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Design, Synthesis, and Structural Characterization of Super Porous Chemically Cross-Linked Hydrogels

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ABSTRACT

In the present study, a guar gum-based hydrogel was synthesized via graft polymerization of acrylic acid onto guar gum using glutaraldehyde as a cross-linking agent. Guar gum, a natural, biodegradable, and biocompatible polysaccharide, was selected as the backbone polymer to enhance the eco-friendly and biomedical potential of the hydrogel. The preparation method was optimized to obtain a stable, homogeneous, and mechanically robust gel network with improved swelling characteristics. The synthesized hydrogel was evaluated for its physicochemical properties, including swelling behaviour, gel fraction, and structural uniformity, to assess its suitability for biomedical applications. The influence of polymer composition and cross-linking density on hydrogel performance was also investigated. The results demonstrate that the guar gum-g-acrylic acid hydrogel exhibits desirable water uptake capacity and structural stability, highlighting its potential as a biodegradable and biocompatible material may use for controlled drug delivery and other pharmaceutical applications.

1. Introduction

Hydrogels have attracted significant attention in recent years as potential solutions to agricultural water management problems [1]. These polymers can absorb water up to several hundred times their own weight and release it gradually into the soil [2]. When incorporated into agricultural fields, hydrogels act as reservoirs that provide water to plants during dry periods, thus improving crop growth and reducing irrigation frequency [3]. Hydrogels are cross-linked polymeric materials capable of absorbing and retaining water within their three-dimensional structures [4]. Their hydrophilic nature allows them to swell in water without dissolving, forming a gel-like consistency [5]. Common examples include polyacrylamide-based, polyvinyl alcohol-based, and natural biopolymer hydrogels such as guar gum and starch blends [6]. The swelling and deswelling properties of hydrogels depend on factors like crosslinking density, polymer composition, pH, and ionic strength of the surrounding medium [7]. A hydrogel is a three-dimensional framework of polymer matrix that broadens in liquid and holds a huge quantity of water even when maintaining structural integrity due to physicochemical cross-linking of the polymerization [8]. To be a hydrate, a substance should comprise water at a rate of 15% of its total weight because of its essential moisture content, hydrogels possess degree of adaptability that is similar to that of normal cells [9]. The frameworks hydrophilicity is due to proximity of hydrophilic meetings, for example, $-NH_2$, $-OH$, $-CONH_2$, $-CONH$ and $-SO_3H$. Physical, chemical and biochemical hydrogels are all possible [10]. Alteration in natural circumstances, such as temperature and ionic levels, pH, such as the production of two materials, causes physical gels to transform from liquid to gel [11]. Organic factors such as photo catalysis or amino acids are utilized in the gel formation of biological hydrogels [12]. Hydrogel's ability to absorb waters comes from hydrogen bonding attached to polymeric backbone, whereas cross-link among structured chains shield them from dissolution [13]. Hydrogels can be made from a variety of products, both natural and synthetic [14]. Natural hydrogels have gradually been replaced by manufactured hydrogels with long assist lives, rising water "Integrated, and excellent tensile quality over the last two centuries [15]. Fortunately, processed polymer usually has well-defined frameworks that may be tweaked to achieve acceptable bioactivity and utility [16]. It also maintains its consistency in conditions of specular highlights and rapid temperature variations [17]. Hydrogels have recently

been described as two- or multi-part structures developed from three-dimensional network of polymer and liquid that fills the gap among macromolecules [18]. These frameworks in equilibrium will comprise different amount of water based on characteristics of material used, as well as type and density of device joints; typically, volume component of polymer in hydrated form [19]. It is assumed that processed that plastic that are liquid when in non-cross-linked form can be used to achieve high levels of expansion [20]. Hydrogels can be made in a variety of "traditional, scientific ways. It included yet another methodology such as polymers and simultaneous cross-linking of multipurpose molecules, as well as various advance techniques such as combining poly clusters with receptive assemblies and subsequent cross-linking, possibly simultaneously by reacting adhesives with suitable border specialists [18]. Plastic developer can project and mix particulate matter with nuclear control over shape, such as bridge density, and personalized properties, such as adsorption, structural quality, and produced by microbes' response to improve performance [20]. Hydrogels may also have different types of physical forms given in Table 1.

