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METHACRYLATED CHITOSAN HYDROGEL FOR DENTAL APPLICATIONS

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ABSTRACT :
Chitosan, a naturally derived cationic polysaccharide obtained from chitin, has attracted significant attention in biomedical research due to its biocompatibility, biodegradability, and mucoadhesive properties. Chemical modification of chitosan is often employed to enhance its physicochemical and biological characteristics for tissue engineering and drug delivery applications. In the present study, methacrylated chitosan was synthesized by reacting chitosan with methacrylic anhydride in an acidic aqueous medium. The resulting polymer was purified through dialysis and subsequently freeze-dried. Structural characterization of the modified polymer was carried out using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). Additionally, antimicrobial activity was evaluated using agar diffusion assays. The results indicated successful methacrylation of chitosan, as confirmed by characteristic FTIR peaks corresponding to methacrylate functional groups. SEM analysis revealed a porous morphology suitable for scaffold-based tissue engineering applications. The synthesized methacrylated chitosan demonstrated moderate antimicrobial activity and shows potential for applications in dental tissue engineering, 3D bioprinting, and drug delivery systems. Further studies involving mechanical reinforcement and incorporation of bioactive components are recommended to enhance its performance.

Keywords : Chitosan; Methacrylation; Hydrogel; Dental tissue engineering; SEM; FTIR; Bio-ink

Introduction : Biopolymers derived from natural sources have gained substantial attention in biomedical engineering due to their inherent biocompatibility, biodegradability, and minimal toxicity. Among these, chitosan is one of the most widely studied polysaccharides owing to its unique physicochemical and biological properties (Rinaudo, 2006).

Chitosan is produced through the deacetylation of chitin, a naturally abundant polysaccharide found in crustacean shells and fungal cell walls. Its cationic nature allows interaction with negatively charged biological membranes, providing good mucoadhesive properties and antimicrobial activity (Dash et al., 2011). These characteristics make chitosan particularly useful for drug delivery systems, wound healing, and tissue engineering applications.

In dentistry, biomaterials with biocompatibility, biodegradability, and antimicrobial properties are essential for regenerative treatments such as periodontal regeneration, pulp tissue engineering, and oral drug delivery. Chitosan-based hydrogels have been explored extensively for such applications as they can mimic the extracellular matrix and support cell adhesion and proliferation (Jayakumar et al., 2010).

Despite its advantages, native chitosan exhibits certain limitations including poor solubility at neutral pH, limited mechanical strength, and restricted structural stability. Chemical modification is therefore frequently used to improve its properties. One promising modification strategy is methacrylation, which introduces methacrylate groups into the polymer backbone. These functional groups allow the polymer to undergo crosslinking reactions, enabling the formation of stable hydrogels suitable for 3D bioprinting and tissue scaffolding (Van Den Bulcke et al., 2000).

Methacrylated chitosan derivatives have demonstrated improved mechanical properties, crosslinking ability, and structural stability, making them promising candidates for bio-ink development and tissue engineering scaffolds(Eliashar et al., 2018). Furthermore, such materials may serve as carriers for therapeutic agents and growth factors in dental regeneration.

Overall, methacrylated chitosan hydrogels represent a promising biomaterial for dental applications due to their biocompatibility, antimicrobial properties, tunable mechanical characteristics, and ability to support tissue regeneration. Continued research in this field may contribute to the development of advanced therapeutic strategies for oral tissue repair, regenerative dentistry, and improved dental implant success.

Therefore, the present study focuses on the synthesis and characterization of methacrylated chitosan hydrogel for potential applications in dental tissue engineering and drug delivery systems.

2. Aim: The aim of this study was to synthesize and characterize methacrylated chitosan hydrogel and evaluate its potential as a bio-ink for dental tissue engineering applications**.**

3. Materials and Methods

3.1 Materials : Low molecular weight chitosan was used as the base biopolymer. Methacrylic anhydride served as the modifying reagent for introducing methacrylate functional groups. Acetic acid (1% v/v) was used to dissolve chitosan. Dialysis membranes with a molecular weight cutoff of 14 kDa were used for purification.

3.2 Preparation of Chitosan Solution: Approximately 0.5 g of chitosan was dissolved in 1% (v/v) acetic acid solution under continuous stirring until a homogeneous solution was obtained.

3.3 Synthesis of Methacrylated Chitosan: Methacrylated chitosan was synthesized using a modified protocol adapted from previously reported methods for the methacrylation of chitosan polymers (Eliyahu et al., 2018).

