hydrothermal synthesis transforms cellulose into carbon quantum dots by using aqueous media under high temperature (between 120 and 280°C), and pressure. This method is eco-friendly, affordable and simple to use, which makes it well-liked for cellulose- based CDs. In a closed system, hydrothermal synthesis converts cellulose into carbon quantum dots using water at high temperature and pressure, in a hydrothermal autoclave at 220 °C for 10 hours. {Formatting Citation}. Fig 2.6 shows the preparation of Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs) from cellulose.

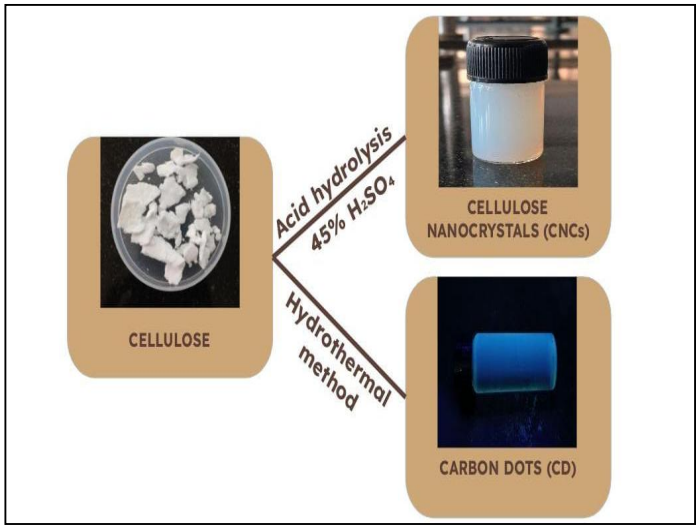

Fig.2.6: Flow chart showing the synthesis of Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs) from cellulose

2.4 ANTIOXIDANT ANALYSIS

The free radical scavenging activity of the sample was evaluated by assessing its ability to scavenge DPPH radicals. A DPPH reagent solution was prepared by mixing 100 µl of DPPH (1 mM) with 100 µl of methanol

(98%). Test solutions were prepared by adding varying volumes of the sample (T1=20 µl, T2=40 µl, T3=60 µl, T4=80 µl, T5=100 µl) to the DPPH reagent solution, adjusting the final volume to 300 µl. A control sample was prepared using solvent alone, and ascorbic acid (10mM) served as the standard. After 30 minutes of incubation in the dark at room temperature, the absorbance was recorded at 515 nm. Antioxidant activity was indicated by a colour change from purple to yellow or colourless, depending on the level of radical scavenging. The IC50 value was determined from the dose-response curve.

$$Radical\ scavenging\ activity\% = \left(\frac{control\ OD - test\ OD}{control\ OD}\right) X100$$

2.5 CHARACTERISATION TECHNIQUES

The raw material (cotton cloth), synthesised cellulose and cellulose nanocrystals (CNCs) were characterized using X- ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The prepared carbon dots (CD) were characterized using UV- visible spectroscopy, photoluminescence spectroscopy and Transmission electron microscopy (TEM)

2.5.1 X-Ray Diffraction (XRD)

X-ray diffraction is an analytical technique utilized to examine the framework of crystalline materials. In this process, sample is exposed with a monochromatic beam of X rays and these rays collide with the regularly spaced lattice planes within the crystal getting scattered in various directions. The angle at which the rays are scattered depends on the interplanar spacing and alignment of the planes. These scattered rays reinforce each other producing well defined peaks through constructive interference giving valuable informations about the arrangement and overall

structural properties. This X-ray diffraction pattern acts as a fingerprint of the material's periodic atomic arrangement. Diffractions can occur whenever the Bragg law is satisfied. The non-destructive nature of this technique makes it stand out from many other analytical methods. It is a quick and convenient process to perform, as it requires only minimal sample preparation and provides highly accurate d-spacing values, enabling real-time analysis and enhancing reliability. This allows characterisation of variety of material types, encompassing single crystal forms, polycrystalline materials and materials exhibiting partial amorphous behaviour. Powder XRD technique is employed with various important features and modern accessories, such as variable- temperature unit and a humidity chamber. These factors expand its range of applications by allowing researchers to study how moisture and temperature influence the properties of a material. Internal structure reflecting diffraction patterns are produced when X rays interact with a crystalline substance. Instead of a single crystal, powder XRD uses powdered sample making it more practical and convenient as it eliminates the need for growing or isolating individual crystals.(59)

