MAJOR PROJECT

SYNTHESIS, CHARACTERIZATION, AND COMPARATIVE STUDY OF BIOMASS AND CARBON NANOTUBES COMPOSITE HYDROGELS

A Major Project Report submitted in partial fulfillment for the award of the degree of

MASTER OF SCIENCE

In

CHEMISTRY

Submitted by

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SESSION 2022-2023

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CANDIDATE’S DECLARATION

We, **Akanksha** (2K21/MSCCHE/04) and **Ritika Sharma** (2K21/MSCCHE/58), hereby certify that the work which is being submitted in this major project report entitled **“Synthesis, Characterization, and Comparative Study of Biomass and Carbon nanotubes composite Hydrogels”** in the partial fulfillment for the award of the degree of Master of Science at **Delhi Technological University** is an authentic record of our own work carried out by us under the supervision of **Dr. Anil Kumar** (Professor, Department of Applied Chemistry, DTU).

We further declare that the project report has not been submitted previously to any other Institute/University for the award of any degree or diploma associateship, Fellowship, or other recognition or any other purpose whatsoever. Also, it has not been directly copied from any source without giving its proper reference or citation.

Place: Delhi

Date:

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CERTIFICATE

This is to certify that **Akanksha** and **Ritika Sharma** students of the degree of Master of Science in Chemistry at Department of Applied Chemistry, **Delhi Technological University** have submitted their major project report entitled **"Synthesis, Characterization, and Comparative Study of Biomass and Carbon nanotubes composite Hydrogels"** for the year 2022-2023 in partial fulfillment of the requirement for the award of the degree of Master of Science, is a record of the project work carried out by the students under my supervision.

They have taken proper care and shown utmost sincerity in completion of this project. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to any university or elsewhere, and certify that this project is up to my expectations and as per the guidelines provided by the university.

Place: Delhi **Supervisor's Signature**

Date:

ABSTRACT

The synthesis and characterization of hydrogels using Guargum and acrylamide as co-monomers, KPS as an initiator, and MBA as a cross-linking agent is the primary focus of this research. Functional derivatives of Guargum, such as oxidized Guargum and carboxymethyl Guargum, have been extensively studied to investigate the impact on the physical and chemical properties of hydrogels. Remarkably, the utilization of these chemically modified derivatives has demonstrated a significant enhancement in the swelling ratio compared to the unmodified Guargum hydrogel.

The motivation behind this study stems from the remarkable applications of biomass in dye removal, prompting the investigation, the ways to enhance the dye removal properties of biomass-based hydrogels. To achieve this, dried BL and WC peels were selected biomass for incorporation in hydrogel matrix. Furthermore, carbon nanotubes were introduced in the hydrogel composition to augment its dye removal capabilities. Synthesized material was characterized by XRD, FTIR, and SEM.

The findings of the study demonstrated a significant improvement in the absorption and swelling properties of the hydrogel upon incorporation of biomass and carbon nanotubes. This suggests that the combination of biomass and nanomaterials within the hydrogel matrix enhances its dye removal efficiency, as they pave the way for more effective and sustainable approaches to address the issue of dye pollution. The results obtained from this research provide a foundation for further exploration and optimization of hydrogel compositions for enhanced dye removal. By leveraging the benefits of biomass and carbon nanotubes, this approach offers a promising avenue for developing efficient and eco-friendly materials for water remediation.

ACKNOWLEDGEMENT

The success and final outcome of this project required a lot of guidance and assistance from many people and we are extremely fortunate to have got this all along the completion of this project work.

We wish to express our gratitude towards our project supervisor and Head of the Department, **Prof. Anil Kumar**, Department of Applied Chemistry, Delhi Technological University, who provided us a golden opportunity to work under his able guidance. His scholastic guidance and sagacious suggestions helped me to complete the project on time.

We are thankful to and fortunate enough to get constant encouragement, support and guidance from all teaching staff of Department of Applied Chemistry, which helped us in successfully completing our project work. We are also thankful to PhD scholars Priyanka Yadav, Priyanka Meena, Bhamini Pandey, Sarla, Jigyasa Pathak, and Indu for their constant support and motivation. Special thanks to Priyanka Yadav for helping and guiding us throughout the project.

Finally, yet importantly, we would like to express our heartfelt thanks to our beloved family and friends who have endured our long working hours and whose motivation kept us going.

Akanksha & Ritika Sharma

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LIST OF ABBREVIATIONS

Gg	Guar gum
AAm	Acrylamide
BL	Bamboo leaves
WCN	Water chestnut
CNT	Carbon nanotubes
MWNT	Multi-walled nanotubes
XRD	X- ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy

CHAPTER – 1

INTRODUCTION

“Hydrogel” term first came into existence in 1894 when it was used for colloidal gel. Later on, in 1960, the first application of hydrogels was found by *Wichterle* and *Lim* in the biomedical field [1] The word “hydrogel” is composed of two words: “hydro” meaning water and “gel” meaning a thick substance that is between a liquid and a solid. So a gel in which the liquid component is water can be said to be a hydrogel[2].

Hydrogels are three-dimensional structures made up of polymeric units. Since they are composed of both liquid and solid phases, they show both solid and liquid-like properties[3]. Hydrophilic groups play a significant role in connecting the polymer chains through physical or chemical bonds, resulting in the creation of a three-dimensional polymer network structure. Hydrogels exhibit elastic behavior because of the presence of various cross-linking interactions in them.

Types of Hydrogels

Different factors can be used to categorize hydrogels. ‘*Peppas’* identified three broad categories, ranking them accordingly,

(1) their preparation process;

(2) Ionic charge;

(3) A certain physical structure

Hydrogels can be categorized into different types based on their composition. Homopolymer hydrogels consist of a single type of hydrophilic monomer unit. Copolymer hydrogels are composed of two co-monomer units, with at least one being hydrophilic. Multipolymer hydrogels are formed when three or more co-monomers react together. Lastly, interpenetrating polymeric hydrogels are characterized by the formation of a second intermeshing network structure that arises from the initial network's reaction[4].