Table 1 Different physical forms of hydrogel

| Physical forms | Examples |
|-------------------------|---|
| Solid molded | Sots contact lenses |
| Pressed powder matrices | Pills or capsules for oral ingestion |
| Micro particles | As bio adhesive carriers or wound treatments |
| Coatings | On implants or catheters; on pills or capsules; or coatings on the inside capillary wall in capillary electrophoresis |
| Membranes or sheets | As a reservoir in a transdermal drug delivery patch; or for 2D electrophoresis gels |
| Encapsulated solids | In osmotic pumps |
| Liquids | That forms gels on heating or cooling |

2. Experimental Methods

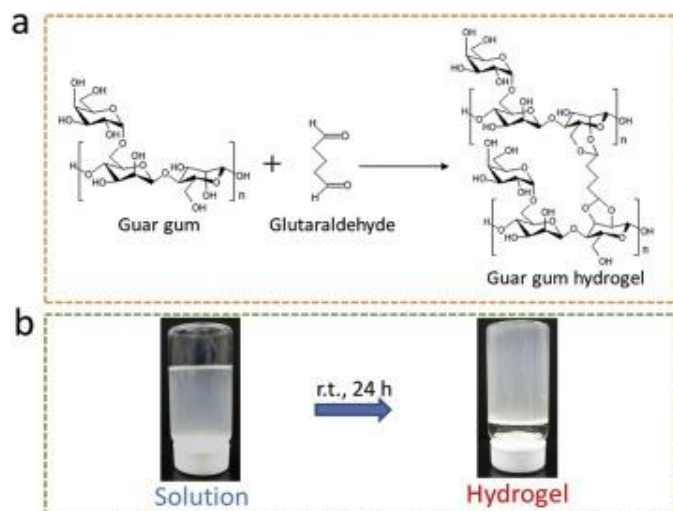
2.1 Hydrogel Synthesis by Glutaraldehyde Crosslinking (Scheme 1)

Guar gum (GG) powder used as the natural polysaccharide base material for hydrogel synthesis. Glutaraldehyde (GA) was employed as the chemical crosslinking agent (Scheme 1). All reagents were of analytical grade and used without further purification. Distilled water was utilized throughout the experimental work. The present work aims to synthesize and characterize hydrogel for agricultural applications using guar gum as the polymer and glutaraldehyde as the crosslinking agent. Guar gum-

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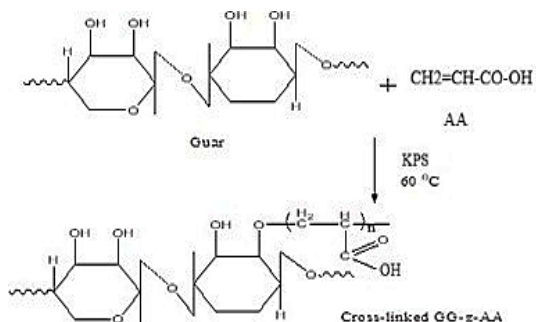
based hydrogel was synthesized by a chemical crosslinking method. Initially (0.5 g) of gaur gum was dispersed in (20 mL) of water and stir magnetically for 1 hour to obtain a uniform and viscous polymer solution. Separately, a glutaraldehyde solution was prepared by diluting (2 mL) of GA with (10 mL) of distilled water the diluted glutaraldehyde was then added dropwise to the guar gum solution under continuous stirring to ensure uniform crosslinking. The reaction mixture was maintained for 4-5 hours at room temperature to allow complete network formation between gaur gum chains and glutaraldehyde molecules. The appearance of a gel-like structure indicated successful hydrogel formation. The synthesized hydrogel was then allowed to stabilize at ambient conditions before being subjected conditions before being subjected to further drying and characterization studies.



Scheme 1 Preparation of gaur gum hydrogel using glutaraldehyde crosslinking

2.2 Hydrogel Synthesis by Free Radical Polymerization (Scheme 2)

Acrylic acid (AA), guar gum (GG), glutaraldehyde (GA), potassium persulfate (KPS), sodium hydroxide (NaOH), and polyvinyl alcohol (PVA) were used in the present scheme (Scheme 2). All reagents were of analytical grade and used as received without further purification. Distilled water was used throughout all experimental procedures.