2.5.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy is employed to identify the different functional groups present in the sample. In this technique, the absorption of infrared radiation is calculated as a function of wavelength or frequency, which are isolated using a grating or a prism. The energy corresponding to each individual frequency is detected by the detector and a well-defined spectrum representing intensity as a function of frequency or wavelength is produced by treating the collected data. FTIR can be applicable for both qualitative and quantitative analysis. Qualitatively, it

supports knowing molecular structure and recognizing compounds by evaluating the characteristic vibrational pattern of various chemical bonds. On the other hand, in quantitative analysis, the quantity of a specific substance can be measured by analysing the peak height or peak area in the spectrum, since these intensity values are directly related to the amount of substance present. IR radiation is transmitted through a sample in IR spectroscopy. The sample absorbs some of the infrared radiation and some of it is transversed. The spectrum that results shows the molecular transmission and absorption producing a molecular fingerprint of the sample. Just as no two fingerprints match, no two distinct molecular structures produce the same IR spectrum. This makes IR spectroscopy suitable for a variety of analysis. IR spectroscopy has proven to be a reliable technique for material analysis in the laboratory for over 70 years. A sample's IR spectrum is its fingerprint, with absorption peaks that corresponds to the frequencies of vibration between the bonds of the atoms composing the content. FTIR analysis was performed on the raw material, purified cellulose, and resulting CNC to ensure that non cellulosic impurities such as hemicellulose and lignin are efficiently removed, as well as that CNC was successfully isolated from cellulose. The infrared spectra were recorded within the mid-IR region, spanning a wavelength range of 4000-400 cm^{-1}. A spectral resolution of 4 cm^{-1} ensured clear identification of characteristic absorption bands. The data were collected in percentage transmittance (%T) units, allowing for comparison of functional group changes between raw material, cellulose, and CNC. This analysis provided clear evidence of the removal of unwanted components as well as the structural transformation caused by CNC extraction. (60)

2.5.3 Transmission Electron Microscopy (TEM)

Transmission electron microscopy is a highly advanced imaging technique widely used for studying the morphology, internal structure, and fine details of materials. In TEM, electrons are transmitted through an ultrathin specimen, enabling representation of features at the nanometer scale. A fundamental requirement for TEM analysis is the presence of a high vacuum within the instrument column. This vacuum minimizes electron scattering and maximises the mean free path of electrons, confirming that a sharp and high-contrast image of the specimen is obtained. During analysis, a high-energy electron beam is directed at a sample with a thickness of less than 100 nm. As the electrons interact with the specimen, they are focused and magnified by a series of electromagnetic lenses, resulting in detailed images. TEM's exceptional resolution allows it to reveal extremely fine structural features that other microscopy techniques cannot. Selected Area Electron Diffraction (SAED) is a technique that complements TEM for analysing the crystallographic structure of nanoscale materials. In SAED, a parallel beam of electrons is directed onto a specific region of an ultrathin sample, resulting in a diffraction pattern that reveals the material's crystal structure, phase identification, and degree of crystallinity. Preparing ultrathin samples yields some difficulty in sample preparation including radiation damages. TEM analysis is very expensive and is widely applied to characterise nanomaterials, polymers, metals and alloys, semiconductors, composite materials, and biological materials etc. (61)