Peppas claims that neutral, cationic, anionic, and ampholytic hydrogels can be developed on the basis of ionic charges. Anionic hydrogels have negatively charged groups, cationic hydrogels have positively charged groups, and ampholytic hydrogels have both negatively and positively charged groups, giving them duality in nature[4]. Finally, three categories of hydrogels are classified:

According to their physical structure, hydrogels can be divided into three subcategories: amorphous hydrogels, which are non-crystalline and contain macromolecular chains that are randomly arranged, whereas semi-crystalline hydrogels are composed of both amorphous and crystalline phases and have dense regions of ordered macromolecular chains and hydrogen-bonded structures are composed of electrostatic interactions [4].

The polymer chains are often cross-linked chemically or mechanically to retain the hydrogel's three-dimensional structure in which the covalent bond is joined with the polymeric chains in a chemically cross-linked structure[5].

Composition of Hydrogels

Hydrogels, regardless of their origin being synthetic grafted polymer derivatives or a combination of natural and synthetic components, are referred to as cross-linked polymers[6]. Naturally occurring polymers are specifically known as natural hydrogels which are highly promoted in the market due to their non-toxic nature and can be categorized on behalf of their chemical structure into different classes, including polysaccharides such as chitosan, chitin, cellulose, starch, alginate, gums, and carrageenan, biological polymers as nucleic acid and DNA, polyamides such as collagen, polyphenols includes lignin, inorganic polyesters like polyphosphazene, organic polyesters[7].

To enhance the mechanical properties of hydrogels derived from natural polymers like chitosan and other polysaccharides, various techniques are employed[8]. These include crosslinking the natural polymers, grafting them with monomers, or blending them with synthetic polymers. These modifications are carried out to improve the overall strength and durability of the hydrogels synthesized from natural polymers, which are inherently delicate in their original form[9].

Natural polymeric hydrogels

Natural polymers exhibit a range of properties, including neutrality, cationic or anionic nature due to their readily availability, abundance, cost-effectiveness, non-toxic, and biodegradability[10]. Variable structural and functional modifications have led to an increasing number of material developments for biomedical applications such as controlled and targeted drug release, tissue engineering, and wound healing[11].

Synthetic Polymeric Hydrogels

Synthetic polymers offer attractive advantages in the creation of hydrogels due to their highly controllable physical and chemical properties, surpassing those of natural polymers. These can be engineered to possess long-chain structures and high molecular weight[12]. However, it is important to note that synthetic polymeric hydrogels generally exhibit lower biological activity as compared to natural hydrogels. The synthesis of synthetic polymer hydrogels can be achieved through various methods, including the polymerization of vinyl monomers or the chemical crosslinking of polymers. Commonly used synthetic polymers for hydrogel synthesis includes poly (vinyl alcohol) (PVA), poly(ethylene oxide) (PEO), poly(2-hydroxyethyl methacrylate) (PHEMA), poly(ethylene glycol) (PEG), poly(acrylamide) (PAAm), and poly(acrylic acid) (PAA), among others[13].

Natural Polymers with Synthetic Polymer Hydrogels

Naturally occurring polymers have constraints in terms of their mechanical strength, which restricts their applications in the field of biomedicine. However, synthetic polymers have emerged as a preferred alternative, addressing this drawback and finding extensive utilization in various applications[14]. Synthetic polymers offer advantages such as ease of synthesis, cost-effectiveness, and the ability to tailor their properties for specific applications. Notably, synthetic polymers exhibit excellent mechanical strength, as compared to that natural polymers. Nevertheless, it is important to acknowledge that synthetic polymers are not environmentally friendly, as the resulting solid waste materials are not biodegradable[15].

Hydrogels Based on Poly (Acrylamide) Derivatives

A wide range of acrylamide hydrogels have been synthesized and employed in various fields. Poly(acrylamide) hydrogels, in particular, exhibit hydrophilic and neutral characteristics, along with valuable physical and chemical properties that make them suitable for numerous potential applications[16]. These applications include their use as biomaterials, for cell and biocatalyst immobilization, in drug delivery systems, for absorbing heavy metal ions, and as bio-separators.

Hydrogels can be categorized based on the type of crosslinking forces between polymeric chains. They are classified as chemical hydrogels, physical hydrogels, or a combination of both known as hybrid hydrogels formed by simultaneous crosslinking. Physical hydrogels are created by utilizing non-covalent forces such as hydrogen bonding, ionic interactions, Van der Waals forces, stereo complexation, polyelectrolyte complexation, and hydrophobic interactions. These interactions give rise to reversible responses to environmental changes since the secondary interactions between the polymer chains are relatively weak. Physical hydrogels typically lack order, are delicate, and exhibit low mechanical strength when subjected to external stimuli. They have a tendency to dissolve in organic solvents and water when heated.

In contrast, chemical hydrogels, also known as permanent hydrogels, are formed through covalent bonding between the polymer chains. These hydrogels do not dissolve in the surrounding medium and lack the reversible responses (sol-gel transition) characteristic of physical hydrogels due to the presence of strong covalent bonds. Chemical crosslinking is accomplished using small molecules such as formaldehyde, glutaraldehyde (GA), diglycidyl ether, genipin, and N, N'-methylene bisacrylamide[5]. Covalent bonds are formed between these small molecules and the polymers, stabilizing the network through condensation reactions or free radical mechanisms.

The process of chemical crosslinking endows hydrogels with notable attributes including superior thermal, mechanical, chemical, and surface properties. Additionally, chemical crosslinking plays a crucial role in maintaining the structural integrity of hydrogels in a fully swollen state, ensuring their stability and functionality.

