PROJECT REPORT

On

DEVELOPMENT OF SUSTAINABLE CELLULOSE AEROGEL FROM CITRUS SINENSIS PEEL: PREPARATION CHARACTERIZATION AND DYE ADSORPTION EVALUATION

Submitted by

NIMITHA ANTONY (AB23CHE010)
BHADRA A (AB23CHE032)

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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ERNAKULAM

B.Sc. CHEMISTRY PROJECT REPORT

Name	: BHADRA A
Register Number	: AB23CHE032
Year of Work	: 2025-2026

This is to certify that the project **"DEVELOPMENT OF SUSTAINABLE CELLULOSE AEROGEL FROM CITRUS SINENSIS PEEL: PREPARATION CHARACTERIZATION AND DYE ADSORPTION EVALUATION'"** is the work done by **BHADRA A.**

Dr. Saritha Chandran A.
Head of the Department

Dr. Raichel Mary Lopez
Staff-member in charge

Submitted to the Examination of Bachelor's Degree in Chemistry

Date: 10/4/26

Examiners: SENJU DEVASSYKUTTY

:

DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

ST. TERESA'S COLLEGE (AUTONOMOUS)

ERNAKULAM

CERTIFICATE

This is to certify that the project work entitled "**DEVELOPMENT OF SUSTAINABLE CELLULOSE AEROGEL FROM CITRUS SINENSIS PEEL: PREPARATION, CHARACTERIZATION AND DYE ADSORPTION EVALUATION**" is the work done by **NIMITHA ANTONY AND BHADRA A** under my 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.

Dr. Raichel Mary Lopez

DECLARATION

I hereby declare that the project work entitled "**DEVELOPMENT OF SUSTAINABLE CELLULOSE AEROGEL FROM CITRUS SINENSIS PEEL: PREPARATION ,CHARACTERIZATION AND DYE ADSORPTION EVALUATION**" 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. RAICHEL MARY LOPEZ, Assistant professor,** 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.

NIMITHA ANTONY
BHADRA A

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Nimitha Antony

Bhadra A

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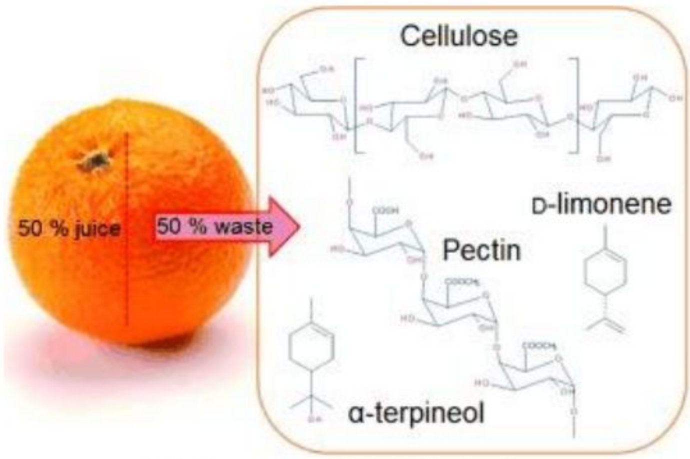
Chapter 1

Introduction

1.1 GLOBAL ORANGE PRODUCTION, WASTE, AND THE UPCYCLING SOLUTION



1.1 Orange Peel Waste Utilization

The production of orange (*Citrus sinensis*) was ca. 70 Mt in 2023. A considerable share (25–30%) of its production is routed to the juice industry, where 50 to 60% of the fruits are converted into by-products. When not landfilled, the orange waste is destined mainly for low added-value endings, such as incineration and cattle feeding. Upcycling food losses and waste in their integrity (or "bulk" waste) i.e., without extracting individual components into bio-based materials has emerged as a promising strategy within the Green Manufacturing and Engineering approach to reduce the use of harsh chemicals, waste generation, and save processing operations, time and energy. A way to valorise orange waste is to make porous 3D materials, which can find numerous potential applications in packaging, acoustic and thermal insulation, agriculture, etc [1].

Several residues after orange fruit processing are produced. Orange peel waste is the most representative fraction. Chemical characterization refers to different compounds present in the matrix of the residue. Orange peels are a rich source of soluble sugars and insoluble polysaccharides. Orange peel consists of sugars (30–40%), pectin (15–25%), cellulose (8–10%) and hemicellulose (5–7%). The most important orange peel waste components are pectin, cellulose, hemicellulose, lignin, protein, and fats. Nevertheless, components such as soluble sugars, terpenes, and phenolic compounds can be found [2,3].

1.2 AEROGELS: FROM TRADITIONAL TO BIO-BASED SUBSTITUTES

Aerogels constitute a special type of porous materials. They are open-pore nanostructured solids, with remarkably low densities (<0.2 g/cm^3) and high specific surface areas (>100 m^2/g). Classical (inorganic and synthetic polymer) aerogels are produced from gels, from which the solvent is removed in such a way that preserve the internal morphology. Bio-based aerogels (also called bio-aerogels) have been systematically investigated in the past two decades. Most of them are made from neat polysaccharides (e.g., starch, pectin, alginate, chitosan, cellulose, and its derivatives) and, sometimes, proteins [1].

Technologies with the potential to be improved by aerogel include those used in the areas of optoelectronics, adsorption catalysis, sound insulation, medical materials, aerospace materials, and many other fields. However, the mechanical properties of silica aerogels are poor, and the precursors of synthetic polymer-based aerogels are toxic and non-degradable [4].