Scheme 2 Mechanism of acrylic acid grafting onto guar gum and formation of cross-linked GG-g-AA

The hydrogel was prepared by free radical polymerization. Potassium persulfate (0.05 g) was dissolved in (50 mL) of distilled water and thermally decomposed at 60 °C to generate free radicals. A neutralized acrylic acid (AA) solution (pH 7.2-7.4) was prepared by dissolving the required quantity of AA in (10 mL) of 8.0 mol/L NaOH solution. The appropriate amount of acrylate monomer and a predetermined quantity of guar gum were added to this solution and stirred for 2 h in a 250 mL flat-bottom flask (designated as solution A). The KPS solution was then added to solution A, and the mixture was stirred at 60 °C for 30 min (forming solution B). glutaraldehyde, serving as the crosslinking agent, was dissolved in (2-3 mL) of distilled water and added dropwise into solution B under constant stirring until a homogenous dispersion was obtained. The resulting reaction mixture was poured into beaker whose inner surfaces were coated with a thin layer of polyvinyl alcohol (PVA) to facilitate easy removal of the hydrogel rods. The beaker was then placed in water bath maintained at 60 °C for 2 h to complete the polymerization reaction. These were immersed in distilled water for 24 h to remove any unreacted components, followed by drying at room temperature for 12 h and further drying in a vacuum oven at 50 °C until constant weight was achieved.

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3. Results and Discussion

3.1 Characterization of Hydrogel Synthesized via Crosslinking (Scheme 1)

3.1.1 FT-IR Spectroscopy Analysis

Fourier transform infrared spectroscopy (FTIR) is an essential analytical technique used to investigate the chemical structure, bonding interactions, and functional group modifications in polymeric hydrogels. In the present study, FTIR analysis was performed to confirm the formation of cross-linked networks between guar gum and glutaraldehyde. This technique provides direct evidence of chemical interactions, such as the formation of acetyl or ether linkages, hydrogen bonding, and the disappearance or shifting of characteristic absorption peaks.

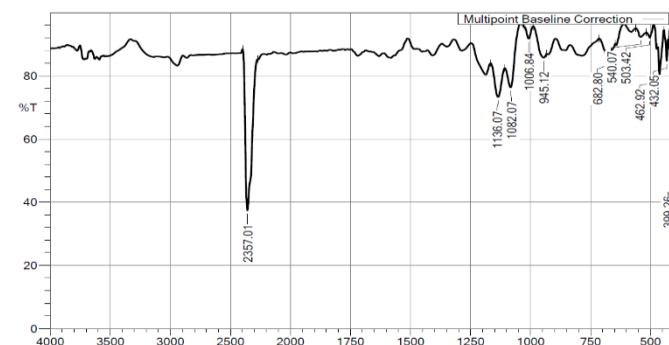


Fig. 1 FT-IR spectrum of the Scheme 1 hydrogel showing characteristic functional groups and evidence of crosslinking

The FTIR spectrum (Fig. 1) of the prepared hydrogel shows characteristic peaks confirming the successful formation of a cross-linked polymeric network. The absorption peaks observed at 1006.84, 1082.07, and 1136.07 cm^{-1} are attributed to the C-O and C-O-C stretching vibrations, which confirm the presence of ether and alcohol linkages in the polymer backbone. The peaks at 540.07 and 682.80 cm^{-1} correspond to C-O deformation and C-H bending, supporting the formation of a stable cross-linked network. The weak band observed at 2357.01 cm^{-1} is due to the asymmetric stretching of CO_2 or the presence of $\text{C}\equiv\text{N}$ stretching, which may arise from minor atmospheric interactions. The peak around 945.12 cm^{-1} also represents C-H deformation in the polymeric matrix. The presence of these characteristic peaks indicates that crosslinking has occurred between the hydroxyl groups of guar gum and the aldehyde groups of glutaraldehyde, resulting in the formation of an ether linkage and a three-dimensional hydrogel network. Overall, the FTIR analysis confirms the successful synthesis of the hydrogel with all expected functional groups present, supporting the effective interaction between polymer and cross linker.

3.1.2 UV-Visible Spectral Study

UV-visible spectroscopy is a simple yet powerful analytical technique used to study the optical properties, electronic transitions, and structural interactions in polymeric and hydrogel materials. In this study, UV-vis analysis was performed to examine the absorption characteristics of the synthesized guar gum hydrogel cross-linked with glutaraldehyde. It helps to confirm the formation of cross-linked structure, the presence of chromophore or conjugated groups, and optical transparency or absorbance changes caused by blending and crosslinking.