Guar Gum

Cyamopsis tetragonoloba, a plant known for its drought resistance and belonging to the legume family (Leguminosae), has become highly important in the agriculture industry.

It is primarily cultivated in India, and the endosperm of its seeds yields guar gum or guaran, which has been utilized since ancient times. While research on guar gum was limited before 1940, it gained significant attention in the 1940s and 1950s, leading to its industrial applications by 1943[17]. Guar gum exhibits strong hydrogen bonding properties in the water, making it an excellent thickener and stabilizer in various chemical processes. Its usage has notably increased in recent years, finding applications in cosmetics, oils, paints, papermaking, emulsification, suspension, and wound healing. The yellowish-white, natural, nonionic, and unprocessed guaran obtained from the cluster bean is crucial for seed germination. When in contact with water, guar gum forms strong bonds with surrounding hydroxyl groups, thereby maximizing its viscosity potential even in cold water. Guar gum shows minimal dependency on pH, displaying its low viscosity potential at pH 3.5 and reaching maximum viscosity potential within the pH range of 6 to 9. This unique characteristic allows it to maintain consistent viscosity across a broad pH range.

Functional modifications, including grafting, cross-linking, etherification, and carboxylation, have been employed to enhance the potential applications of guar gum in diverse scientific fields. However, it is important to address its inherent deficiencies in order to utilize it effectively for long-term applications. The raw structure of guar gum, characterized by a high molecular weight, can be tailored to achieve specific properties suitable for various applications. Numerous derivatizations have been explored to expand the use of guar gum in fields such as the food industry, flocculating agents, explosives, agriculture, pollutant removal agent, paper industry, cosmetics, pharmaceuticals, and drug delivery[18]. Careful consideration of these modifications allows for the development of guar gum with desired properties for specific applications, enhancing its versatility and effectiveness across a range of industries.

Guar gum is a naturally occurring nonionic polysaccharide with a high molecular weight. It is extracted from the endosperm of seeds and is composed of a straight chain of D-mannose subunits connected by β (1-4) glycosidic bonds. Additionally, D-galactose subunits are linked together by (1-6) glycosidic bonds at alternate positions along the chain[19].

CHAPTER – 2

MATERIALS AND SYNTHESIS

Materials

Highly purified and finely powdered Guargum, oxidized Guargum and Carboxymethyl Guargum were provided by Hindustan Gum Pvt. Ltd. Extra pure acrylamide, KPS (Potassium persulphate), MBA(N, N-Methylenebisacrylamide), Malachite green dye (MG), Eriochrome Black T dye (EBT), Methylene blue(MB), Congo red(CR), Crystal Violet(CV) were provided by CDH, Bamboo leaves were collected from the bamboo trees in Delhi Technological University campus, water chestnuts were purchased from local market. All the samples were prepared in distilled water.

Synthesis

Synthesis of Gg/AAm hydrogel

0.3 g of Gg was added to 20 mL of deionized water and stirred until homogeneous solution obtained. Then, 2.0 g of acrylamide was added to above solution followed by the addition of 0.03g of KPS, as an initiator and it was stirred for 30 minutes. At the end, 0.02g of MBA was added to the mixture and kept on stirring for half an hour. Then the obtained solution was poured in test tube and placed in a water bath at 60 ^{0}C for 60 minutes[20]. Then, the obtained product was cut into small pieces and immersed in deionized water to remove impurities. Then, small discs were air dried at room temperature, followed by oven drying at 60 ^{0}C to get a constant weight.

Synthesis of BL/Gg/AAm hydrogel

Preparation of bamboo leaves biomass: Bamboo leaves were collected and washed thoroughly with distilled water to remove any impurities. The leaves were sundried for 5-6 hours followed by oven drying for 12 hours. The dried leaves were crushed in a mixer to obtain the biomass powder[21].

0.3 g of Gg was added to 20 mL of deionized water and stirred until homogeneous solution obtained. Then, 2.0 g of acrylamide was added to above solution followed by the addition of 0.03g of KPS, as an initiator and 0.0042g of dried bamboo leaves powder was added to the reaction mixture to incorporate the bamboo leaves biomass into the hydrogel and it was stirred for 30 minutes. At the end, 0.02g of MBA was added to the mixture and kept on stirring for half an hour. Then the obtained solution was poured in test tube and placed in a water bath at 60 ^{0}C for 60 minutes[20]. Then, the obtained product was cut into small pieces and immersed in deionized water to remove impurities. Then, small discs were air dried at room temperature, followed by oven drying at 60 ^{0}C to get a constant weight.

Synthesis of WCN/Gg/AAm hydrogel

Preparation of water chestnut biomass: Water chestnut peels were collected and washed thoroughly with distilled water to remove any impurities. The peels were sundried for 5-6 hours followed by oven drying for 12 hours. The dried peels were crushed in a mixer to obtain the biomass powder[22].

0.3 g of Gg was added to 20 mL of deionized water and stirred until homogeneous solution obtained. Then, 2.0 g of acrylamide was added to above solution followed by the addition of 0.03g of KPS, as an initiator and 0.0042g of dried water chestnut peel powder was added to the reaction mixture to incorporate the water chestnut biomass into the hydrogel and it was stirred for 30 minutes. At the end, 0.02g of MBA was added to the mixture and kept on stirring for half an hour. Then the obtained solution was poured in test tube and placed in a water bath at 60 ^{0}C for 60 minutes[20]. Then, the obtained product was cut into small pieces and immersed in deionized water to remove impurities. Then, small discs were air dried at room temperature, followed by oven drying at 60 ^{0}C to get a constant weight.

