SYNTHESIS AND CHARACTERIZATION OF NANO-SIZED HYDROGELS FROM SEED EXTRACT AND IT'S ANTIBACTERIAL ACTIVITY

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ABSTRACT

Hydrogels are a form of polymeric material that has a hydrophilic structure, allowing them to hold large volumes of water in three-dimensional networks. The ability to absorb water is attributed to hydrophilic functional groups attached to the polymeric backbone. The majority of investigations have focused on hydrogel synthesis utilizing chemical techniques. In this study, we replaced harmful chemicals with plant-based alternatives and effectively produced hydrogels from halim seeds. Hydrogels can be chemically linked by covalent bonds, physically coupled through non-covalent interactions, or both. Capillary, osmotic, and hydration forces are necessary for water sorption, which are balanced by the forces provided by the cross linked polymer chains to prevent expansion. The cross-linked hydro gel polysaccharide base hydrogel networks were created utilizing a redox initiating free radical polymerization technique with halim seeds and flaxseed as grafting backbones. Potassium persulphate as an initiator, Borax as a cross-linker, and N,N,N',N'-tetramethylethylenediamine (TEMED) as the binding agent and redox initiator pair. We selected halim seed and flax seed, which have carbohydrates as a key component and are used to produce hydrogel nano particals, were analyzed using many techniques such as UV-Visible, fluorescence studies, FT-IR, SEM, EDAX, and XRD. To this the absorbing capacity and swelling test also conducted. The UV-Visible shows peak at 458nm for Halim seed. The fluorescence studies indicate that halim seed have more intense emission qualities 467nm. The XRD results imply that the average size of Hydrogel nanoparticle is 5.5733nm. The antibacterial impact investigations demonstrate that the nanoparticle has a high zone inhibition against the selected pathogens. Halim seed has higher absorbing capacity and swelling ratio.

Keywords: Hydrogel nanoparticals, Halim seed, Polymer, Antibacterial activity.

1. INTRODUCTION

Hydrogels are a type of polymeric material with a hydrophilic structure that allows them to store huge amounts of water in their three-dimensional networks. Hydrogels absorb water due to hydrophilic functional groups connected to the polymeric backbone, but their resistance to dissolution is due to cross-links between network chains. Many materials, both natural and manmade, match the concept of hydrogels [1]. Because of its high water content, hydrogels characteristics are similar to those of biological tissues, resulting in great biocompatibility .The magnitudes of these opposing actions establish the equilibrium swollen state, which dictates several critical aspects of the hydrogel, such as internal transport and diffusion characteristics and mechanical strength. In this work, we successfully created hydrogels from halim seeds by substituting toxic chemicals with plant-based alternatives [2].Crosslinking can occur in two settings: in vitro, during the synthesis of a hydrogel, and in vivo (in-situ), following application to a specific area on the human body. To begin chemical crosslinking, a low molecular weight crosslinking agent and a polymer must be added to the reaction mixture. The hydrophilic linear polymer chains dissolve in water in the absence of crosslinking sites due to the polymer chain and water thermodynamic compatibility. Nonetheless, in the presence of crosslinking points, solubility is counterbalanced by the retractive force of the elasticity of the crosslinking points in the network [3].Natural polymers which create hydrogels include polysaccharides like starch, alginate, and agarose, as well as proteins like collagen and gelatine. Chemical polymerization techniques are typically used to prepare synthetic polymers that result in hydrogels. We use halim seeds to make hydrogels because they are high in fiber and carbohydrates and have good compatibility and biodegradability.

2. MATERIALS AND METHODS

2.1 MATERIALS

- Halim seeds
- Borax powder (98% purity, LR grade Spectrum)
- Potassium persulphate (PPS) (98% purity, LR grade Spectrum)
- N, N, N’, N’-tetramethyl ethylenediamine (TEMED) (99.5% purity LR grade SLR)
- Double distilled water (DDW)

These materials were used for the synthesis of hydrogel and for the preparation of solutions required in this study.

2.2 METHODS

2.2.1 Synthesis of Hydrogel from Halim seed

5g of halim seed was weighed and washed well with distilled water. It was boiled with double distilled water for 15 minutes and allowed to cool. The seeds were squeezed with clean white cloth and the gel was collected. Then it was concentrated by heating at 60 ºC for 10 minutes to remove excess amount of water. About 5g of sample was mixed with initiator PPS (0.05 g in 1 mL of DDW) and cross-linker Borax powder (0.10 g in 1mL of DDW). The reaction mixture was heated at 60 ºC for 5 minutes with occasional stirring. To this reaction mixture TEMED (1 mL) was added, which acts as redox-initiating pair and initiates free radical polymerization along with PPS. The reaction mixture was sonicated about 60 ºC for 10 minutes. The polymerization reaction results in the formation of hydrogel within 10 minutes of reaction time. The prepared hydrogel was equilibrated with water for 3 days to remove unreacted monomers and reagents. The hydrogel was dried in hot air oven.

