

Extraction and Characterization of Nanocellulose-Based Hydrogels for Water Conservation in Omani Agriculture

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Abstracts: This research project focuses on extracting and characterizing nanocellulose from agricultural waste for hydrogel production. The nanocellulose samples achieved a 98% purity through EDX analysis, and SEM, EDX, FTIR, and XRD tests confirmed their structural and compositional properties. Cross-linking the nanocellulose with Arabic gum and chitosan produced environmentally friendly hydrogels. Laboratory tests evaluating water absorption and retention determined that higher amounts of Chitosan resulted in longer absorption times, while 15% Chitosan with 30g exhibited the highest absorption capacity. Additionally, 15% Arabic Gum with 20g demonstrated rapid absorption within approximately 5 minutes, suggesting enhanced absorption properties. These findings contribute to sustainable agriculture in Oman, offering an innovative solution for water conservation. The results provide valuable insights for developing efficient hydrogel formulations and implementing water management strategies that promote environmental stewardship and facilitate water conservation in crop cultivation.

Keywords: Nanocellulose, Hydrogels, Water Conservation, Arabic Gum, Chitosan.

1. INTRODUCTION

In the Sultanate of Oman, where the agricultural sector grapples with the adversities of an arid climate and limited water resources, a research initiative seeks innovative solutions through the application of nanotechnology[1]. The government, recognizing the significance of agriculture despite challenging conditions, has implemented measures such as subsidies and investments in modern farming techniques to support the sector[1]. However, high evaporation rates, exacerbated by Oman's hot and arid climate, remain a formidable challenge, particularly during summer months when temperatures can reach up to 50°C (122°F) in certain regions [2]. The annual average evaporation rate in Oman ranges from 1,660 mm/year in the south (Salalah plain) to 2,200 mm/year in the interior, making it one of the highest rates globally [3]. To address these challenges, a research project is underway, focusing on the extraction of nanocellulose from agricultural waste materials [4][2][5][6]. This research delves into various extraction methods, with a particular emphasis on acid hydrolysis [7][8][4][9][6]. Reed plants and date palm empty fruit bunches are identified as cellulose-rich sources, and the process involves preparing a sulfuric acid solution, treating the fibers, rinsing, and drying to isolate cellulose fibers[1]. The use of Chitosan and Arabic Gum in hydrogel production is introduced, with these hydrogels, especially superabsorbent variants, demonstrating potential applications in agriculture, environmental protection, and energy storage[10][4][3][11]. The cross-linking agents, including Arabic Gum, Chitosan, and Epichlorohydrin, are identified as pivotal components in optimizing the properties of hydrogels[9][9][4][12]. These hydrogels, envisioned as a solution to combat high evaporation rates, hold promise in enhancing water retention near crops, thereby contributing to efficient water utilization in Oman's agricultural practices[4]. The project's holistic scope encompasses nanocellulose extraction, hydrogel preparation, and comprehensive crop testing, underscoring its commitment to addressing water scarcity and fostering sustainable agricultural practices[11][1]. The broader context of this research aligns with the principles of Green Nanotechnology, emphasizing environmentally friendly and sustainable approaches[4][11]. By utilizing nanotechnology to combat water scarcity and optimize agricultural practices, the project not only contributes to the preservation of precious water resources but also minimizes the environmental impact of conventional farming methods[11][1][13]. The goal is to inspire innovation, scientific exploration, and the adoption of responsible water use practices, contributing to the overarching objectives of global sustainability. In addition to its environmental implications, the project carries significant societal benefits for Oman. By addressing water scarcity challenges, it ensures a stable water supply for both agricultural and domestic needs. This innovation stands to improve the livelihoods of farmers by increasing crop productivity and reducing costs[11][1]. Furthermore, it creates economic

opportunities, including job creation and the development of new industries. The intellectual growth and advancement of society are also promoted through the encouragement of innovation and scientific exploration[2][1]. In conclusion, the research project in Oman represents a multifaceted approach to addressing agricultural challenges in an arid climate[1]. Through the extraction of nanocellulose and the development of hydrogels with strategic cross-linking agents, the project aims to revolutionize water management in agriculture, fostering sustainability and resilience in the face of climate challenges. The alignment with Green Nanotechnology principles underscores the commitment to environmentally friendly solutions, emphasizing the potential for positive societal and economic impacts in Oman[6][13].