1.3 CELLULOSE

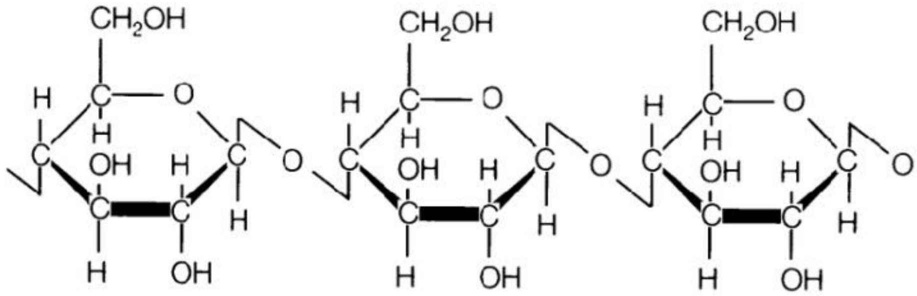


Fig 1.2 Structure of Cellulose

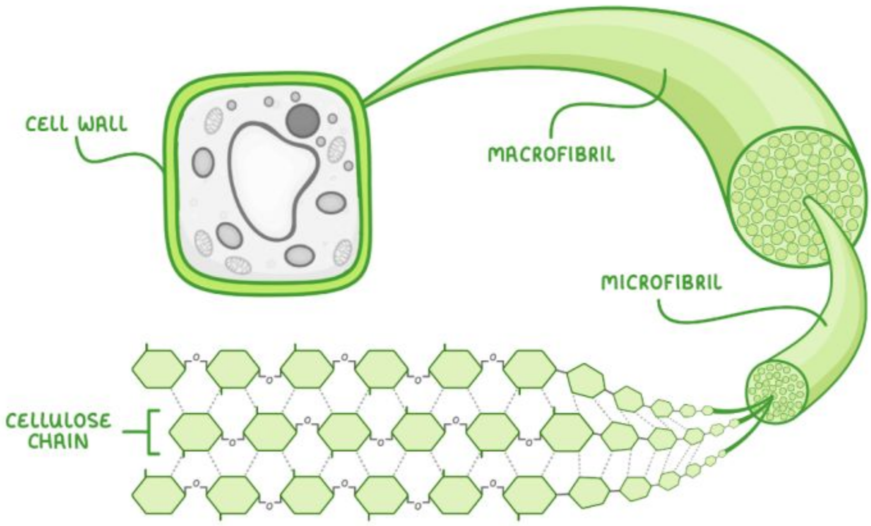


Fig 1.3 Cellulose in the Plant Cell Wall

Cellulose is a complex polysaccharide and the primary structural component of plant cell walls. It is the most abundant organic polymer on Earth making up a significant part of the dry weight of plants, especially in wood, cotton, and other fibrous plant materials. As a biopolymer, cellulose plays a crucial role in providing structural integrity and rigidity to plants, helping them maintain their shape and resist mechanical stress. Cellulose is made up of long chains of β-D-glucose molecules connected by β(1→4) glycosidic bonds, with hydroxyl groups at the C2, C3, and C6 positions. Its chemical formula is $(C_6H_{10}O_5)_n$, with the C4-OH group acting as the non-reducing end and the C1-OH group as the terminating end. These individual glucose units are called cellobiose, and the polymer chain can be very long, consisting of hundreds to thousands of glucose units. Four different types of cellulose crystallinity have been identified and are classified as Type I, II, III, and IV. The structure of cellulose is highly ordered, with each glucose unit rotated 180 degrees relative to the previous one, allowing the formation of a linear, straight chain. These long chains are aligned in parallel to each other, forming microfibrils, which are bundles of cellulose molecules held together by hydrogen bonds. These microfibrils are organized into larger structures and contribute to cellulose's high strength and rigidity [5].

1.4 CELLULOSE AEROGELS: A SUSTAINABLE AND MULTIFUNCTIONAL MATERIAL

Cellulose has many properties that are different from those of petroleum-based polymers, such as biocompatibility, biodegradability, thermal stability, chemical stability, and low cost. With advancing research on the physical and chemical properties of cellulose, environmentally-friendly functional cellulose-based materials, such as cellulose fibers, cellulose films, cellulose hydrogels, cellulose aerogels, and cellulose-based composites, have been developed. In particular, cellulose aerogels have the renewability, biocompatibility, and biodegradability of cellulose, while also having additional advantages such as low density, high porosity, and a large specific surface area, making it one of the most promising materials in the 21st century. Cellulose aerogel is a porous solid material. The specific surface area (10–975 m^2/g), porosity (84.0–99.9%), and density (0.0005–0.35 $g \cdot cm^{-3}$) of cellulose aerogels are comparable to those of traditional silica aerogels and synthetic polymer aerogels, but cellulose aerogels have a higher compressive strength (5.2 kPa–16.67 MPa) and better biodegradability. Therefore, cellulose aerogels are a type of environmentally-friendly and multi-functional new material that has great potential in the application of adsorption and oil/water separation, heat insulation, biomedical materials, metal nanoparticle/metal oxide carriers, the preparation of carbon aerogels, and many other areas [4].