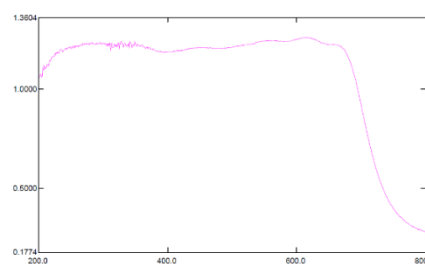


Fig. 2 UV-visible absorption spectrum of the Scheme 1 hydrogel indicating electronic transitions within the polymeric network

The UV-visible spectrum of the prepared hydrogel sample was recorded in the wavelength range of 200–800 nm using a UV-2600 spectrophotometer (Fig. 2). The spectrum shows a major absorbance peak in the UV region, indicating the electronic transitions occurring within the polymeric network. The strong absorption in the lower wavelength region

(around 230–300 nm) corresponds to the $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ electronic transitions, which are typically associated with the presence of carbonyl (C=O) and unsaturated (C=C) groups present due to glutaraldehyde crosslinking and polymer backbone structure. The overall spectral pattern indicates that the hydrogel possesses good optical transparency in the visible region and strong UV absorption, suggesting its potential use in biomedical and environmental applications such as UV-blocking or controlled drug delivery systems. This result confirms that the prepared hydrogel network has been successfully synthesized with stable electronic transitions indicative of proper crosslinking and polymer formation.

3.1.3 XRD Analysis

X-ray diffraction (XRD) is a powerful analytical technique used to study the crystalline and amorphous nature of materials. In the present study, XRD analysis was performed to evaluate the structural arrangement, degree of crystallinity, and effect of crosslinking on the guar gum-PVA hydrogel network. Since both Guar gum are semi-crystalline or amorphous polymers, crosslinking with glutaraldehyde can alter their molecular packing and crystallinity. XRD helps to confirm these structural changes. The XRD pattern (Fig. 3) of the hydrogel displays a broad diffraction peak centered around $2\theta = 20^\circ$ – 30° , which is characteristic of semi-crystalline polymeric hydrogels. The presence of this broad hump indicates the coexistence of both amorphous and slightly ordered regions within the hydrogel matrix. The observed pattern suggests that crosslinking within the hydrogel network resulted in partial alignment of the polymer chains, enhancing structural stability and compactness. This semi-crystalline behaviour confirms the successful formation of the hydrogel structure with improved mechanical integrity and controlled water-holding capacity, which are essential for its potential applications in biomedical and environmental fields.

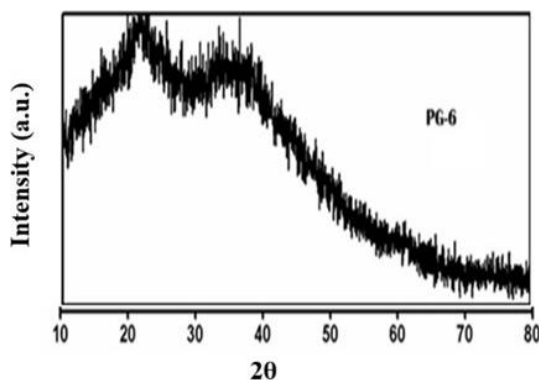


Fig. 3 XRD pattern of the hydrogel synthesized under Scheme 1

3.1.4 FE-SEM Analysis

Field emission scanning electron microscopy (FE-SEM) is an important tool to analyse surface morphology and structure of materials on Nano scale. FE-SEM uses the source which is field emission electron gun which produces high energy and highly focused electron beam, when these electron beam interact with surface of sample signals are emitted. The signals are detected and converted into the images which gives visualization of surface topography, size and shape of synthesized materials. From FESEM image (Fig. 4), it is confirmed that hydrogel is having porous structure and posing smooth surface area, and observed self-assembled fibrillary networks and their alignment and from FESEM visualizing the size, shape, and interconnectedness of pores, which impacts properties like swelling, diffusion, and cell infiltration for tissue engineering applications in future.