2. MATERIAL & METHODOLOGY

2.1 Material

The materials employed include date palm leaves sourced from the College of Engineering at the National University of Science and Technology, chitosan, Arabic Gum obtained from the Matrah souq, and solvents such as distilled water, acetic acid, sulfuric acid (H₂SO₄), and N-Methyl-2-pyrrolidone (C₅H₉NO).

2.2 Extraction Nanocellulose

In the experimental procedure, date palm leaves were washed and placed in a beaker, followed by drying in an oven at 60°C. Sulfuric acid solution was prepared and added to weighed samples of dry reed plants and date palm leaves. The mixture was stirred at 45°C for 3 hours, filtered under vacuum with continuous addition of distilled water until reaching a pH of 7. The resulting cellulose samples were dried in an oven at 60°C. Different weights of dried cellulose were mixed with Methyl pyrrolidone solution and stirred at 80°C, yielding cellulose solutions. This procedure outlines the steps involved in extracting cellulose from date palm leaves and preparing cellulose solutions using Methyl pyrrolidone as a solvent.

2.3 Hydrogel Preparation

Arabic Gum preparation, 13 grams of Arabic Gum were added to a beaker containing 100 ml of distilled water. The mixture was stirred until the Gum completely dissolved. The cellulose solution was then added to the Gum solution at two different ratios (1:3). The solutions were stirred on a magnetic stirrer until they formed a homogeneous mixture.

To prepare the Chitosan solution, 1.3 grams of chitosan were combined with 100 ml of acetic acid in a beaker. The mixture was thoroughly stirred until the chitosan completely dissolved in the acetic acid. Subsequently, the cellulose solution was added to the chitosan solution in a 1:3 ratio. The combined solutions were stirred using a magnetic stirrer until a homogeneous mixture was obtained.

3. RESULTS AND DISCUSSION

After achieving the desired outcomes and effectively extracting nanocellulose, the results revealed that 3.5 grams of nanocellulose were obtained from 10 grams of raw date palm leaves.

3.1 FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy is a technique used to identify chemical bonds in molecules by generating an infrared spectrum. This technique is used to examine and analyze samples, characterizing their covalent bonding information. The spectrum is represented by the horizontal axis (x-axis), with absorption bands or peaks representing the vibrations of atoms in the sample. The mid-range infrared range is between 4000 and 400 cm⁻¹, while the vertical axis (y-axis) represents absorption or frequency. The experiment results showed that all

materials exhibited major absorption areas, which were consistent with previous scientific papers but with greater clarity. The range between 700 to 1800 cm^{-1} is a low range, while 2700 to 3500 cm^{-1} is a high range. The range between 2200 and 2400 cm^{-1} is a narrow range, and specific absorption peaks can be identified for each specific component in the sample.