3. RESULTS AND DISCUSSION

3.1 UV-Analysis

The UV-Visible spectrum for HH was observed in the 200-900 nm region. The particle exhibits three absorption peaks at **220 nm, 367 nm, 458nm** for HH [4]. The synthesized hydrogel shows n– π* due to C-N bond [5] and π-π* due to C=O bending and C=C twist [6] transition states of FH and HH are indicated by the UV-visible absorption peak.

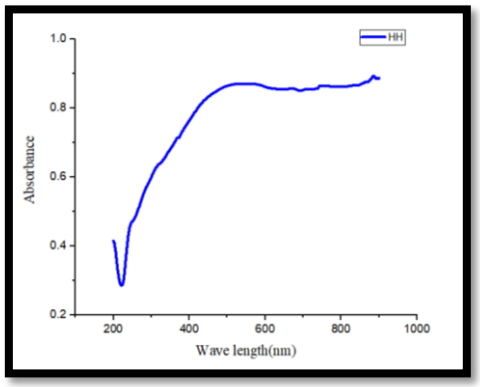

Fig 3.1.a UV-Visible Spectra of HH (Absorbance Vs Wavelength)

3.2 BAND GAP ENERGY

The absorption maxima in a nanomaterial could provide information about its band gap energy. From calculation the band gap energies for HH are **3.3787 eV** and **2.707eV.** The band gap energy > 1.5 to 3.0 belong to semiconductors. As a result, the hydrogels that was created act as a semiconductors [7].

3.3 Fluorescence Spectroscopy

The fluorescence graph for synthesised hydrogel nanoparticles recorded between the range 200nm-900nm. The high intensity **444.235** excited at **467nm**for HH. Form the emission spectral studies the samples FH has high emission property compare to HH [8].

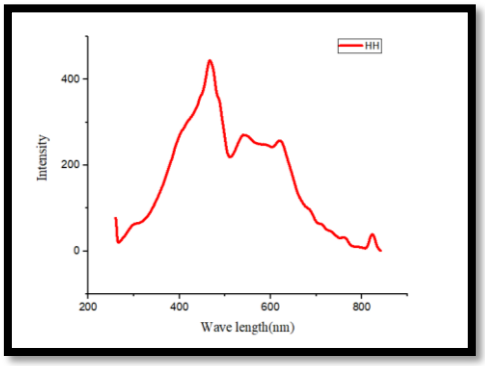

Fig 3.3.a Fluorescence Spectra of FH and HH (Intensity Vs Wavelength)

3.4 FTIR studies

The hydrogel synthesis of HH was identified using FT-IR. Samples have been analyzed between 400 and 4000 cm^{-1}(Fig 5.2.a &5.2.c). The peak stretching in HH at 3842.20 cm^{-1} might be interpreted as an O-H bond [9]. The N-H bending vibration may be seen in the vast bond spots for HH at 1543.50 cm^{-1}[10].The C-O bending vibration is represented by the peak at 972.12 cm^{-1}[11].

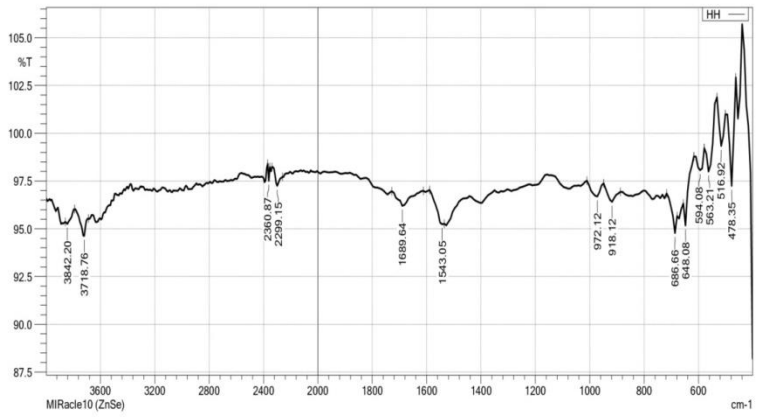

Fig 5.3.a FT-IR Spectrum of HH

3.5 XRD Calculation

The average crystallite size (D) was calculated using the well known Scherer's formula