2.5.4 UV-Visible spectroscopy

UV spectroscopy, also known as UV–visible spectrophotometry (UV–Vis or UV/Vis), is an analytical method that measures absorption or reflection in the visible and ultraviolet portions of the electromagnetic spectrum. It

will directly affect the apparent colour of materials because it operates with visible light. Electronic transitions occur in the atoms and molecules in this area. Because molecules with bonding or nonbonding electrons can absorb visible or UV light and drive these electrons into higher-energy antibonding molecular orbitals, absorption spectroscopy is supplementary to fluorescence spectroscopy. The wavelength of light absorbed is determined by the ease of excitation; electrons that are easily excited absorb longer wavelengths. In analytical chemistry, this method is frequently used to analyse a wide range of analytes, including biological macromolecules, highly conjugated chemical compounds, and transition metal ions. For instance, contact with ligands can change the hue of transition metal ions, which are typically coloured by d-electron transitions. A UV-visible spectrophotometer is the equipment utilized in this method. It calculates a sample's transmitted light intensity (I) and contrasts it with the initial intensity prior to the sample (I_0). The formula for the percentage transmittance (%T) is A = -log(%T/100).(62)

2.5.5 Photoluminescence Spectroscopy

Photoluminescence spectroscopy is an effective technique for characterizing materials, particularly carbon-based compounds like carbon dots (CDs). In order to move electrons into higher energy states, material must be excited by photons of a specific wavelength from a lamp or laser light. Luminescence is the term for the lower-energy photons that are released when electrons reach the base level. Photoluminescence spectra provide valuable insights into CDs' surface properties, quantum efficiency, and emission characteristics. Changes in surface functionalization, size, and chemical composition can be studied since the emission peaks in the spectra are caused by specific energy transitions in CDs. Quantum yield, a

measurement of the efficiency of light emission when excited, can also be evaluated using this method. In addition to emission efficiency, photoluminescence spectroscopy may be used to assess the photostability and stability of CDs under different environmental circumstances and to look at how functional groups and surface states impact their properties.

Chapter 3

Results and discussion

3.1 INTRODUCTION

This chapter focuses on the comprehensive characterisation and analysis of Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs) synthesized from cotton cloth. Characterisation techniques play a crucial role in understanding the chemical, structural, and morphological changes that occur during the conversion of raw materials into nanomaterials. X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) were employed to evaluate the structural and chemical properties of CNCs, while UV–visible spectroscopy, photoluminescence (PL), and transmission electron microscopy (TEM) were used to investigate the optical and morphological features of the synthesized carbon dots. The results provide insight into the physical and chemical transformations occurring throughout the synthesis process and confirm the successful formation of the nanomaterials. Furthermore, the antioxidant activity of the carbon dots was assessed to explore their potential applications**.**

3.2 CHARACTERISATION OF CELLULOSE NANOCRYSTALS

3.2.1 Physical Appearance

The physical appearance of the raw material, extracted cellulose and cellulose nanocrystals obtained from waste cotton cloth are shown in the Fig.3.1

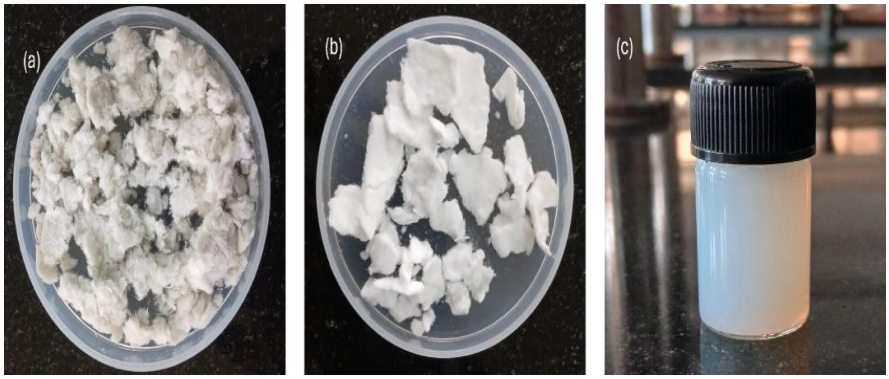

Fig.3.1: (a) raw material, (b) cellulose, (c) Cellulose Nanocrystal (CNCs)

The alkali pulping and bleaching using NaOH and Hydrogen peroxide changed the colour and texture of the raw material. The extracted cellulose was white in colour, which indicates the effective removal of non-cellulosic components like hemicellulose, lignin, pectin etc. The obtained CNC is also white in colour and perfectly crystalline in nature.