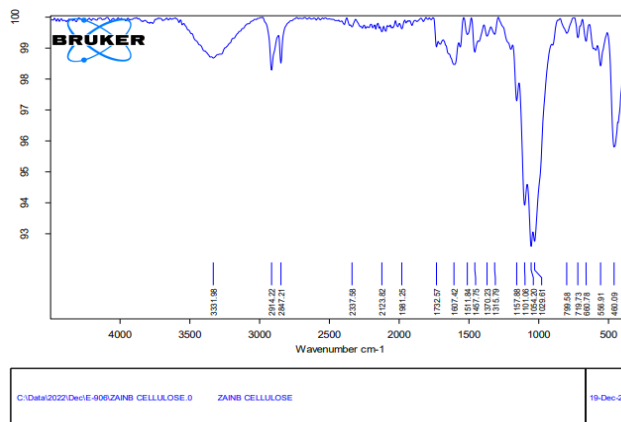


Figure.1 FTIR of cellulose

3.2 XRD Analysis

The X-ray diffraction analysis was carried out using a desktop D2 Phase X-ray meter (Bruker Corp) with irradiation parameters set at 30 kV and 10 A. The diffraction angle range was from $2\theta=4^\circ$ to 40° with a step size of 0.33° . The crystallinity index (CI) exhibited the highest intensity deviation at the (200) peak. Cellulose diffraction patterns were obtained, illustrating the distinct peaks observed in different samples. Specifically, in fronds, peaks were observed at $2\theta=12.5^\circ$ (corresponding to the (010) plane) and $2\theta=15.5^\circ$ (corresponding to the (001) plane). In leaves, a peak was observed at $2\theta=15.5^\circ$ (corresponding to the (001) plane). In coir, peaks were observed at $2\theta=16.5^\circ$ (corresponding to the (100) plane), $2\theta=19.5^\circ$ (corresponding to the (021) plane), and $2\theta=22.5^\circ$ (corresponding to the (200) plane). These findings provide valuable insights into the crystalline structure of cellulose in the respective samples.

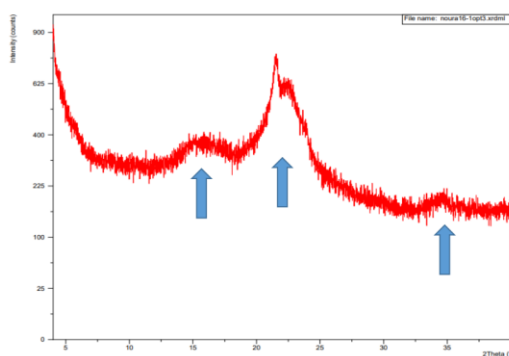


Figure.2 XRD of cellulose

3.3. SEM &EDX analysis

Cellulose nanoparticles in palm fronds were characterized using scanning electron microscopy (SEM) after wax removal and cleaning. SEM images showed cellulose particles with dimensions ranging from 1000 nm to 20000 nm, displaying various shapes and sizes. These findings provide valuable information about the size, shape, distribution, and composition of cellulose nanoparticles, highlighting their potential applications.

The electron diffraction pattern of cellulose exhibited multiple peaks, indicating crystal structure characteristics such as crystal symmetry, lattice constants, and unit cell diameters. The degree of order in cellulose chains, known as crystallinity, was determined by calculating the crystallinity index or degree of crystallinity. The high percentage of compound C observed in the sample suggests a high cellulose content.