Synthesis of CNT/Gg/AAm hydrogel

Preparation of ox-MWNTs- Oxidized multi-walled carbon nanotubes (ox-MWNTs) were synthesized using a two-step process. Initially, a small amount of pristine MWNTs was dispersed in a mixture of sulfuric and nitric acid (3:1, v/v)[23]. The dispersion was then subjected to sonication for 24 hours at room temperature. Subsequently, deionized water was gradually added to the mixture at 0°C, resulting in dilution. The ox-MWNTs were separated by filtration, followed by resuspension in water and repeated washing until the pH of the filtrate reached neutrality. The resulting black powder was then dried under vacuum overnight to obtain the final product of ox-MWNTs[24].

0.0030 g of ox-MWNTs were sonicated for 2 hours. 0.3 g of Gg was added to 20 mL of deionized water and stirred until homogeneous solution obtained. Then, 2.0 g of acrylamide was added to above solution followed by the addition of 0.03g of KPS, as an initiator along with the sonicated MWNTs and it was stirred for 30 minutes[25]. At the end, 0.02g of MBA was added to the mixture and kept on stirring for half an hour. Then the obtained solution was poured in test tube and placed in a water bath at 60 ^{0}C for 60 minutes. Then, the obtained product was cut into small pieces and immersed in deionized water to remove impurities. Then, small discs were air dried at room temperature, followed by oven drying at 60 ^{0}C to get a constant weight.

CHAPTER – 3

CHARACTERIZATION

Swelling Study

The equilibrium water content of hydrogels refers to the amount of water retained by the hydrogel when it reaches a state of balance with its surrounding environment[26]. It is an important parameter that characterizes the water-holding capacity of hydrogels and is often determined experimentally.

Understanding the equilibrium water content of hydrogels is significant for several reasons. Firstly, hydrogels are highly absorbent materials due to their three-dimensional network structure, which allows them to absorb and retain large amounts of water[27]. Determining the equilibrium water content helps in evaluating the effectiveness of a hydrogel for applications such as drug delivery, wound healing, or tissue engineering[28]. It provides insights into the water-absorbing capacity and hydration behavior of the hydrogel.

Secondly, the equilibrium water content affects the mechanical properties and functionality of hydrogels[29]. Water plays a crucial role in maintaining the integrity, swelling behavior, and overall performance of hydrogels. The water content directly influences parameters such as swelling ratio, elasticity, and porosity, which are crucial for applications that require specific mechanical properties or controlled release of substances[30].

Thirdly, by studying the equilibrium water content, researchers can gain insights into the interactions between the hydrogel matrix and water molecules. Hydrogels are typically composed of hydrophilic polymer networks that can form physical or chemical interactions with water[31]. Understanding the water-polymer interactions helps in tailoring the hydrogel formulation, optimizing the composition, and designing hydrogels with desired water-absorption characteristics.

Overall, determining the equilibrium water content of hydrogels is essential for assessing their water-retention capacity, mechanical behavior, and performance in various applications[32]. It provides valuable information for the development and optimization of hydrogel-based materials and systems.

$$Swelling\ Ratio\ (\%) = \frac{(W_s - W_d)}{W_d}$$

where, W_s is the weight of swollen hydrogel and W_d is the weight of dry hydrogel[23].

Powder X-ray Diffraction (PXRD)

PXRD is a technique used to analyze the crystal structure and crystalline properties of materials. It is commonly employed in the study of hydrogels for several reasons.

PXRD analysis is performed on hydrogels to investigate their structural characteristics. While hydrogels are primarily composed of polymer chains in a three-dimensional network, some polymers may exhibit partial crystallinity or ordering at the molecular level. It allows researchers to examine the presence of crystalline regions within the hydrogel matrix and provides insights into the arrangement and packing of polymer chains[33].

By conducting PXRD analysis, the degree of crystallinity and crystalline phases within hydrogels can be assessed. The diffraction pattern obtained from XRD measurements provides information about the spacing and orientation of crystal planes in the hydrogel structure. Changes in the diffraction pattern can indicate variations in the degree of crystallinity or the presence of different crystalline phases as a result of different processing conditions or composition variations.

Moreover, XRD spectroscopy is used to study the structural changes in hydrogels due to external stimuli or environmental factors. By subjecting hydrogels to different temperature, humidity, or pH conditions, XRD analysis can reveal alterations in the crystalline structure, phase transitions, or the amorphous-to-crystalline ratio[34]. This information aids in understanding the responsiveness and behavior of hydrogels under different conditions.

XRD analysis plays a crucial role in characterizing the crystal structure, degree of crystallinity, and phase behavior of hydrogels. It provides valuable insights into the molecular organization and ordering within the hydrogel matrix, aiding in the design and optimization of hydrogel formulations for various applications.

Fourier Transform Infrared Spectroscopy

FTIR (Fourier Transform Infrared Spectroscopy) is a technique used to analyze the chemical composition and molecular structure of materials by measuring the absorption and transmission of infrared light[35]. It is commonly employed in the study of hydrogels due to several reasons.

FTIR spectroscopy is utilized for hydrogels to examine their chemical bonds and functional groups. Hydrogels are composed of various polymers, cross-linkers, and additives, and FTIR analysis provides valuable information about the specific chemical bonds present within the hydrogel matrix. It enables researchers to identify and characterize functional groups, such as hydroxyl (OH), carbonyl (C=O), and amide (NH) groups, which are typically found in the constituent polymers and crosslinking agents of hydrogels[36].

By conducting FTIR analysis, the degree of crosslinking within hydrogels can be investigated. The presence of specific absorption peaks and shifts in the FTIR spectra can indicate the formation of chemical bonds resulting from crosslinking reactions. This information helps in understanding the structural integrity, stability, and mechanical properties of the hydrogel.

Moreover, FTIR spectroscopy is used to assess the interaction between hydrogels and other molecules or substances. By comparing FTIR spectra before and after exposure to various environments, changes in the hydrogel's chemical structure, such as the uptake or release of molecules can be determined, and the compatibility and performance of hydrogels for applications such as drug delivery or bio sensing can be evaluated.