1.5 SOURCES OF CELLULOSE

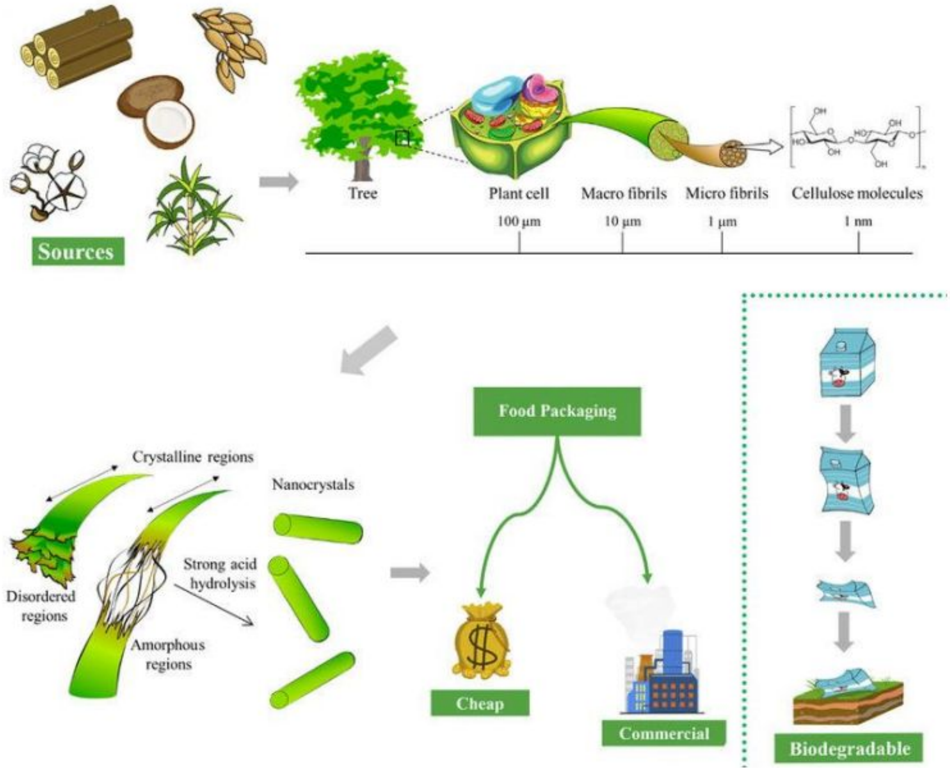


Fig.1.4 Sources and applications of cellulose

Cellulose can be acquired from a variety of sources including plants, algae, bacteria, and tunicates. However, different organisms produce cellulose of various levels of Degree of crystallinity (DC) which leads to drastically different mechanical properties and application possibilities. For example, as amorphous cellulose successfully prevents adhesion, it is effectively used as anti oil-fouling material for water oil separation, while Microcrystalloine cellulose (MCC) is applied in bio-composites, owing to its chemical inactivity and hygroscopicity Because of this, finding cheaper and more efficient ways to produce cellulose would have the potential of lowering the cost of such bio-composites. A wide variety of plant sources can be used for cellulose extraction, with the most important being wood and cotton; however, corn, banana, rice, aloe vera, and many others can serve the same purpose. Properties and structure of cellulose from plant sources depend on different parameters including climate conditions, soil type and characteristics, botanical origin amongst others. On an industrial level, it is a low-cost, high-volume, sustainable option, with a relatively simple method of extraction, but for non-industrial conditions, other sources of MCC may be easier to synthesize—the process of extracting cellulose from plant sources is complex and involves several steps suitable only for large-scale industrial operations such as physical break-down of the plant matter using processes such as grinding and homogenization [5].

Cellulose is insoluble in water and most common solvents; the poor solubility is attributed primarily to the strong intramolecular and intermolecular hydrogen bonding between the individual chains.

In spite of its poor solubility characteristics, cellulose is used in a wide range of applications including composites, netting, upholstery, coatings, packing, paper, etc. Chemical modification of cellulose is performed to improve process ability and to produce cellulose derivatives (cellulosic's) which can be tailored for specific industrial applications. Cellulosic's are in general strong, reproducible, recyclable and biocompatible, being used in various biomedical applications such as blood purification membranes and the like [6].

1.6 CELLULOSE DISSOLUTION FOR AEROGEL PREPARATION

N,N-dimethylacetamide (DMAc)/LiCl, N,N-dimethylformamide (DMF)/N2O4, dimethyl sulfoxide (DMSO), etc. have been found to dissolve cellulose well, but these solvents will cause environmental pollution. Ionic liquids have been used for a long time as cellulose solvents to prepare cellulose aerogels. In an ionic liquid system, there exists no gelation process, and the network structure of cellulose aerogels is formed through non-solvent induced phase separation. However, the high cost of ionic liquid has limited the industrialization of cellulose aerogels. NaOH/urea solution can rapidly dissolve cellulose at a low temperature and replace the ionic liquid owing to its low cost and no pollution. In current research, it is most widely used as a solvent to dissolve cellulose. The cellulose sol formed in NaOH/urea solution can be gelated by adding a cross-linking agent, adjusting pH, etc [7].

1.7 APPLICATION OF CELLULOSE AEROGEL:

1.7.1 Dye adsorption

Approximately 10% to 25% of dyes produced annually are discharged into surface water streams during manufacturing and processing operations, with textile industry accounting for nearly 2% of these dyes. Improper wastewater treatment and discharge without adequate management can lead to pollution and various health issues and wellness problems (e.g., eye burns, breathing difficulties, nausea, vomiting, and methemoglobinemia). Detoxifying toxic dyes from wastewater before discharge is therefore crucial and a very important aspect. Adsorption is widely employed due to its (i) effectiveness; (ii) versatility in removing different types of dyes; (iii) eco-friendliness; and (iv) absence of harmful substances or secondary contaminants. Natural and synthetic adsorbents, including carbon-based materials, metal oxides, bio-adsorbents, and polymer-based materials, are commonly used for dye removal.

Chemicals and dyes are major sources of the environmental contamination of effluent wastewater from various industries. Synthetic dyes, in particular, raise concerns due to their potential toxicity and negative effects on human life and ecosystems. Methylene blue, a cationic dye commonly used in textile manufacturing processes for dying cotton, wood, and silk, is a toxic and non-degradable dye that remains stable at elevated temperature, under light exposure, heat, and in the presence of other chemical reactions. Over the recent decades, several conventional techniques for the decolorization of water containing residual dyes have been reported, including physical methods like membrane filtration (nano-filtration, reverse osmosis, electrodialysis), coagulation/flocculation, and irradiation, as well as chemical and biological methods like advanced oxidation processes, electrochemical degradation or ozonation, and decolorization by aerobic or anaerobic degradation [8].

1.7.2 Cellulose as an adsorbent for dyes

Cellulose aerogels possess multifunctionality due to the reactivity of cellulose. There are three main methods for modifying them. They are currently used for thermal insulation, flame retardancy, and bio-applications, and can also be used in carbon aerogel production and the transport of metal nanoparticles/metal oxides. In the textile industry, they can be used for thermal protection, wound care, water purification, and carbon capture. However, challenges remain in some applications, such as avoiding the use of toxic crosslinking agents in water purification [9].

1.7.3 Cellulose as an adsorbent for oil spill contamination

Oily wastewater generated by human activities is a typical hazardous substance. Natural microfibrils/regenerated cellulose carbon aerogel (NM/RCA) exhibits superhydrophobicity/superoleophilicity, high compressibility, excellent elasticity, and superior fatigue resistance, demonstrating high efficiency in oil-water separation and the recovery of oil and organic solvents. It also shows broad potential applications in oil purification and pollutant remediation [10].