3.2.2 X-RAY DIFFRACTION (XRD)

The X-RAY diffraction pattern of the raw material, cellulose and cellulose nanocrystals are shown in Fig.3.2. This technique was used to analyse the structural and crystallographic changes that take place in the raw material during the chemical treatments like alkali pulping, bleaching and acid hydrolysis. In the raw material there is a broad and relatively low intense peak at 2θ =14.4° and 22.9°, which indicates the presence of amorphous content and non-cellulosic components such as lignin, pectin, hemicellulose and waxes. After alkali pulping and bleaching, we obtained cellulose, where peaks become more intense and sharper. Peaks at 2θ =14.4°, 22.9° and 34° corresponds to the crystallographic planes (110), (002) and (004) respectively, which indicates the cellulose I crystalline structure. CNC obtained after acid hydrolysis had even more sharper peaks, which indicates

the reduction in amorphous content and the effective removal of non-cellulosic components (59)

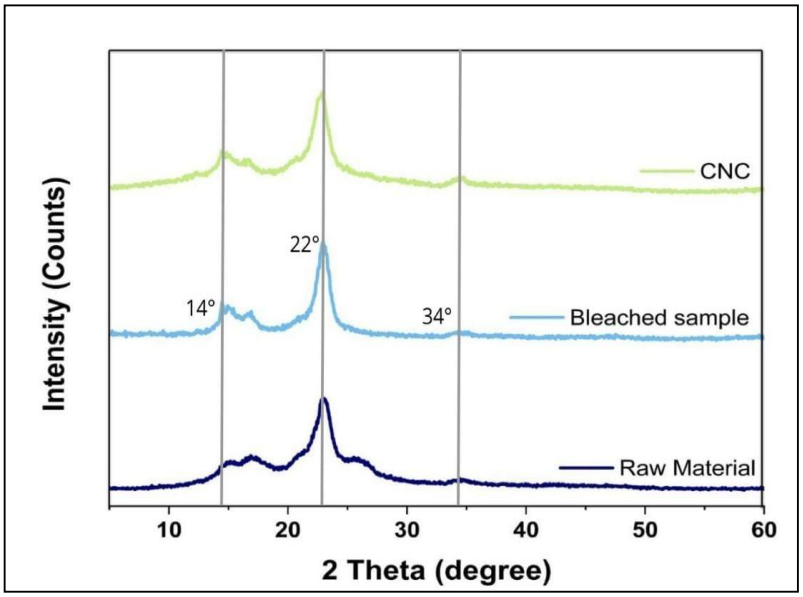

Fig.3.2 XRD spectra of raw material, extracted cellulose and Cellulose Nanocrystals (CNCs)

3.2.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was used to study the changes in functional groups present in raw material, cellulose and cellulose nanocrystals (CNC) after chemical treatments. The Fig.3.3 shows the FTIR spectrum of raw material, extracted cellulose and cellulose nanocrystal obtained during alkali pulping, bleaching and acid hydrolysis. The strong broad peak at 3415 cm^{-1} corresponds to the OH stretching vibration due to inter/intramolecular H bonding in cellulose.

The peak at 2900 cm^{-1} indicates CH stretching vibration. The peak observed at 1718 cm^{-1} shows C=O stretching of acetyl or ester group in hemicellulose and COOH in lignin. This peak disappears in the case of cellulose which

indicates the effective removal of hemicellulose and lignin. The peak observed at 1506 cm^{-1} represents the C=C aromatic stretching vibration in lignin which is absent in cellulose and which were removed after the bleaching yield cellulose. The peak at 1430, 1372, cm^{-1} are the characteristic peaks of cellulose which represent the CH_2 bending vibrations of the pyranose ring and OH bending vibration. From this graph we conclude the complete removal of non-cellulosic materials.(59)