Initially, 0.5 g of chitosan was dissolved in 1% (v/v) acetic acid solution under continuous magnetic stirring until a homogeneous solution was obtained. Subsequently, 1 mL of methacrylic anhydride was added dropwise to the chitosan solution while maintaining constant stirring to ensure uniform reaction throughout the solution. The reaction mixture was maintained at 40°C for approximately 12 hours, allowing the methacrylation reaction to proceed between the amine groups of chitosan and methacrylic anhydride. Following the reaction, the obtained solution was transferred into a dialysis membrane with a molecular weight cut-off of 14 kDa and dialyzed against distilled water for four days to remove any unreacted reagents and by-products.

After purification, the dialyzed solution was freeze-dried, resulting in a white porous methacrylated chitosan polymer suitable for further characterization and analysis.

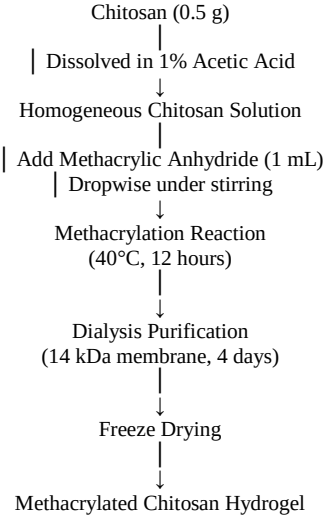

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Figure 1. Schematic representation of the synthesis of methacrylated chitosan through the reaction of chitosan with methacrylic anhydride followed by dialysis purification and freeze-drying.

3.4 Dialysis and Purification

The synthesized polymer solution was transferred into a 14 kDa dialysis membrane and dialyzed against distilled water for four days to remove unreacted reagents and by-products.
After dialysis, the purified solution was freeze-dried to obtain methacrylated chitosan in solid form.

4. Characterization

4.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was performed to identify functional groups present in the synthesized polymer and confirm the successful introduction of methacrylate groups into the chitosan backbone.

4.2 Scanning Electron Microscopy (SEM)

SEM analysis was used to evaluate the **surface morphology and micropstructure** of the synthesized methacrylated chitosan hydrogel.

4.3 Antimicrobial Activity

The antimicrobial activity of the synthesized polymer was assessed using the **agar diffusion method**. Samples were placed on microbial culture plates and incubated to observe inhibition zones around the sample.

5. Results and discussion :

5.1) FTIR was used to confirm the successful modification of chitosan through methacrylation. The spectrum of methacrylated chitosan exhibited characteristic peaks corresponding to functional groups associated with chitosan as well as newly introduced methacrylate groups. A broad absorption peak observed around **3270 cm^{-1}** corresponds to O–H and N–H stretching vibrations. The peak at approximately **2870 cm^{-1}** represents C–H stretching vibrations. Peaks observed near **1650 cm^{-1} and 1550 cm^{-1}** correspond to amide I and amide II bands, confirming the presence of the chitosan backbone. Additional peaks between **1150–1020 cm^{-1}** indicate C–O stretching vibrations, which are characteristic of polysaccharide structures. These spectral features confirm the successful modification of chitosan with methacrylate groups.

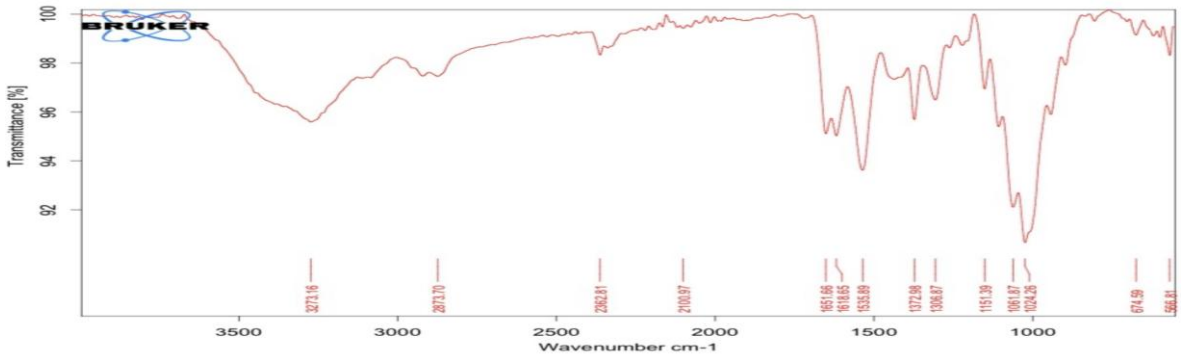

Figure 1. ATR-FTIR spectrum of methacrylated chitosan showing characteristic absorption peaks corresponding to hydroxyl (O–H), amine (N–H), amide (C=O), and polysaccharide backbone vibrations, confirming the successful chemical modification of chitosan