1.7.4 Biomedical applications of cellulose based aerogels

Cellulose-based aerogels have been also utilized in many regeneration studies to regenerate many tissues and organs, such as bone, skin and cartilage. Cellulose-based aerogels have been suggested to solve the issue of the potential complications associated with autografts since they have been widely used in tissue engineering scaffold preparations. Other medical applications have benefited from cellulose aerogels. The combination of polyethylenimine (PEI)-grafted cellulose nanofibril aerogels for drug delivery, and reported that the materials are promising and can be used as new generation controlled drug delivery carriers. 'The unique pH and temperature-responsiveness of cellulose-based aerogels, together with their excellent physical, chemical and mechanical properties, as well as their biodegradability, biocompatibility and low cytotoxicity, offer a simple and safe alternative to the conventional systems (synthetic polymers) [11].

1.7.5 Meat Preservence using cellulose based aerogels

Cellulose-based aerogels are increasingly being recognized as a transformative class of materials for meat packaging and preservation because they provide a unique combination of superabsorbent capacity, structural flexibility, bioactive loading potential, and environmental sustainability [12]. Aerogels have a loose, porous structure, which allows them to effectively absorb and maintain moisture and can be used as excellent carriers of antibacterial agents. Aerogel preservation pads can not only support fresh meat and protect it from mechanical damage but also prevent microbial cross-contamination between meat products. [13,14]. The aerogels can inhibit the growth of spoilage bacteria and pathogenic microorganisms by absorbing exudate from the meat and form a relatively dry environment, thereby prolonging meat shelf life [15].

1.7.6 Thermal Insulation using cellulose based aerogels

Cellulose nanofibrils, the building block of cellulose aerogels, present a natural variability as a function of the feedstock and extraction process [16]. Materials must not only exhibit excellent thermal insulation but also meet rigorous standards for weight, strength, and durability. One prominent application of aerogels lies in spacecraft insulation. Their unique combination of properties including ultralow thermal conductivity, exceptional thermal stability, and remarkable mechanical resilience - makes them particularly suitable for protecting spacecraft components from the intense heat generated during atmospheric re-entry and other high-temperature operations [17]. Benefiting from a unique porous microstructure, aerogel materials are endowed with lightweight properties and low thermal conductivity, demonstrating significant application potential in thermal management fields, including flame-retardant building materials, electronic devices and thermal protection [18].

1.7.7 CO_2 Capture and Utilization by cellulose based aerogel

Global warming poses a major threat to our planet. Excessive use of fossil fuels has led to an increase in atmospheric CO_2 levels, requiring urgent action. Cellulose-based materials emerge as protagonists in the narrative of CO_2 capture and utilization. The porous structure of cellulose, coupled with its chemical functionality, provides it with an innate capacity to sequester CO_2 from diverse sources, ranging from industrial flue gases to ambient air. Moreover, the catalytic potential of cellulose derivatives opens avenues for transforming captured CO_2 into value-added products, thereby forging a circular economy that transcends the traditional boundaries of waste and resources [19].

1.8 SUSTAINABLE AEROGEL FROM ORANGE PEEL WASTE

In this study, we demonstrated the preparation of a cellulose aerogel derived from orange peel (*Citrus sinensis*) waste. The environmentally friendly and low-cost NaOH/urea solution was used to dissolve the extracted cellulose powder to form a cellulose sol, and the gelation of this sol was achieved using biopolymer cross-linkers and temperature control.

1.9 OBJECTIVES

1. Preparation of cellulose aerogel from orange peels.
2. Characterisation of prepared aerogel by XRD, SEM, FTIR analyses.
3. To evaluate the efficiency of prepared samples towards Dye removal.

1.10 SYNTHESIS AND PERFORMANCE OF AEROGELS FROM AGRO-WASTE: A LITERATURE REVIEW

Waste material	**Preparation process**	**Density g/cm^3**	**Porosity %**	**Contact angle**	**Absorption capacity g/g**	**Application**	**Reference**
Orange Waste	High-pressure homogenization + Supercritical CO_2	0.08 to 0.09 g/cm^3	94 - 95%			Upcycled bio-aerogels for solvent capture	[1]
Wood	Freeze drying	0.032				Thermal insulation	[20]
Spent coffee grounds	Freeze drying	0.045	92–95	139°	16	Oil absorption and thermal conductivity	[21]
hemp hurd	Freeze drying	0.008	99.5	150°	56-125	oil absorption	[22]
Durian peel	Freeze drying				0.084	Dye adsorption capacities	[23]

Fresh fruits, vegetables, and mushrooms	supercritical CO_2 drying	0.01–0.12			99	Oleogelation	[24]
Hemp stem	supercritical CO_2 drying, freeze drying				2.20	Water sorption	[25]
Citrus Peel + Chitosan	Sol-gel + Freeze-drying	0.025			0.842-Cu(II)	Heavy metal removal	[26]
Sorghum bagasse	Ultrasonication	6.3	99.4		89	oil absorption capacity o	[27]
Banana leaf	Freeze drying	0.015		149^0	35-115	Adsorption of oil/organic solvents	[28]
Pineapple leaf	Freeze drying	0.127 – 0.326	97-99			Thermal insulation	[29]
Jackfruit	Freeze drying	0.035 - 0.048	95-98			Desalination electrode, NaCl removal capacity.	[30]

Pineapple leaf + Cotton waste	Freeze drying	0.019-0.046	96			Thermal insulation	[31]
Peanut shell	Freeze drying + Carbonization		98	141^0	27-50	Adsorption of oil/organic solvents	[32]
Seaweed solid waste	Freeze drying + Carbonization			153^0	11-30	Adsorption of oil	[33]
Rice straw	Freeze drying	0.012 0.024-0.025	99.5 96	120^0 --------	28-70 24- 26	Adsorption of oil/organic solvents	[34]
Rice straw	Freeze drying	0.002-0.024	98.4 - 99.8	151^0	98-170	Adsorption of oil/organic solvents	[35]
Rice straw	Freeze drying	0.05-0.06	97	150^0	13	Thermal insulation	[36]
Rambutan peel		0.027± 0.002	98	153^0	52	Oil adsorption	[37]

Pomelo	Freeze drying			132.75	94.92	Adsorption of oil/organic pollutants	[38]
Sugarcane bagasse	Freeze drying	0.016-0.122	91.9 - 98.9		31.9-55.02 , 17.1-37.7	Adsorption of oil/organic solvents	[39]
Sugarcane bagasse	Freeze drying	0.012-0.108	92.9 - 99.2	140^0	23	Thermal insulation /oil adsorption.	[40]
White bamboo	Freeze drying	0.085-0.144	90-95	114^0-132^0		Adsorption of oil	[41]
Orange Peel	Freeze drying	0.010417	99	102	13.55 (sandy) 9.60(clay soil)	Oil adsorption	[42]

Chapter 2

Materials and Methods

This chapter gives a brief description of the materials and experimental procedures adopted for the present investigation.