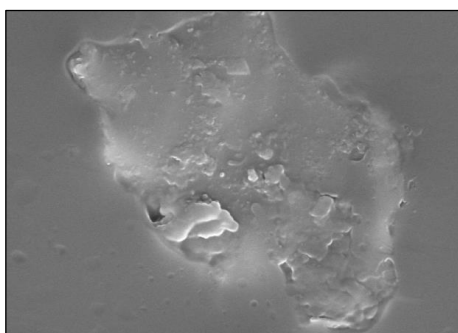


Fig. 4 FE-SEM images of the Scheme 1 hydrogel showing a porous and interconnected network structure

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3.2 Characterization of Hydrogel Synthesized via Free Radical Polymerization (Scheme 2)

3.2.1 FT-IR Spectroscopy Analysis

The FTIR spectrum of the prepared hydrogel sample was recorded in the range of 4000–500 cm^{-1} and shown in Fig. 5. The broad peaks observed at 3714.90 cm^{-1} and 3184.48 cm^{-1} are assigned to the O–H stretching vibrations, which confirm the presence of hydroxyl groups and strong hydrogen bonding within the hydrogel matrix. The absorption band at 2937.59 cm^{-1} corresponds to C–H stretching vibrations of the aliphatic $-\text{CH}_2$ groups present in the polymer backbone. A distinct and sharp peak at 1730.15 cm^{-1} indicates the C=O stretching vibration of carbonyl groups, confirming the successful crosslinking between glutaraldehyde and the hydroxyl groups of guar gum or polyvinyl alcohol. The peaks appearing at 1672.23 cm^{-1} and 1558.48 cm^{-1} correspond to C=C stretching and C–N bending, respectively, which may result from slight conjugation or residual aldehyde interactions after crosslinking. The band at 1404.18 cm^{-1} is attributed to C–H bending, suggesting the presence of aliphatic components in the polymer structure. Strong absorption bands at 1278.81 cm^{-1} , 1186.32 cm^{-1} , and 1086.92 cm^{-1} are due to C–O–C and C–O stretching vibrations, which confirm the formation of ether linkages and alcohol functionalities characteristic of the hydrogel's polymeric network. Additional peaks at 956.68 cm^{-1} and 848.08 cm^{-1} are assigned to C–O–H deformation and C–H wagging vibrations, respectively, while the lower wavenumber peaks at 644.08 cm^{-1} , 507.28 cm^{-1} , and 470.63 cm^{-1} correspond to C–O bending or M–O vibrations, indicating structural stability of the cross-linked matrix. Overall, the FTIR analysis confirms the presence of key functional groups such as hydroxyl (–OH), carbonyl (C=O), ether (C–O–C), and aliphatic (C–H) linkages. These characteristic bands clearly demonstrate the successful crosslinking reaction between the polymer and glutaraldehyde, leading to the formation of a stable, three-dimensional hydrogel network.

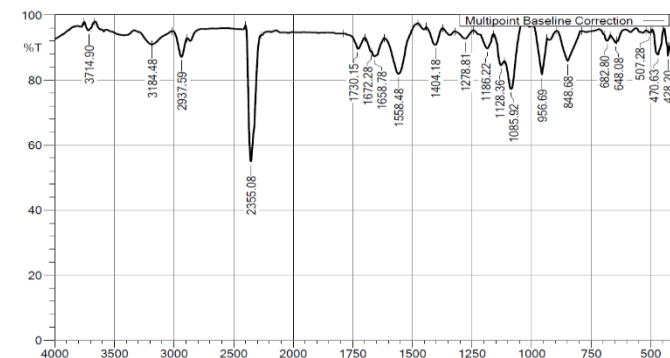


Fig. 5 FT-IR spectrum of the Scheme 2 hydrogel showing characteristic functional group interactions and crosslinking

3.2.2 UV-Visible Spectral Study

The UV-visible spectrum of the synthesized hydrogel was recorded as shown in Fig. 6 and the obtained spectrum exhibited a broad absorption band in the ultraviolet region (200–400 nm) with a distinct absorption peak at 324 nm showing an absorbance of 1.3229. This broad absorption behaviour is characteristic of hydrogels containing organic components or dispersed nanoparticles within the polymeric matrix. The presence of a prominent peak at 324 nm suggests the existence of chromophore groups responsible for electronic transitions. These may correspond to $\pi \rightarrow \pi^*$ transitions of conjugated or aromatic structures and $n \rightarrow \pi^*$ transitions of carbonyl or imine groups associated with the polymer network or cross linker. If plant-based extracts were involved, the observed peak can also be attributed to phenolic or flavonoid compounds acting as natural reducing and stabilizing agents. Hence, the UV-vis analysis confirms the successful incorporation of functional molecules or nanoparticles within the hydrogel matrix, contributing to its optical activity and stability.