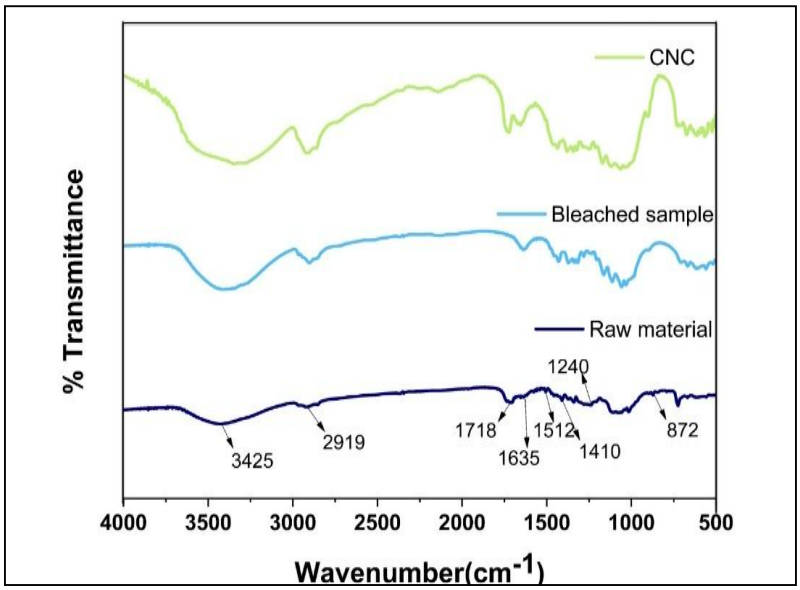

Fig.3.3 FTIR spectra of raw material, extracted cellulose and Cellulose Nanocrystals (CNCs)

3.3 CHARACTERIZATION TECHNIQUES OF CARBON DOTS

3.3.1 UV-Vis Spectroscopy

Carbon dots (CDs) exhibit a prominent absorption peak at 278 nm in their UV–Visible spectrum as shown in Fig.3.4. This peak originates from the π–π* transition of the aromatic C=C bonds, suggesting that the CDs contain graphitic structures or sp^2 hybridized carbon. (63) The formation of carbon

dots was confirmed by this characteristic peak. Around 340 nm, a small flat peak is also visible, which is associated with n–π* transitions of C=O groups on the CD surface.

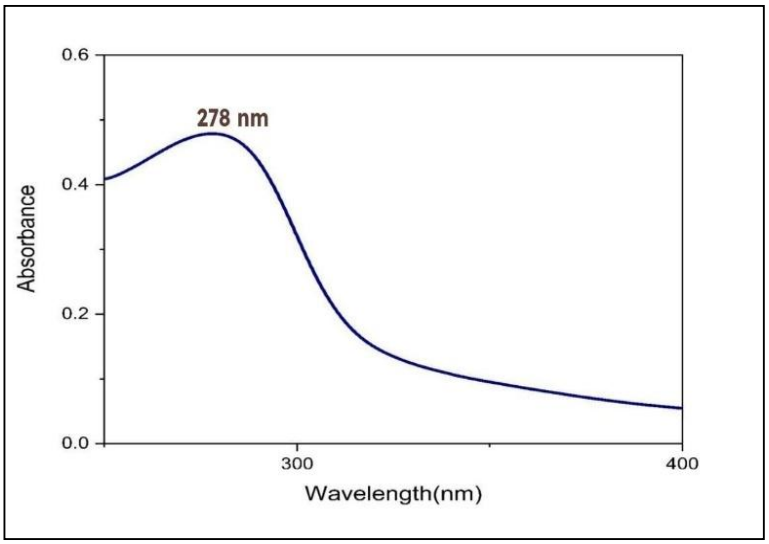

Fig.3.4: UV-Visible Spectroscopy

3.3.2 Photo Luminescence Spectroscopy

The photoluminescence (PL) spectra of carbon dots (CDs) is shown in Fig.3.5, which display the excitation-dependent emission behaviour. This indicates that when the excitation wavelength is changed, CDs emit different colours and intensities of light. Particle size variations as well as the existence of different surface functional groups and defect states are the reason behind this behaviour. (64) The spectra show multiple emission peaks with varying intensities according to the wavelength of excitation, which ranges from 270 nm up to 390 nm. Lower excitation wavelengths yield the highest intensity, demonstrating a robust UV fluorescence response. The highest emission intensity is obtained at an excitation wavelength of ~300nm. The extensive emission bands show the presence of multiple emissive states, which could be due to quantum confinement

effects, surface functional groups, or different sizes of carbon nanoparticles. The emission maxima exhibit a redshift as the excitation wavelength increases.