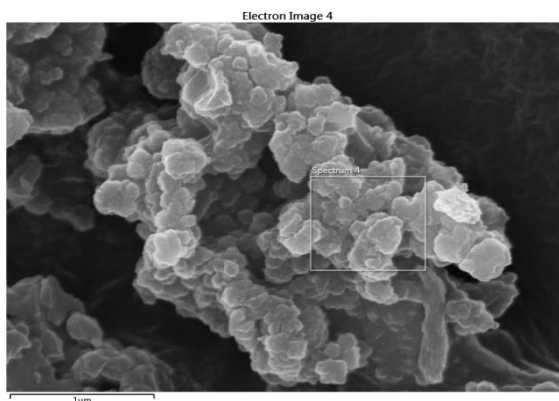


Figure.3 SEM of cellulose

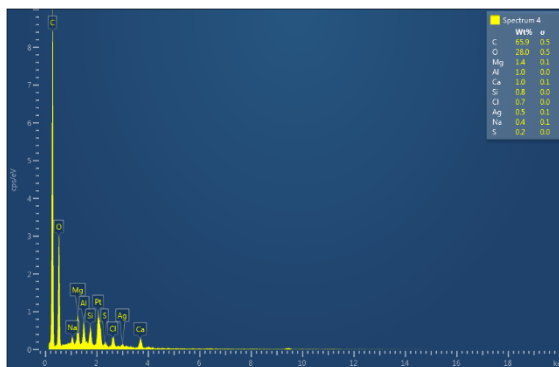


Figure.4 EDX of cellulose

3.4 Hydrogel Tests.

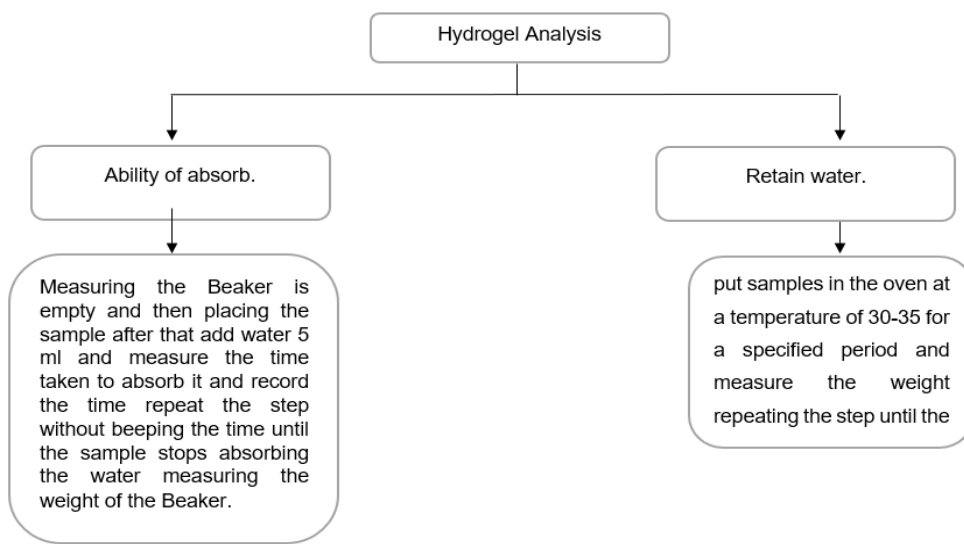


Figure.5 d. Hydrogel tests

3.4.1 Ability to Absorb

In the experiment, 2 ml of distilled water was added to the hydrogel, and the time was recorded once it was observed that the hydrogel had absorbed the entire 2 ml of water. This result demonstrates the water-absorbing capacity of the hydrogel and provides valuable information regarding its swelling behavior. Two different concentrations of Chitosan solution, namely 0.1 g/ml and 0.15 g/ml, were prepared. Each concentration was examined as two separate samples with a mass ratio of 1:2.