FTIR analysis plays a vital role in the characterization and development of hydrogels, providing insights into their chemical composition, crosslinking efficiency, and interaction capabilities. It assists in tailoring the hydrogel formulation, optimizing synthesis conditions, and evaluating the suitability of hydrogels for specific applications[37].

Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is a technique used to visualize the surface morphology and topography of materials at high magnification. It involves scanning a sample with a focused electron beam and detecting the emitted electrons to generate an image[38].

SEM is commonly employed to examine hydrogels due to several reasons. Firstly, hydrogels possess a three-dimensional structure that can exhibit intricate surface features and internal structures, which can be observed through SEM imaging. Secondly, SEM allows for the visualization of the surface roughness, pore structure, and interconnectivity of hydrogels, providing valuable insights into their microstructure. Thirdly, SEM enables researchers to investigate the distribution and arrangement of components within the hydrogel matrix, such as particles, fibers, or crosslinked networks[39]. Additionally, SEM can be used to assess the effect of different parameters, such as composition, processing conditions, or crosslinking methods, on the morphology and structure of hydrogels.

By employing SEM, a deeper understanding of the microstructural characteristics of hydrogels, which is crucial for assessing their performance, mechanical properties, swelling behavior, and intermolecular interactions can be gained. This information aids in the development and optimization of hydrogels for various applications, including tissue engineering, drug delivery, and biomaterial design[40].

CHAPTER – 4

RESULTS AND DISCUSSION

Swelling Studies

The swelling ratio of different compositions is shown in Table 1.

It was observed that upon incorporating the hydrogels with biomass and carbon nanotubes, the swelling ratio increased significantly.

The introduction of oxidized Guar gum results in an almost twofold increase in the swelling ratio. Similarly, the carboxymethyl derivative of Guar gum showcases a remarkable fourfold increase in the swelling ratio. This significant increase can be attributed to the following reasons:

Increased hydrophilicity - Functional derivatives of Guar gum, such as oxidized Guar gum and carboxymethyl Guar gum, introduce hydrophilic groups into the polymer structure. These hydrophilic groups have a higher affinity for water, leading to enhanced water absorption and increased swelling of the hydrogel[41].

Improved polymer chain flexibility: The introduction of functional derivatives alters the polymer chain conformation and increases its flexibility. This increased flexibility allows for easier penetration and diffusion of water molecules into the hydrogel network, leading to higher swelling capacity.

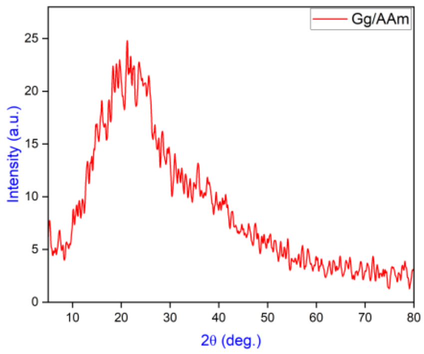
Increased ionization: The functional derivatives can introduce ionizable groups into the Guar gum structure. These ionizable groups can enhance the ionic interactions between the hydrogel and the surrounding aqueous medium. This results in an osmotic pressure difference between the hydrogel and the external environment, leading to water uptake and subsequent swelling.

Enhanced crosslinking efficiency: Functional derivatives of Guar gum can improve the crosslinking efficiency of the hydrogel network. The introduction of functional groups can create additional sites for crosslinking agents, such as KPS (potassium persulfate) and MBA (N, N'-methylenebisacrylamide), to form stronger and more extensive crosslinking networks. The enhanced crosslinking density contributes to the increased swelling ratio.

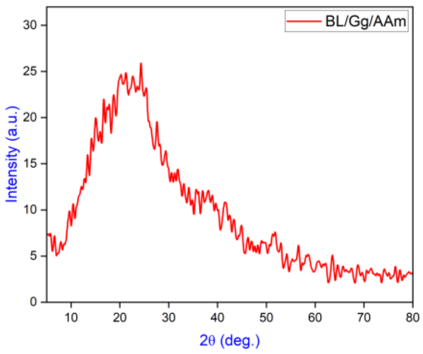
SAMPLE	WEIGHT OF DRY HYDROGEL (g)	WEIGHT OF SWOLLEN HYDROGEL (g)	SWELLING RATIO (%)
Gg/AAm	0.2142	2.0659	864.47
o-Gg/AAm	0.0297	0.5370	1708.08
cmg-Gg/AAm	0.0204	0.6490	3081.37
CNT/Gg/AAm	0.2152	2.1607	904.04
BL/o-Gg/AAM	0.0415	0.7149	1622.65
WCN/o-Gg/AAm	0.0407	0.6246	1434.64
BL/cmg-Gg/AAm	0.0270	0.8206	2939.25

Table-1: Swelling ratio of different hydrogel compositions

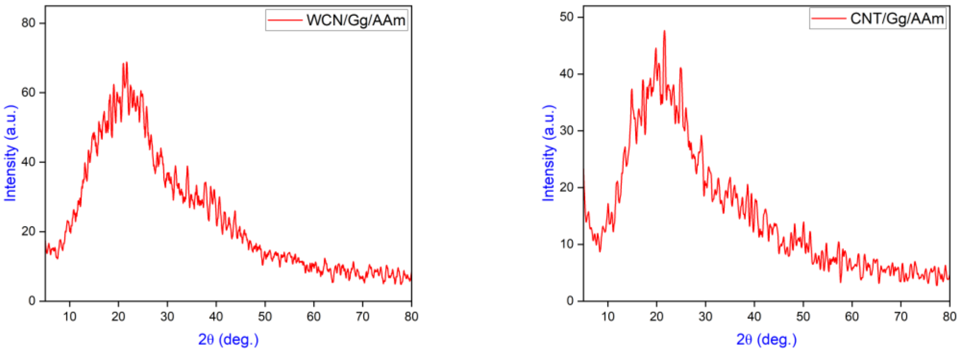
X-RAY DIFFRACTION



(a)