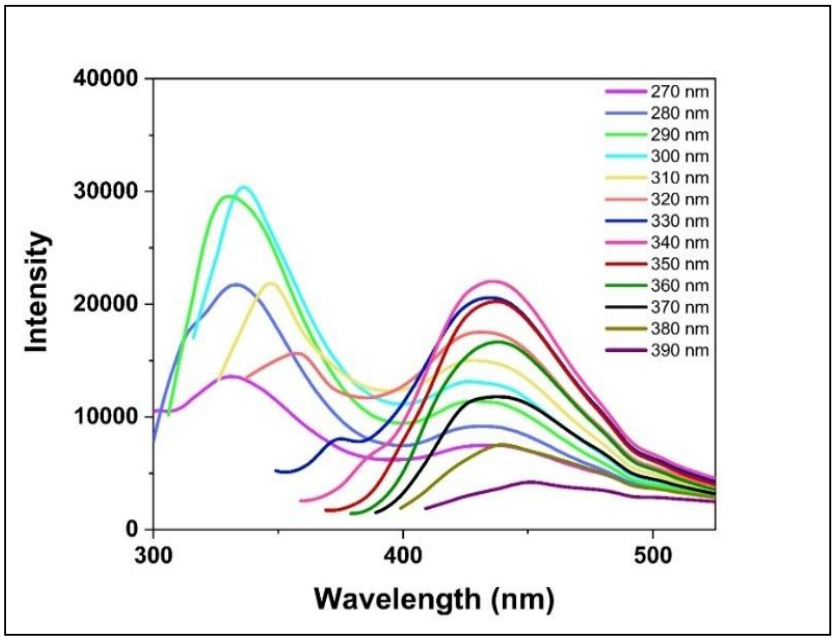

Fig.3.5: PL Spectra of Carbon Dots (CD)

3.3.3 Transmission Electron Microscopy (TEM)

TEM images of the carbon dots (CD) synthesised from cellulose extracted from cotton cloth is shown in Fig.3.6 Carbon dots are zero-dimensional nanomaterials with particle size of less than 10 nm. It acts as a promising building block for large carbon-based materials. CDs possess a core-shell structure, with the shells comprising diverse surface functional groups. (65)

The TEM image displays the morphology of the carbon dots, revealing that the particles are nearly spherical with a size less than 10 nm. The SAED pattern in Fig.3.6 shows a broad diffused ring- indicating that the synthesised carbon dots are amorphous in nature, lacking a well-defined crystalline structure.