3.2 Morphological and Topographical Characterization Using SEM

The surface morphology of the synthesized methacrylated chitosan was investigated using scanning electron microscopy (SEM). SEM analysis revealed a heterogeneous and porous structure within the polymer matrix.
At a **50 µm scale**, the material exhibited an irregular and interconnected porous structure distributed throughout the surface. Such porous architecture is beneficial for biomedical applications as it allows diffusion of nutrients, oxygen, and cellular infiltration in tissue engineering scaffolds.
At a **higher magnification of 10 µm**, the microstructure revealed detailed surface roughness and micro-scale pores within the polymer network. The presence of interconnected pores indicates that the material may provide an appropriate environment for cell attachment and proliferation, which is desirable for regenerative dental applications.
These morphological characteristics demonstrate that methacrylated chitosan possesses a suitable structure for potential applications in 3D bioprinting, tissue engineering scaffolds, and drug delivery systems.
SEM images of methcrylated chitosan copy (SEM) image of methacrylated chitosan at 50 µm scale, showing the overall porous and heterogeneous morphology of the synthesized hydrogel.

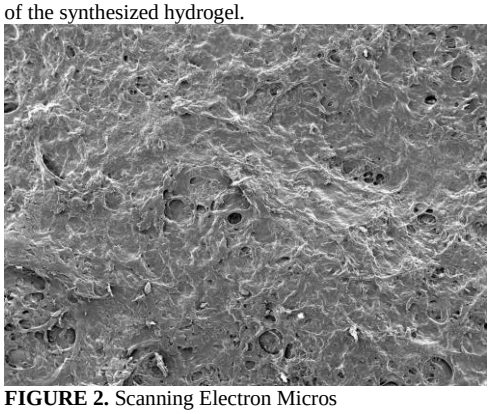

FIGURE 2. Scanning Electron Micros

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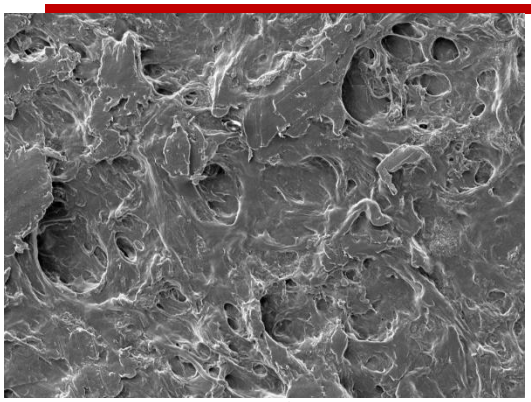

FIGURE 3. SEM micrograph of methacrylated chitosan at 10 um scale, illustrating the detailed microstructure and interconnected pores within the polymer matrix.

The successful synthesis of methacrylated chitosan demonstrates the feasibility of modifying natural biopolymers to enhance their applicability in biomedical engineering.
Methacrylation introduces polymerizable double bonds, enabling crosslinking and hydrogel formation. Such hydrogels are widely used in 3D bioprinting and regenerative medicine because they provide a supportive microenvironment for cell growth (Drury & Mooney, 2003).
SEM observations confirmed the presence of a porous microstructure, which is advantageous for scaffold materials intended for tissue engineering. The porous architecture allows diffusion of nutrients and oxygen, thereby supporting cell survival and proliferation.
The antimicrobial activity observed in this study is consistent with previous reports that chitosan derivatives exhibit antimicrobial effects due to interactions between the positively charged amino groups and microbial cell membranes (Dash et al., 2011).
However, the moderate antimicrobial activity observed suggests that further modifications, such as incorporation of antimicrobial nanoparticles or blending with polymers such as polyvinyl alcohol (PVA), may enhance the material's effectiveness for clinical applications.
Overall, methacrylated chitosan shows promise as a bio-ink material for dental tissue engineering and regenerative medicine.

7. CONCLUSION :

This study successfully synthesized and characterized methacrylated chitosan hydrogel using methacrylic anhydride. The material exhibited suitable structural and morphological properties for biomedical applications.
The findings suggest that methacrylated chitosan could be used for 3D bioprinting, dental tissue engineering, and drug delivery systems.
Further research focusing on mechanical reinforcement and enhanced antimicrobial properties could improve its clinical potential.

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