$$D = K\lambda / \beta\cos\theta$$

$$\beta = (\pi /180)\ 2\theta$$

The average crystallite size (D) of synthesized nanoparticle HH is **5.5733nm.**

For HH, the lattice value $a=\lambda/2\sqrt{K}$

$$a = 1.5406/2\sqrt{0.0182}$$

$$a = 5.7101\ A°$$

The phase composition and crystal structure for HH is characterized by X-ray diffraction, using Cu-Ka1 radiation 1.541874 Å in the range of 10.188º - 80.160º. In FH the peaks corresponding to 30.7, 44.7 and 55.7 were attributed to the hkl values of (102), (202) and (212) and for HH, the peaks corresponding to 30.5, 31.0 and 44.8 were attributed to the hkl values of (200), (121) and (202) .The crystal phase synthesised hydrogel of HH is orthorhombic with a lattice parameter of about a = 5.84234 Å, b= 8.12817 Å and c= 5.66218 Å respectively, accordance withstandard JCPDS cards no.96-430-3944 [12] . It is also matched with other JCPDS cards such as 96-101-1020 [13], 96-350-0065 [14], 96-150-6831[15].

According to the XRD results the lattice values for the synthesised nanoparticals HH was **5.7101 *A*°**. The structure of HH corresponds to the face center cubic. (FCC) [16].

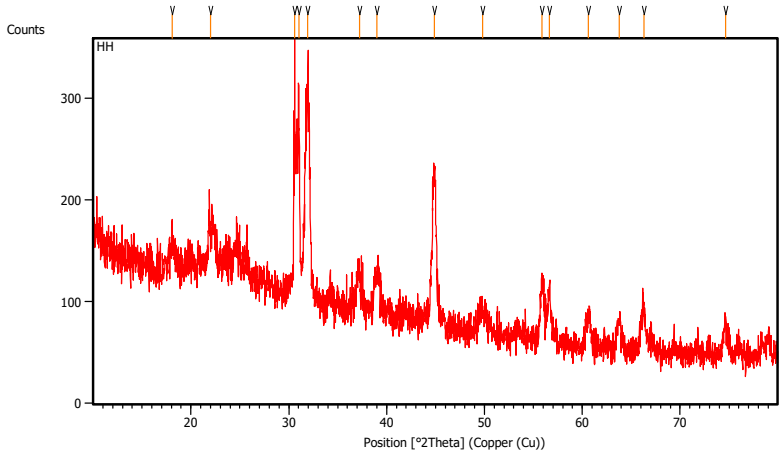

Fig 3.5.a XRD Spectrum of HH

3.6 SEM Characterization

The Field Emission Scanning Electron (FE-SEM) can be used to measure the morphology and size distribution of HH nanoparticals. The average size ranges from 20 um to 500 nm. HH has a needle structure [17]. SEM image for samples HH is given at different magnification.