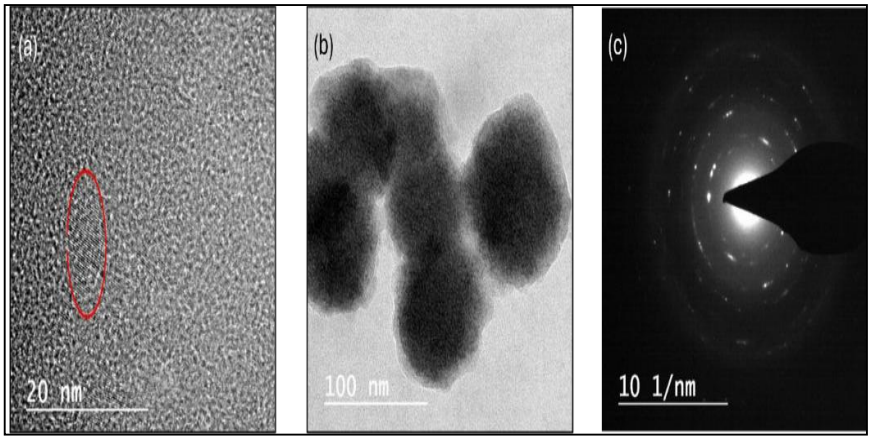

Fig.3.6:(a), (b) TEM images of CD and (c) SAED pattern of CD

3.4 APPLICATIONS OF CARBON DOTS

Carbon Dots (CDs) have gained popularity due to their excellent photoluminescence, high water solubility, biocompatibility, low toxicity, and ease of surface functionalization. These distinct properties make them useful in a wide range of applications, including bioimaging, sensing, catalysis, drug delivery, and environmental remediation. CDs have demonstrated remarkable antioxidant activity in these applications, scavenging free radicals and reactive oxygen species and thus reducing oxidative stress. Because of their antioxidant potential, they hold great promise for biomedical and therapeutic applications, such as protective against cellular damage and oxidative stress-related diseases.

3.4.1 ANTIOXIDANT ANALYSIS

$$Radical\ scavenging\ activity\% = \left(\frac{control\ OD - test\ OD}{control\ OD}\right) X100$$

Table 1: Percentage scavenging activity of DPPH by CD

Sample dose (µl)	$Radical\ scavenging\ activity\% = \left(\frac{control\ OD - test\ OD}{control\ OD}\right) X100$
20	33.85
40	59.31
60	89.02
80	90.64
100	92.60
Standard = 100 (10mM ascorbic acid)	93.13

Fig.3.7. Represents the radical scavenging activity of synthesised Carbon Dots (CD)

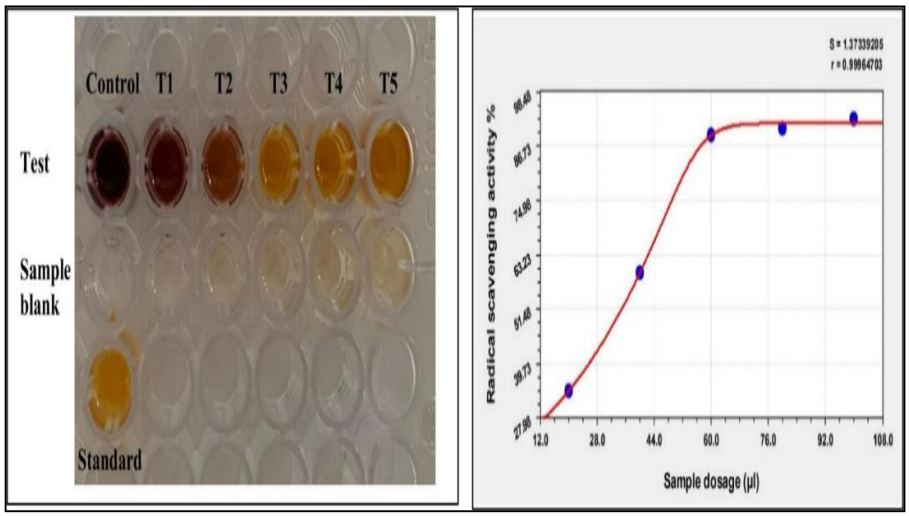

Fig.3.7: Radical scavenging activity of Carbon Dots (CD)

Percentage of radical scavenging activity = 92.606 %

The IC50 value is found to be 42.37

The radical scavenging activity of the synthesized carbon dots was assessed using the DPPH assay, which measures the antioxidant's ability to neutralize DPPH radicals through hydrogen or electron donation, resulting in a decrease in absorbance at 515 nm. Carbon dots demonstrated dose-dependent scavenging activity, ranging from 33.85% at 20 µL to 92.61% at 100 µL. At 40 µL, activity increased significantly by 59.31%, indicating efficient interaction with DPPH radicals. The IC_{50} value of 42.37µL indicates high antioxidant efficiency at a relatively low concentration. At 100 µL, the scavenging activity was comparable to that of ascorbic acid (93.13%), confirming the significant antioxidant potential of the synthesized carbon dots. The high radical scavenging activity is due to their small particle size, π-electron-rich carbon core, large surface area and abundant surface functional groups capable of donating hydrogen or electrons.