(b)



(c) **(d)**

Fig.1: XRD plots of (a) Gg/AAm hydrogel, (b) BL/Gg/AAm hydrogel, (c) WCN/Gg/AAm hydrogel, (d) CNT/Gg/AAm hydrogel

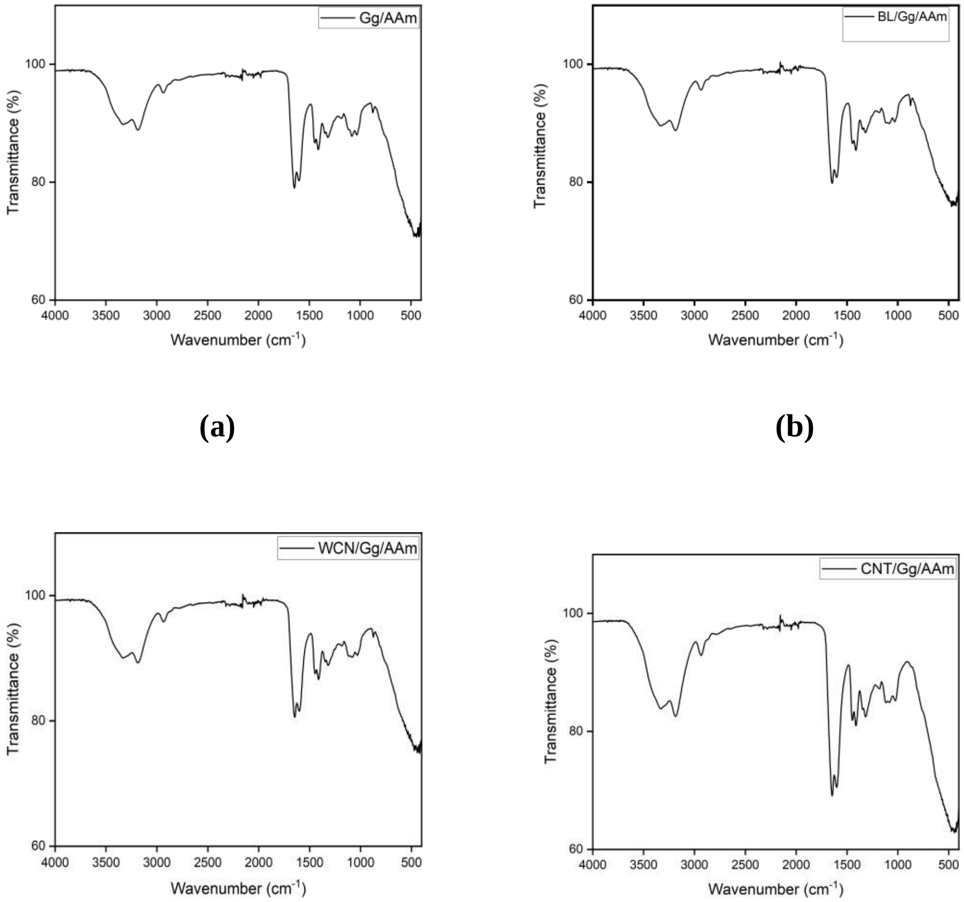
The PXRD analysis of the hydrogels showed the presence of a characteristic XRD peak around 20-25 degrees which is consistent with the prior literature. A very small shift is observed in the case of incorporated hydrogels which might be attributed to the interactions between the biomass or carbon nanotubes with the hydrogel matrix[42].

The plot exhibits an amorphous pattern because the components used in the hydrogel formulation, such as guar gum and acrylamide, are typically amorphous or lack significant long-range order.

In the XRD plot of the hydrogel, a broad and featureless baseline is observed, indicating the absence of crystalline regions or well-defined crystallographic planes. This is consistent with the amorphous nature of the hydrogel matrix, where the polymer chains are not arranged in a highly ordered manner[41].

Overall, the XRD plot of the hydrogels display a predominantly amorphous pattern, indicative of the amorphous nature of the hydrogel matrix and the absence of significant crystallinity.

Fourier Transform Infrared Spectroscopy



(a) (b)

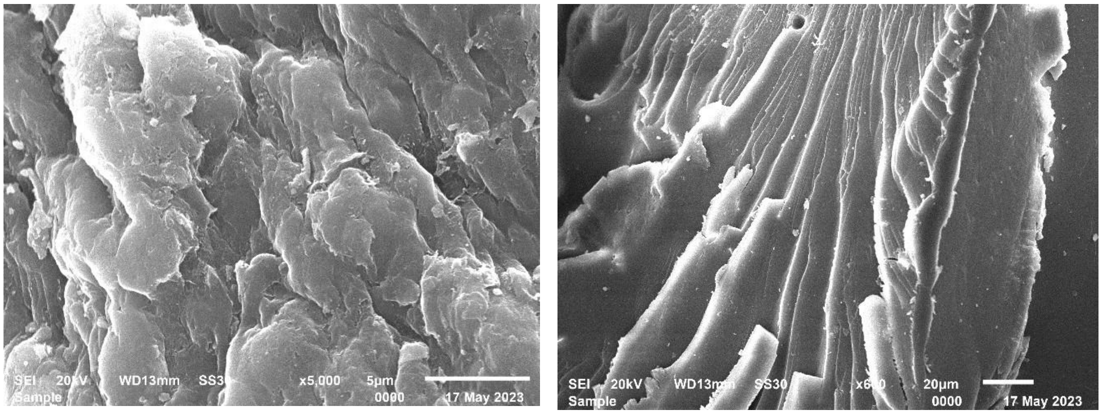
(c) (d)