2.1 MATERIALS REQUIRED

- NaOH
- Urea
- Distilled Water
- Gelatin
- Orange peel

2.2 APPARATUS REQUIRED

Temperature Control Magnetic Stirrer and Hot air oven and Freeze dryer.

2.3 EXTRACTION OF CELLULOSE

Orange peels, collected from household waste, are washed and dried in a hot air oven at 125 °C for 48 hours. It is then ground into fine powder, and about 40 g of powdered sample is treated with 4% NaOH (in a 1:10 solid-to-liquid ratio). It was treated with NaOH to remove the non-cellulosic components such as lignin, pectin, etc., from the sample. It is stirred continuously in a magnetic stirrer for 6 hours at 40 °C. Then it is filtered and washed several times with distilled water until the pH of the supernatant liquid becomes neutral. It is again dried in a hot air oven at 60°C for 24 hours.

Fig 2.3.1 Orange peel

Fig 2.3.2 Dried at 125 °C

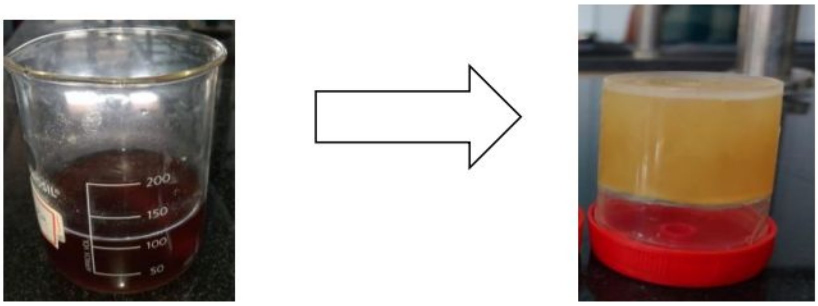
Fig 2.3.3 Powder form

Fig 2.3.4 Powder +4%NaOH

Fig 2.3.5 Dried at 60 °C

2.4 PREPARATION OF CELLULOSE AEROGEL

About 1.5 g of the treated powder, 25 mL of distilled water, 0.75 g of NaOH, and 1.5 g of urea were added, and the mixture was stirred continuously for 2 hours. Separately, a gelatin solution was prepared by dissolving gelatin in distilled water under heating. The prepared gelatin solution was then added to the previously obtained mixture and stirred for an additional 1 hour to ensure homogeneity. The final mixture was allowed to cool to room temperature, poured into molds, and subsequently frozen for 24 hours. The frozen samples were then subjected to freeze-drying for 56 hours to obtain the final product.



Pretreated powder+NaOH+Urea+water Gelatin +Mixture

Freeze dried sample after 56 hours

2.5 CHARACTERIZATION TECHNIQUES

The raw material (orange peel powder), synthesized cellulose and cellulose based aerogel were characterized using X-Ray Diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), Scanning electron microscopy (SEM).

2.5.1 X-RAY DIFFRACTION (XRD) ANALYSIS

XRD analysis was carried out to understand the structural changes occurring during the conversion of orange peel powder into cellulose and cellulose aerogel. The diffraction pattern of raw orange peel showed broad peaks, revealing its predominantly amorphous nature due to the presence of non-cellulosic constituents. After cellulose extraction, the XRD pattern displayed sharper and more distinct peaks, indicating an increase in crystalline order as amorphous components were removed. In contrast, the cellulose aerogel comparatively broader and weaker peaks, suggesting a slight loss of crystallinity caused by chemical modification and aerogel processing.

The crystallinity index (CI) of aerogel was calculated using the Segal equation from XRD diffraction peaks [7].

The Diffraction patterns of the carbon aerogel samples was obtained using a Bruker AXS-D8 Advance diffractometer using Cu-Kα (λ=1.5406 Å) radiation at 40 kV and 30 mA, using continuous scan mode. The X-ray patterns were recorded in the scan range 2θ=30 -800, at a scan rate of

1°/min. The determination of XRD parameters were evaluated using Origin 8.0 software.

2.5.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy is a technique used to study materials by analysing how they absorb infrared radiation. When infrared light passes through a sample, different chemical bonds absorb specific frequencies, causing molecular vibrations. The absorbed energy is recorded and displayed as an intensity versus frequency spectrum, which serves as a unique molecular fingerprint of the material. Each peak in the spectrum corresponds to a particular bond or functional group present in the sample. FTIR is mainly used for qualitative identification and structural analysis, while the amount of material can be estimated from the intensity of the peaks. Owing to its fast, sensitive, and non-destructive nature, FTIR is widely applied in material characterization. In this study, FTIR analysis of the prepared orange peel cellulose aerogel sample was carried out to confirm the successful cross-linking between cellulose and gelatin [8]. The FTIR spectrum of the respective carbons was obtained by analysis on a Thermo Nicolet Avatar 370 over the frequency range of 4000–400 cm^{-1} by placing the carbon samples on KBr pellets. The resolution of the instrument is 0–9 cm^{-1} with a signal–to–noise ratio 15000:1 and measured wavelength range 7800–375 cm^{-1}.

2.5.3 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscopy (SEM) is a technique used to study the surface structure and morphology of materials. In this method, a high-energy electron beam is focused onto the sample surface, and the emitted signals are collected to form a detailed image. The main parts of an SEM include an electron source, lenses, and detectors. SEM operates with electron energies ranging from about 100 to 30,000 eV and provides high-resolution images with magnifications from a few times up to several hundred thousand times. It does not require ultra-thin samples and involves simple sample preparation compared to techniques like TEM. Due to its ability to produce clear surface images in a short time, SEM is widely used in materials science, biology, forensics, and electronics [43].