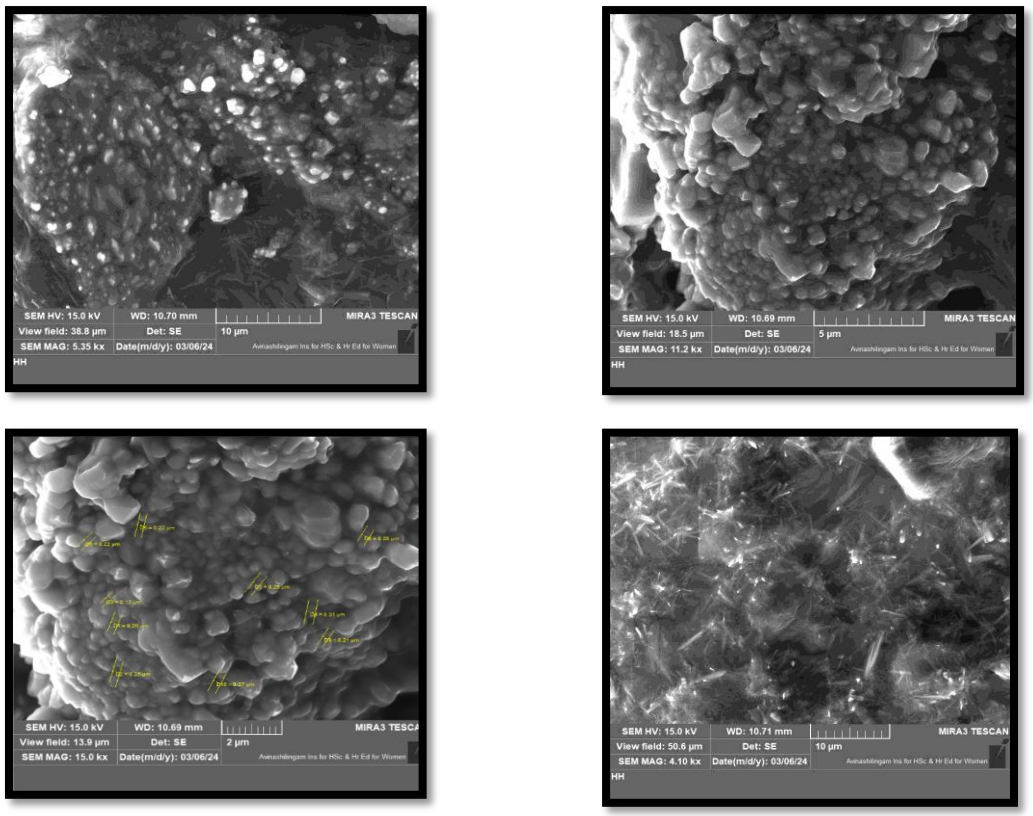

Fig 3.6.a. a, b, c, d SEM images of FH at various magnifications such as 10µm, 5 µm, 2 µm, and 1µm

3.7 EDAX Analysis

The Energy Dispersive X-RaySpectroscopy (EDAX) identifies the elemental composition of hydrogels in two samples. The data show that oxygen is present in higher concentrations. Potassium and carbon have been identified in higher concentrations near oxygen than other elements such as potassium, sodium, and phosphorus. Calcium is also present because it is naturally contained halim seeds.

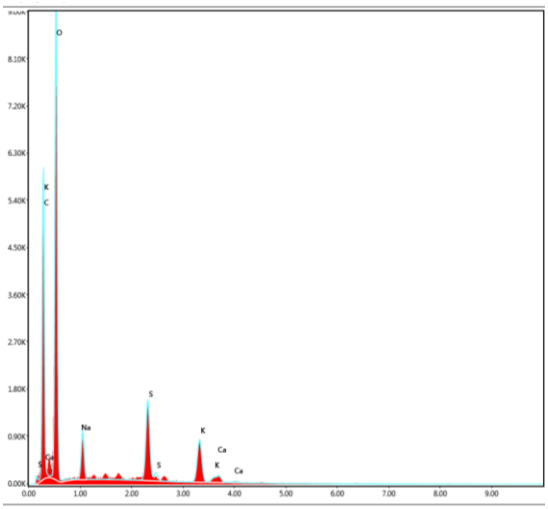

Fig 3.7.a EDAX spectrum for HH

3.8 Absorption capacity

The absorption capacities of HH hydrogel is determined and studied. HH has a maximum absorption capability.

3.9 Swelling Ratio

The swelling ratio is the fractional increase in the weight of the hydrogel caused by water absorption. The swelling capacity of HH is determined by their ability to expand inside a specific solution volume. While the swelling process of HH depend on the availability of hydrophilic functional groups, cross-linking levels, polymer porosity, and network flexibility. The HH has a maximum swelling ratio.

3.10 Antibacterial activity

3.10.1. Composition of Muller Hinton Agar Media

Beef Extract	: 02.00 g
Acid Hydrolysate of Casein	: 17.50 g
Starch	: 01.50g
Agar	: 17.00 g
PH	: 7.3 0.

The analysis included both Gram-negative like *Pseudomonas aeruginosa* and *Escherichia coliand* Gram-positive bacteria like *Staphylococcus aureus, Bacillus subtilis, Bacillus cereus*. HH has greater antibacterial activity .

4. CONCLUSION

Hydrogel was created utilizing a green methodology, utilizing Flax seeds and Halim seeds from sustainable sources. Using EDAX, XRD, and UV, FTIR, and SEM, hydrogel was analyzed. Additionally use antibacterial activity (Disc diffusion method) to ascertain the hydrogel nanoparticle's microbial activity

- ✓ The π-σ* and π-π* transition states of HH is indicated by the UV-visible absorption peak.
- ✓ Higher emission properties are exhibited by the HH at 265, nm, 467nm, 539nm, 803nm.
- ✓ The FT-IR studies reveal N-H bending, C=C twist, C≡N amide bond and O-H stretching in HH and the band gap energy reveal that the synthesised hydrogel is act as a semiconductor.

- ✓ XRD reveals that the hydrogels that were generated is amorphous in nature. HH have crystal size of 5.7101nm. This verifies that the synthesized hydrogel exhibited a nanometer range.
- ✓ The SEM results show that HH have a structure that resembles a needle.
- ✓ Carbon 26% and oxygen 46% in HH were detected by EDAX analysis.
- ✓ The synthesised hydrogel HH exhibits inhibition with the five pathogens under study, which is readily explained by the antibacterial activity. HH sample has greater antibacterial activity.
- ✓ The synthesised hydrogel has important characteristics like mechanical strength, absorbance capacity, biocompatibility, and biodegradability.
- ✓ The hydrogel that was created was utilized in a variety of applications such as drug delivery, tissue regeneration, adhesion to moist tissues, prevention of bleeding, imaging contrast, shielding organs or tissues from radiation, and enhancing the biocompatibility of medical implants.

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