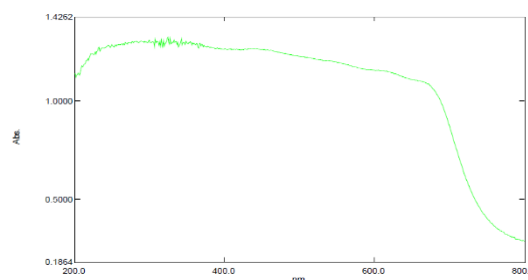


Fig. 6 UV- Visible of the hydrogel prepared via Scheme 2

3.2.3 XRD Analysis

The XRD spectrum (Fig. 7) exhibits a broad and diffused hump in the 2θ range of approximately 10° – 50° , indicating the amorphous nature of the material. The absence of sharp and intense peaks confirms that the sample lacks long-range crystalline order and is composed mainly of randomly oriented polymer chains. Such an amorphous structure is commonly observed in natural polymers or uncrosslinked polymeric materials and is associated with high flexibility and the ability to absorb water efficiently. The broad nature of the peak suggests that the material possesses a disordered molecular arrangement, which contributes to its soft and gel-like characteristics.

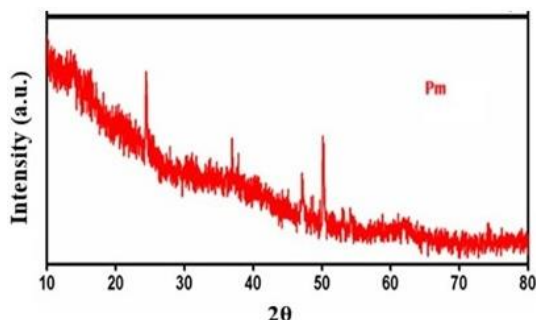


Fig. 7 X-ray diffraction (XRD) pattern of the hydrogel prepared via Scheme 2 showing its amorphous nature

3.2.4 FE-SEM Analysis

From FESEM image Fig. 8, it is confirmed that, synthesised hydrogel is having porous structure and having smooth surface area. Also, it has been observed self-assembled fibrillary networks and their alignment and from FESEM visualizing the size, shape, and interconnectedness of pores, which impacts properties like swelling, diffusion, and cell infiltration for tissue engineering applications in future.

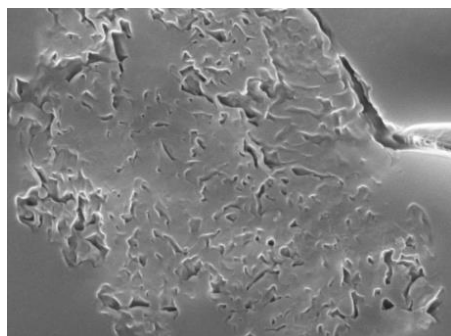


Fig. 8 FE-SEM microstructure of the Scheme 2

4. Conclusion

In the present study, guar gum-based hydrogels were successfully synthesized using glutaraldehyde as a cross-linking agent. Two formulations were developed to evaluate the influence of polymer composition and cross-linking on water absorption behaviour. The blended guar gum-acrylic acid-polyvinyl alcohol (PVA) hydrogel exhibited improved mechanical stability, higher swelling capacity, and enhanced hydrophilicity compared to the directly cross-linked guar gum hydrogel. FTIR analysis confirmed the formation of covalent linkages between polymer chains. Also, UV-visible studies indicated structural modification and uniform network formation. SEM micrographs revealed a porous and interconnected morphology responsible for efficient water

uptake and retention. The results demonstrate that replacing conventional synthetic components such as MBA and silicon oil with glutaraldehyde and PVA provides an eco-friendly, biodegradable, and high-performance hydrogel system. These hydrogels show promising potential for applications in agricultural moisture retention, wastewater treatment, and controlled release systems. Further optimization of cross-linking density, swelling kinetics, and pH-responsive behaviour may broaden their industrial and environmental applicability.

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