The surface morphology was evaluated using the Scanning Electron Microscope - JEOL Model JSM - 6390LV working at a high resolution of 3.0 nm in an accelerating voltage of 0.5 to 30 kV with a magnification of 5x to 300,000x. The carbon samples were coated with gold using a sputter coating unit (model: JFC1600) prior to SEM analysis for 10 s at 10 mA.

2.5.4 METHYLENE BLUE ADSORPTION KINETIC STUDIES

Prepared Aerogel sample for Dye Adsorption Analysis

Adsorption kinetic studies were conducted using the prepared OPCAG sample and methylene blue solution of 50ppm concentration. 25mg of the sample were shaken with 25ml of methylene blue solutions at room temperature in a rotatory shaker. A definite volume of methylene blue solution was drawn from the mixture at different intervals of time and the final solution were analysed in UV -visible spectrophotometer at λ_{max}=668nm. The resultant spectrum of absorbance against time and the percentage removal of adsorption were plotted using the Origin 8.0 software [7,31,33, 45]. The adsorption efficiency was calculated by using the equation,

$$\text{Adsorption efficiency} = [\,[\,(A_o - A_t)/A_o]\,] \times 100$$

Where,

A_o = Absorbance of initial concentration

A_t = Absorbance at the time t

Shimadzu, model number: 1900i uv-vis spectrophotometer

Chapter 3

Results and discussion

3.1 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR spectroscopy was employed to study the changes in functional groups present in the raw orange peel and the resulting cellulose aerogel after chemical treatment and cross-linking. The broad and intense absorption band centered at 3340 cm^{-1} and 3205 cm^{-1} corresponds to the O-H stretching vibrations of hydroxyl group, indicative of the extensive hydrogen-bonding network present in the cellulose structure and the N-H groups from the gelatin cross-linker. The peak at 2921 cm^{-1} and 2851 cm^{-1} is assigned to the C-H stretching vibration, a characteristic feature of the cellulose polymer backbone [3]. The presence of two key peaks provides definitive evidence for the successful incorporation of gelatin. The peak at 1621 cm^{-1} is attributed to Amide I (C=O stretching) of the peptide bonds in gelatin [46].

Furthermore, in the peak at 1433 cm^{-1} indicate CH_2 bending [46]. The peak at 1337 cm^{-1} is by the C-H bending [47]. The distinct peak at 1250 cm^{-1} is assigned to Amide III, arising from C-N stretching and N-H bending vibrations in the protein, confirming its role as a cross-linking agent within the aerogel structure [44].

The "fingerprint" region of cellulose is clearly represented by several peaks. The strong absorption at 1160 cm^{-1} is due to the C-O-C asymmetric stretching vibration of the glycosidic linkages [46]. The band at 1081 cm^{-1} and 1033 cm^{-1} corresponds to C-O stretching vibrations of the pyranose ring in cellulose [3].

Finally, the peak at 881 cm^{-1} due to C-H rocking is a characteristic signature of the β-glycosidic linkages between glucose units, confirming the preservation of the cellulose structure throughout the synthesis process [48]. Thus, the spectrum confirmed the successful extraction of cellulose and its subsequent cross-linking with gelatin to form the aerogel matrix.

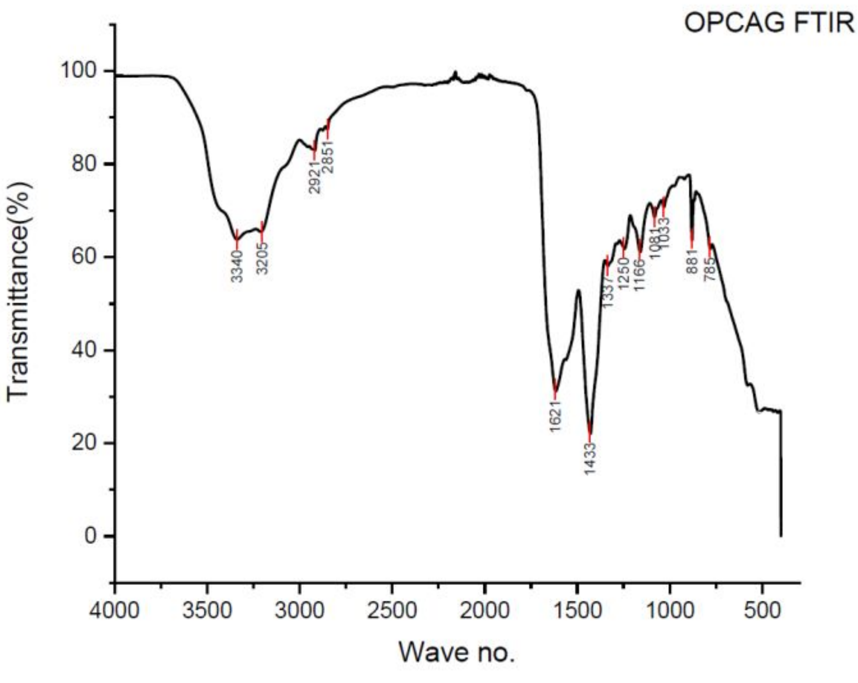


Fig 3.1 FTIR spectrum of OPCAG

3.2 X RAY DIFFRACTION (XRD) ANALYSIS

The X-ray diffraction patterns of the raw material(OP CA G) and the cellulose aerogel are presented in Figure 3.2. A broad peak around 2θ = 24.44° was observed in the XRD spectrum of the sample (OP CA G), indicating that it has an amorphous arrangement. The absence of sharp, intense peaks confirms that the material lacks long-range crystalline order, showing that both the cellulose network and the incorporated gelatin form a highly disordered, porous structure. Small minor peaks appearing at higher angles (such as around 30°, 34°, 37°, 41°, and 46°) are low-intensity signals typically attributed to residual impurities from the biomass or from the drying/processing steps, but they do not contribute to crystallinity [49,50].

In general, the obtained cellulose aerogel is highly amorphous and porous, which is desirable for aerogel applications due to better flexibility, large surface area, and excellent absorption capabilities.
Overall, the XRD results clearly demonstrates the successful extraction of cellulose from orange peel and the formation of a porous cellulose-based aerogel structure.