Chapter 4

Conclusions

The growing accumulation of organic, industrial, and textile waste poses serious environmental challenges and demands sustainable management strategies. The waste-to-wealth approach offers an effective solution by converting discarded materials into valuable products, thereby reducing pollution and conserving natural resources. Guided by the principles of green chemistry and the three R's - reduce, reuse, and recycle, this strategy promotes responsible resource utilization and environmental sustainability. The transformation of textile waste into extremely valuable nanomaterials such as Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs), highlights the potential of waste-derived products in advanced scientific and technological applications. Such practices not only minimize landfill burden but also create opportunities for innovation and economic value generation, demonstrating that waste can serve as a meaningful resource for sustainable development.

Cellulose Nanocrystals (CNCs) have been shown to possess significant potential as an eco-friendly material for applications in packaging, water purification, and healthcare. To produce CNCs, the cotton cloth was subjected to a systematic three-stage process that included alkali pulping, bleaching, and acid hydrolysis. The alkaline treatment eliminated hemicellulose, waxes, and other soluble impurities while bleaching further removed residual lignin to obtain pure cellulose. The amorphous portions

of the cellulose were then hydrolysed, leaving behind highly crystalline nanocellulose in the form of CNCs. The increase in crystallinity following acid hydrolysis was verified by X-ray diffraction (XRD) analysis. XRD analysis shows sharp and well-defined diffraction peaks, confirming the increased crystallinity and successful formation of CNCs. Effective cellulose purification was demonstrated by the disappearance of distinctive lignin and hemicellulose functional groups, as shown in Fourier-transform infrared spectroscopy (FTIR). The analysis confirms the removal of lignin and hemicellulose functional groups, indicating effective purification of cellulose.

Carbon Dots (CDs) exhibit diverse applications in catalysis, biomedicine, drug delivery, photocatalysis and sensing. CDs produced from waste sources are attractive due to their low-cost, non-toxicity, good water solubility, high biocompatibility, large surface area and strong mechanical properties. They have recently gained attention as promising alternatives to conventional fluorescent materials. Hydrothermal synthesis is a widely used technique for the preparation of Carbon dots (CDs). It enables precise control over reaction parameters including temperature, pressure, reaction time, and solvent choice. In a closed hydrothermal system, cellulose is transformed into CDs using water under high pressure and high temperatures ranging from 120°C to 280°C.

The UV−Visible absorption spectrum of the carbon dots synthesized from cotton cloth exhibits a prominent absorption peak at 278 nm, which is attributed to the $\pi \rightarrow \pi^*$ transition of C=C bonds present in the carbon core structure confirming the formation of CDs.

The PL spectra show clear excitation-dependent emission, with the strongest intensity at approximately 290-300 nm excitation. The emission

maximum red-shifts with increasing excitation indicating multiple emissive surface states and confirming the successful synthesis of highly fluorescent carbon dots.

TEM images display the morphology of the carbon dots, revealing that the particles are nearly spherical with a size of less than 10 nm.

The SAED pattern shows a broad, diffused ring, indicating that the synthesised carbon dots are amorphous in nature and lacks a well-defined crystalline structure. This confirms the successful formation of carbon nanoparticles.

The synthesised Carbon Dots exhibited significant antioxidant activity in the DPPH free radical scavenging assay, showing a dose-dependent increase in radical scavenging percentage from 33.85% at 20 µL to 92.60% at 100 µL. The calculated IC_{50} value of 42.37 µL indicates the concentration required to scavenge 50% of DPPH radicals. Compared to the standard ascorbic acid (93.13% at 100 µL), the sample demonstrates strong antioxidant potential, suggesting its effectiveness as a natural free radical scavenger.

In conclusion, this project presents a progressive strategy that integrates waste management with sustainable development through the synthesis of two high-value, functionally integrated materials - Cellulose Nanocrystals (CNCs) and Carbon Dots (CDs). This approach introduces a forward-thinking approach to sustainability by reducing waste generation while increasing value creation, thereby contributing to both environmental protection and economic growth.

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