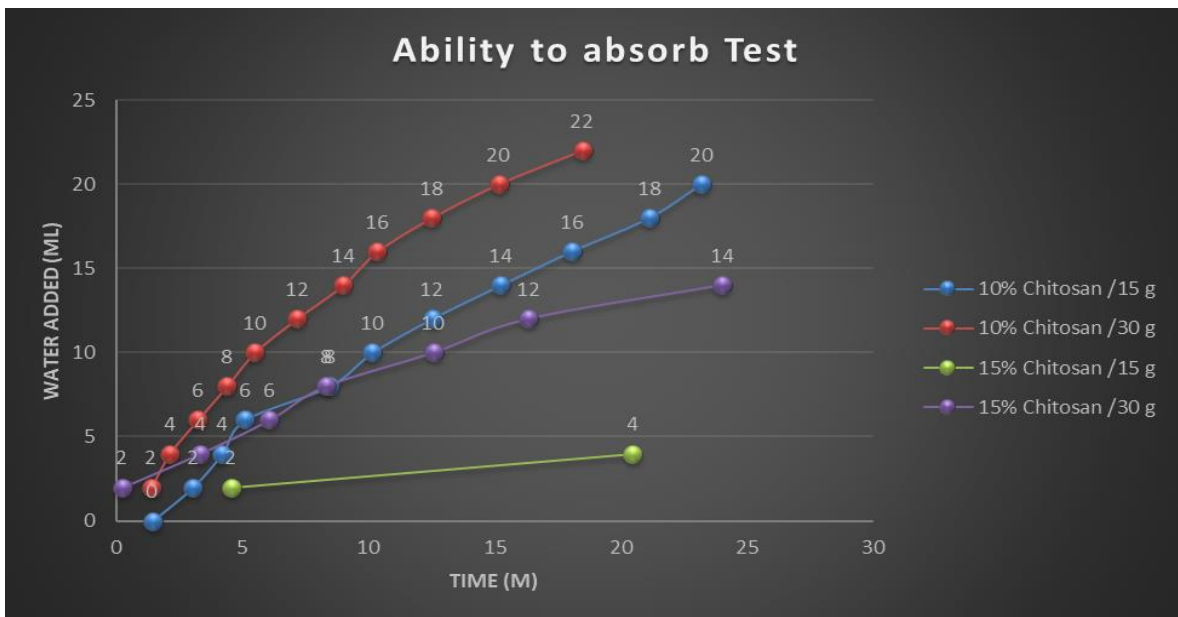


Figure.6 Ability to absorb Test 10% Chitosan



Figure.7 Ability to absorb Test 10% Arabic Gum

3.4.2 Water Retention Capacity

The water retention capacity of the samples was evaluated by measuring their weight after water absorption and subsequent drying in an oven at 30-35°C. Initially, the samples were weighed after absorbing water to obtain their initial mass. They were then placed in the oven, and the weight measurements were recorded at 10-minute intervals. The recorded weights allowed for tracking the loss of water over time and provided insights into the samples' ability to retain water. The data obtained from these measurements demonstrated the water retention properties of the samples under controlled drying conditions, highlighting their effectiveness in retaining moisture.