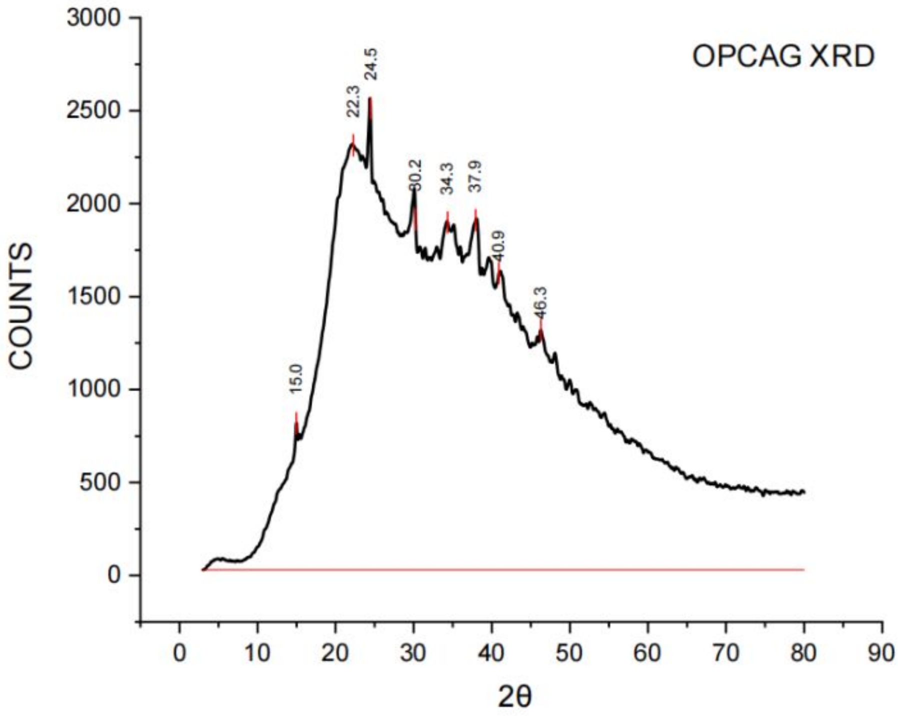


Fig 3.2 XRD diffraction pattern of OPCAG

3.3 SCANNING ELECTRON MICROSCOPY (SEM)

The SEM sample shows a highly porous, irregular flaky structure. The structure consists of unevenly distributed cellulose fragments with loose, sheet-like layers, indicating that the aerogel has undergone significant structural expansion during the drying process. The rough and fractured surfaces suggest the presence of interconnected pores and voids, which are essential for lightweight and absorbent aerogel materials. The morphology confirms that the cellulose network is non-uniform, brittle, and highly porous, supporting its suitability for applications such as adsorption, insulation, and biodegradable material development [8,50]. The SEM images of OPCAG are given below.

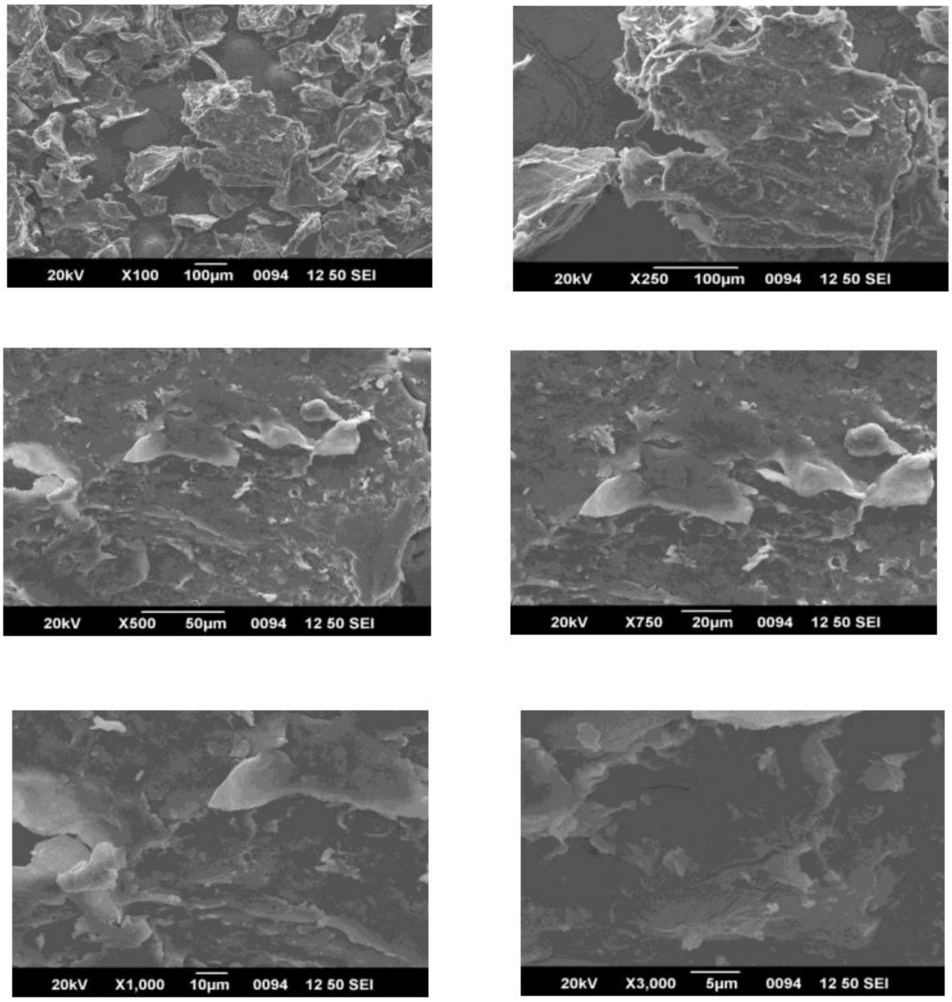


Fig .3.3 SEM images of OPCAG

3.4 METHYLENE BLUE ADSORPTION KINETIC STUDIES

The fig.3.4 shows the UV–Visible absorption spectrum with the time-dependent spectral changes of methylene blue (MB) adsorption by OPCAG. The x-axis represents wavelength (nm) and the y-axis represents absorbance of methylene blue at λ_{max} of 668nm. The highest peak at 668nm shows the strong monomer peak of methylene blue. As the concentration increases methylene blue molecules start to aggregate and the shoulder peak appears at 610nm corresponds to the dimer of methylene blue molecules. The initial spectrum (black curve) exhibits the highest absorbance, confirming the presence of a high initial concentration of MB at the start of the experiment.