Fig.2: FTIR plot of (a) Gg/AAm hydrogel, (b) BL/Gg/AAm hydrogel, (c) WCN/Gg/AAm hydrogel, (d) CNT/Gg/AAm hydrogel

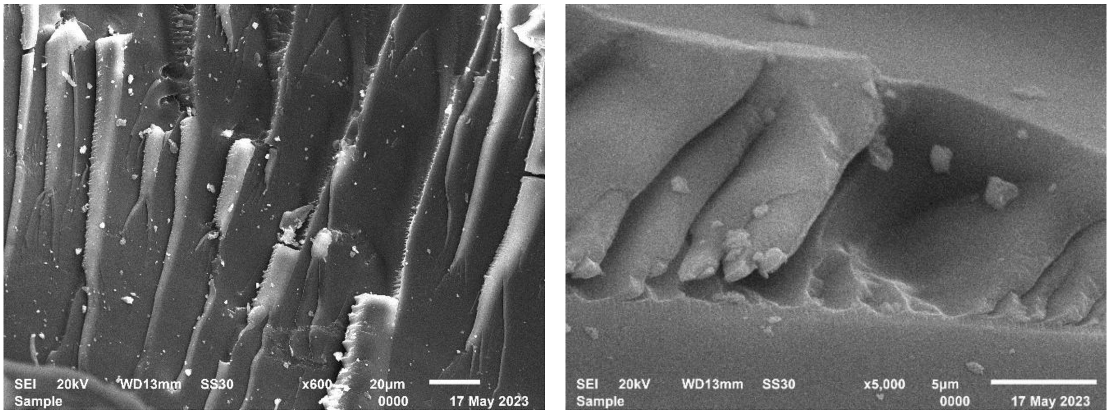
The broad absorption peak observed at 3332 cm-1 can be associated with the –OH stretching vibration, it indicates the presence of –OH groups, which are commonly found in guar gum and acrylamide. The peaks at 1648 cm-1 and 1600 cm-1 can be attributed to the carbonyl stretching vibration and –NH bending vibration indicating the presence of amide groups, which are characteristic of acrylamide and MBA. The presence of –CH bonds which are commonly found in both guar gum and acrylamide can be confirmed by the characteristic peak at 2927 cm-1 indicative of the –CH stretching vibration[42].

Similar absorption peaks were also observed for the biomass and carbon nanotube incorporated hydrogels. This implies that the incorporation is not leading to any change in functionalization of the hydrogels[43].

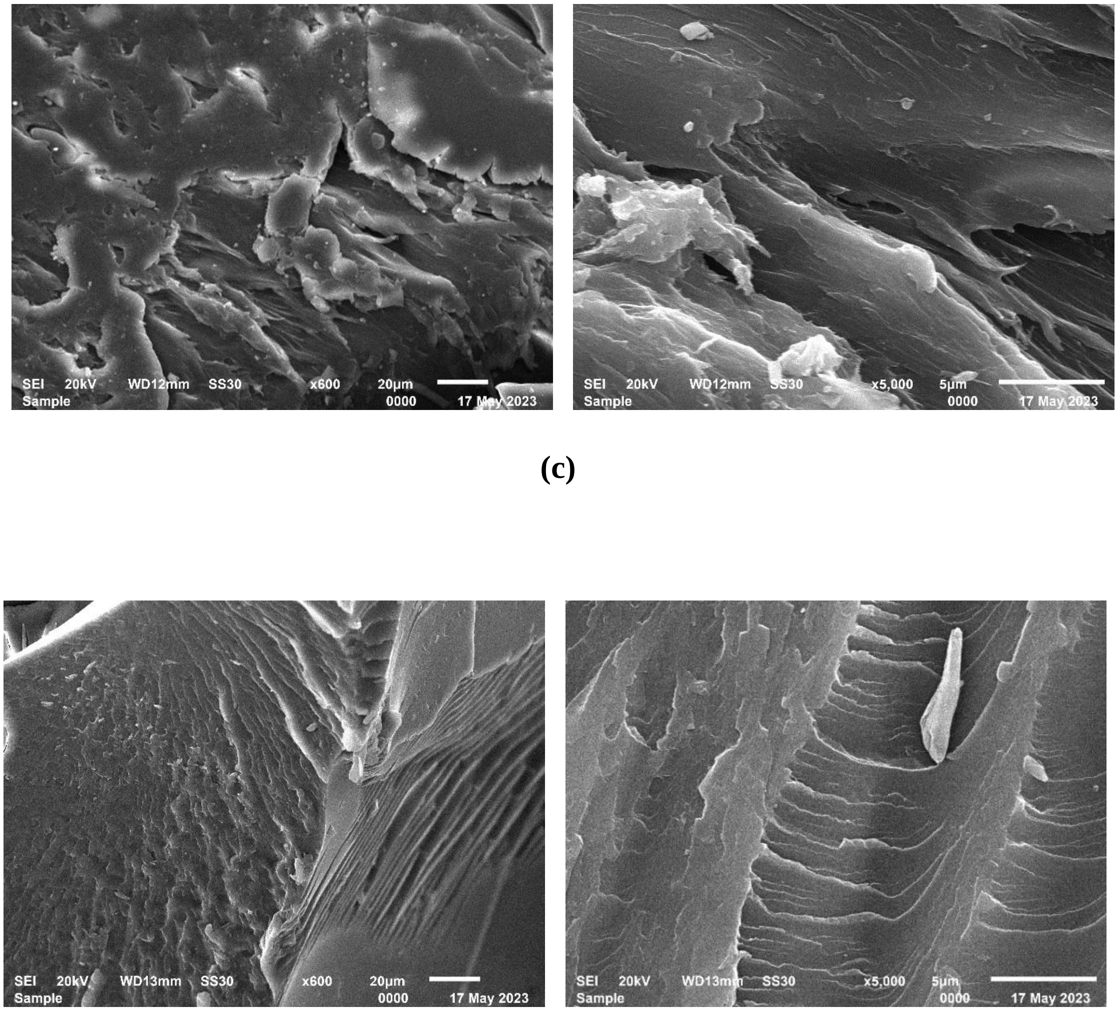
Scanning Electron Microscopy



(a)