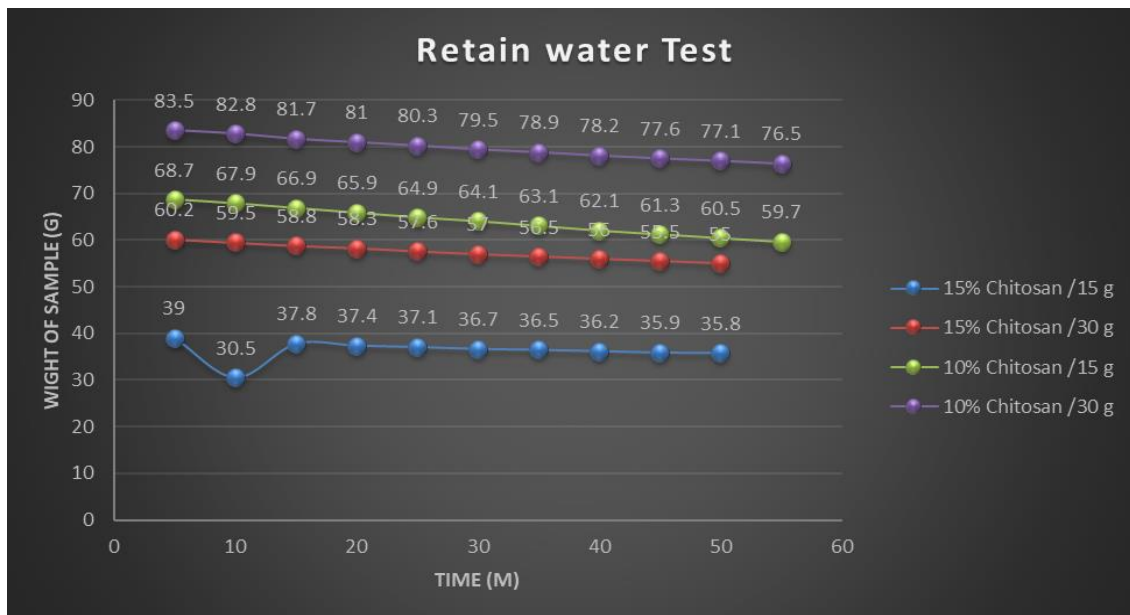


Figure.8 Retain water Test Chitosan

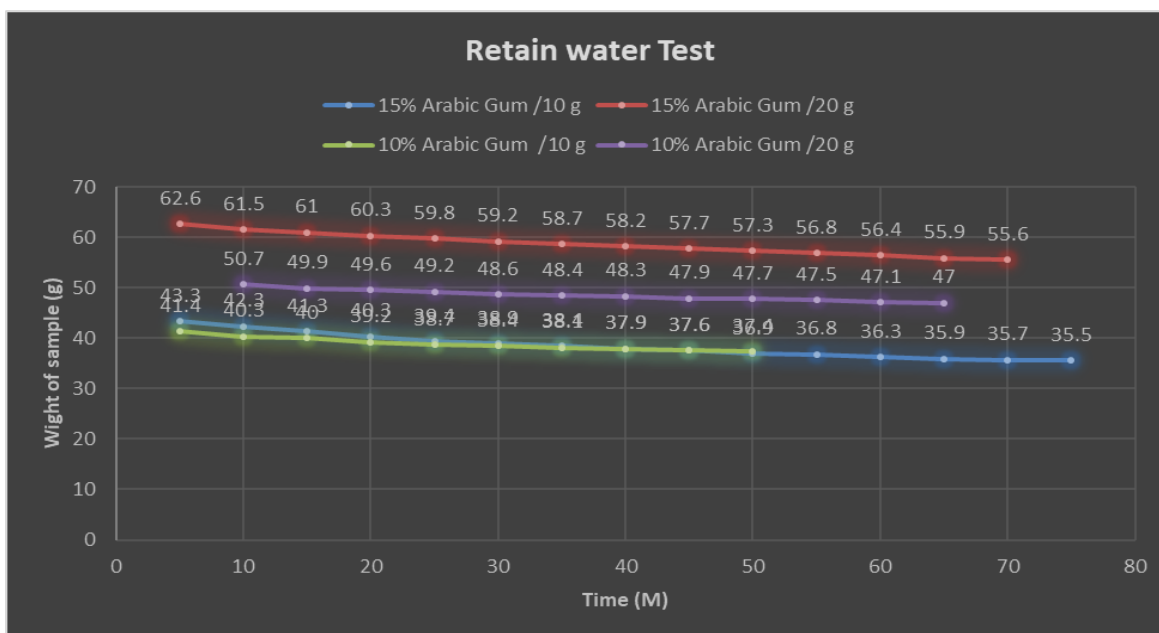


Figure.9 Retain water Test Arabic Gum

The data obtained from the experiments reveals a notable relationship between the amount of Chitosan (at a 10% concentration) used (either 15g or 30g) and the absorption time. It is evident that higher amounts of Chitosan lead to longer absorption times. Interestingly, when comparing the absorption ability of 15% Chitosan with 30g and 15g, it was observed that the former exhibited a higher absorption capacity. This discrepancy warrants further attention and analysis to determine the underlying factors contributing to this difference. Regarding Arabic Gum, the results indicate that the absorption time is generally consistent at around 30 minutes for all quantities and concentrations tested. However, an exception was noted for 15% Arabic Gum with 20g, where the data showed a remarkably quick absorption within approximately 5 minutes, resulting in the absorption of approximately 16 mL. This rapid absorption rate observed for this specific combination of concentration and quantity suggests a potential enhancement in the absorption properties of the material.

These findings highlight the influence of the amount and concentration of Chitosan and Arabic Gum on their respective absorption behaviors. The variations observed in the absorption times and capacities emphasize the need for further investigation to understand the underlying mechanisms and optimize the formulation parameters for enhanced absorption performance. Additional experiments and analyses are warranted to gain deeper insights into these observations and explore the potential applications of Chitosan and Arabic Gum in absorption-related fields.

CONCLUSION

In conclusion, this research project successfully yielded 3.5 grams of nanocellulose from 10 grams of raw date palm leaves. The analysis techniques, such as FTIR and XRD, provided valuable information about the chemical composition and crystalline structure of the nanocellulose. The SEM images revealed cellulose nanoparticles of varying sizes and shapes, while the EDX analysis confirmed their composition. The hydrogel tests demonstrated the hydrogel's ability to absorb and retain water, with absorption times influenced by the concentration and amount of Chitosan and Arabic Gum used. These results offer insights into the properties and potential applications of nanocellulose-based hydrogels, contributing to water management and agricultural sustainability. Further research is needed to optimize formulation parameters for improved absorption performance.

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DOI: <https://doi.org/10.15379/ijmst.v10i3.3359>

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