As time progresses from 1 hour to 7 hours, a gradual and continuous decrease in absorbance intensity at the characteristic MB peak is clearly observed, without any significant shift in λ_{max}. The steady decline in absorbance suggests effective adsorption of methylene blue by OPCAG, following time-dependent kinetics. The absence of new peaks implies that no strongly absorbing intermediate species are formed during the process.

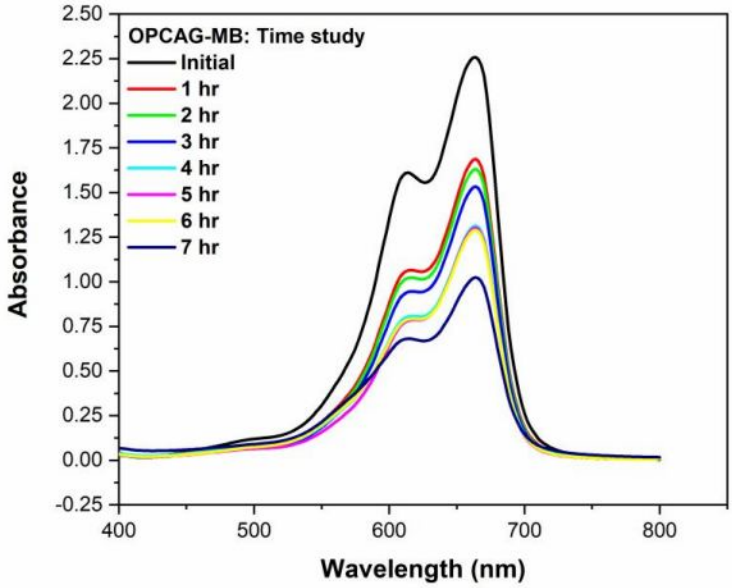


Fig.3.4 Absorbance spectrum of OPCAG at regular intervals of time

The removal efficiency of methylene blue against time by the sample OPCAG in Fig 3.5. As the time increases, the removal percent increases and reaches a higher percent of 50.99%. Adsorption nature of Cellulose aerogels is due to their highly porous structure shown by the SEM images with a very large specific surface area. Methylene blue (a cationic dye) interacts strongly with cellulose through their electrostatic attraction (MB^+ with negatively charged –OH/–O^- groups on cellulose), hydrogen bonding, and π–π interactions leads to an adsorption percentage of 50.99% [8,51].

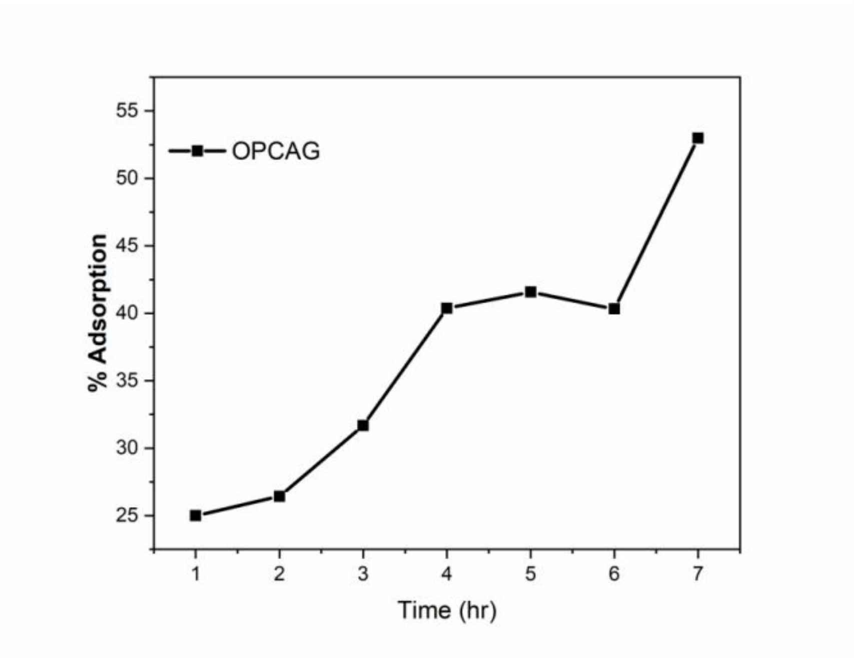


Fig.3.5 The percentage removal of methylene blue by OPCAG

Chapter 4

Conclusions

The reuse and recycling of Agri-commercial wastes especially orange peel (Citrus sinensis) offer immense potential for creating innovative and environmentally sustainable materials. Globally, orange peel is widely utilized for the extraction of essential oils, formulation of cosmetic products, and the development of various other value-added materials. In this project, orange peel was effectively transformed into a lightweight, highly porous cellulosic aerogel through a series of extraction and processing steps. The orange peels were subjected to four step process: alkali treatment extraction of cellulose, preparation of aerogel and freeze drying. These steps successfully removed non cellulosic biomass from the sample. The characterization was performed using XRD, SEM, and FTIR, which confirmed that the aerogel possesses high porosity, an amorphous structure, and low density. The FTIR spectrum showed the main characteristic peaks of cellulose, confirming the presence of essential functional groups in the aerogel. The XRD pattern displayed prominent peaks around $2\theta \approx 15°$ and 22–24°, indicating the retention of the cellulose crystalline structure. SEM analysis revealed a porous and interconnected network, which is typical of aerogel materials. Together, these results confirm that the prepared cellulose aerogel is stable, sustainable, and suitable for future applications. With further improvement in the preparation process, its properties can be enhanced for wider and more advanced uses.

The adsorption study confirms that cellulose aerogel is highly effective in adsorbing methylene blue from aqueous solutions. This adsorption behavior is mainly due to the aerogel's interconnected porous network and the availability of surface hydroxyl groups, which enhance dye–adsorbent interactions. The findings highlight the potential of cellulose aerogel as a sustainable and efficient material for dye removal in water purification applications.

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