(b)



(c)

(d)

Fig.3: SEM images of (a) Gg/AAm hydrogel, (b) BL/Gg/AAm hydrogel, (c) WCN/Gg/AAm hydrogel, (d) CNT/Gg/AAm hydrogel

SEM micrographs of the Guar gum hydrogel is shown in Fig.3. The structural morphology of the hydrogel was confirmed by comparing with different research articles. The resulting hydrogels exhibit a varied textured surface, which can be attributed to the intermolecular interactions and cross-linking reactions occurring among the polymer chains. These processes give rise to a three-dimensional hydrogel structure, influencing both the external appearance and internal morphology of the guar gum hydrogels[44].

The SEM micrograph of the bamboo leaves incorporated hydrogel shows the structural and morphological difference in the hydrogel on infusing it with the biomass. The surface becomes more rough and the pore size increases. There can be seen regions of entanglement where the biomass maybe interacting with the hydrogel matrix and changing the overall structure[21].

It can be seen from the SEM micrographs of Water chestnut peel incorporated hydrogel that the surface has more irregularities as compared to the hydrogel without any biomass. The incorporation has led to the change in overall structure of the hydrogel as the biomass particles influence the void spaces and porosity of the hydrogel matrix. Interconnected pores seen in the SEM can be associated with the presence of biomass in the hydrogel matrix[22].

The incorporation of CNT can very clearly be seen in the SEM micrographs of the CNT incorporated hydrogel. The CNTs are aligned along the hydrogel matrix, improving the overall mechanical strength of the hydrogel. As CNTs are protruding at places from the hydrogel matrix, this also provides the hydrogel with a textured or irregular surface. Since CNTs are highly conductive in nature, the network of CNTs throughout the hydrogel can create pathways for electron transfer and can also form conductive bridges[23], [45].

Absorption Study

A stock solution with a concentration of 100 ppm was prepared by dissolving 0.01 g of the dye in 100 mL of distilled water. The dyes used were MG, CV, MB, CR, and EBT[46], [47]. From the stock solution, standard solutions with varying concentrations were prepared for the absorption study[48].

To investigate the dye removal capacity, 50 mg of hydrogels were immersed in 50 mL of the standard dye solution and placed in an orbital incubator shaker. At specific time intervals (10 min, 20 min, 30 min, 60 min, 90 min, 120 min, and 150 min), small aliquots of the solution were withdrawn from the flask. These samples were then analyzed using a UV-Visible Spectrophotometer to determine the percentage of dye removal. It was observed that the equilibrium time for maximum absorption was 120 min, during which significant dye removal occurred.

The hydrogels incorporated with biomass exhibited a remarkably high percentage of dye removal, reaching up to 95.54%. This was significantly higher compared to the plain hydrogel. Additionally, the hydrogels incorporating multi-walled carbon nanotubes (CNT) also demonstrated an increased percentage of dye removal.

However, despite attempting to replicate the experiments, consistent results could not be obtained. Further research is necessary to understand the underlying reasons for this inconsistency.

APPLICATIONS

Guargum hydrogels have found applications in numerous fields, demonstrating their versatility and usefulness. Here are a few examples:

Food industry: Guargum hydrogels are widely employed in the food industry as thickeners, stabilizers, and emulsifiers. They can improve the texture, viscosity, and stability of various food products, including sauces, dressings, ice creams, and bakery items.

Pharmaceutical industry: Guargum hydrogels have been explored for pharmaceutical applications such as controlled drug release systems, wound healing dressings, and mucoadhesive formulations. The biocompatibility and swelling properties of Guargum make it suitable for drug delivery and tissue engineering applications.

Agriculture: Guargum hydrogels have been utilized in agriculture to enhance soil moisture retention and improve water management. These hydrogels can absorb and retain water, gradually releasing it to plants' root systems, improving crop yields, and reducing irrigation requirements.

Personal care products: Guargum hydrogels are used in personal care products like shampoos, lotions, and creams as thickeners and stabilizers. They enhance the consistency, texture, and overall performance of these products.

Oil and gas industry: Guargum hydrogels are employed in hydraulic fracturing (fracking) operations to increase viscosity and improve the efficiency of fluid transport. They help in carrying proppants into fractures, providing better fracture conductivity and enhancing oil and gas recovery.

Textile industry guargum hydrogels find applications in textile printing and dyeing processes. They act as thickening agents in dye pastes, improving color adhesion to fabrics and enhancing print quality.

These applications highlight the wide-ranging utility of Guargum hydrogels in various industries, making them a subject of interest for research and development in fields such as food science, pharmaceuticals, agriculture, and more[12].

CHAPTER- 5

CONCLUSION

Hydrogels were synthesized using different concentrations of initiator and cross-linker to examine how their variations impact the composition of the hydrogel. Functional derivatives of Guar gum, such as oxidized Guar gum and carboxymethyl Guar gum, were extensively studied to investigate the impact on the physical and chemical properties of hydrogels. Remarkably, the utilization of these chemically modified derivatives has demonstrated a significant enhancement in the swelling ratio compared to the unmodified Guar gum hydrogel.

The hydrogel formulation demonstrating the highest water retention capacity was selected. This hydrogel composition was further modified by incorporating Bamboo leaves, Water chestnut peels biomass, and Carbon nanotubes to investigate their potential for dye removal applications. Preliminary findings revealed promising dye removal properties; however, replication of these results was not successful. Consequently, further research is required to determine the underlying cause for this inconsistency.

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