

"Synthesis of Gold and Silver Nanoparticles Using Cellulose Hydrogels"

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Abstract: This work reports the preparation of a carboxymethylcellulose (CMC)-based hydrogel crosslinked with citric acid and reinforced with microcrystalline cellulose (MCC), which enhances the swelling capacity and structural stability of the polymeric network, allowing the hydrogel to retain its shape and consistency. Citrate ions and polysaccharides have been reported to promote the formation of metallic nanoparticles, such as gold (AuNPs) and silver nanoparticles (AgNPs). Accordingly, this study hypothesizes that the citric acid used as a crosslinking agent, together with the hexose units present in CMC and MCC, can facilitate the in situ formation of AuNPs and AgNPs within the hydrogel matrix.

A green synthesis approach is proposed as an alternative to conventional nanoparticle synthesis methods, including coprecipitation, sol-gel, hydrothermal processes, microemulsions, and chemical reduction. Traditional methods often require complex instrumentation and toxic reducing agents, posing environmental and health concerns. In contrast, the synthesized hydrogel acts simultaneously as a reducing and stabilizing agent, eliminating the need for hazardous chemicals.

Beyond demonstrating efficient metal ion reduction and nanoparticle formation, this approach yields a biodegradable and biocompatible polymeric network with potential applications in biomedical, environmental, and catalytic fields. The metal ion reduction capability of the hydrogel was evaluated using silver nitrate and chloroauric acid trihydrate solutions, while the characteristic functional groups of carboxymethylcellulose were analyzed by Fourier-transform infrared spectroscopy (FTIR-ATR).

Introduction

Hydrogels are three-dimensional polymeric networks capable of absorbing large amounts of water or biological fluids while maintaining structural integrity. Their high-water content and tunable mechanical properties make them suitable for biomedical applications, including wound dressings, drug delivery systems, and tissue engineering scaffolds. Recently, the combination of hydrogels with nanostructures has led to the emergence of nanocomposite hydrogels, which exhibit enhanced mechanical, chemical, and functional properties compared to traditional hydrogels (Schexnailder and Schmidt, 2008).

Among natural polymers, cellulose derivatives have received particular attention due to their abundance, renewability, and biocompatibility. Carboxymethylcellulose (CMC) is a water-soluble cellulose derivative that forms hydrogels with excellent swelling capacity and

biocompatibility. Crosslinking CMC with citric acid yields biodegradable, non-toxic hydrogels suitable for medical and environmental applications (Kokkarachedu et al., 2017). On the other hand, microcrystalline cellulose (MCC), a refined form of cellulose obtained by partial hydrolysis of cellulose fibers, provides rigidity and improved mechanical strength when incorporated into polymeric matrices (Trache et al., 2016).

In this study, we developed and characterized CMC/MCC-based nanocomposite hydrogels crosslinked with citric acid and loaded with natural extracts from Moringa, Paramenteira, and orange. These extracts were used to induce the in-situ synthesis of silver (Ag) and gold (Au) nanoparticles within the hydrogel matrix. The resulting materials were evaluated in terms of their structural, physicochemical, and antimicrobial properties to assess their

potential as green and biocompatible biomaterials.

Methods

Materials

Carboxymethylcellulose sodium salt (CMC), microcrystalline cellulose (MCC), and citric acid were used as the main polymeric and crosslinking components. Natural extracts were prepared from *Moringa oleifera*, *Paramenteira*, and orange peels through aqueous extraction. Silver nitrate (AgNO_3) and chloroauric acid (HAuCl_4) served as metallic precursors for nanoparticle formation.

Preparation of Hydrogels

The CMC-MCC hydrogels were synthesized by dissolving the CMC in distilled water under magnetic stirring until complete homogenization, followed by the addition of dispersed MCC. Citric acid was introduced as a crosslinker, and the mixture was poured into Petri dishes and dried at moderate temperature (50–60 °C) to obtain hydrogel films. The crosslinking reaction proceeded without additional catalysts, relying on esterification between the carboxylic groups of citric acid and the hydroxyl groups of cellulose.

Synthesis of the Metallic Nanoparticles

Hydrogels were immersed in aqueous solutions of metallic salts (AgNO_3 or HAuCl_4) to enable the in situ synthesis of metallic nanoparticles. Subsequently, the samples were exposed to UV radiation for 40 minutes in the case of silver nanoparticles and to microwave irradiation for 10 seconds in the case of gold nanoparticles.

Results and discussion

Hydrogel Synthesis

A 4.00% carboxymethylcellulose (CMC) solution was prepared in acetone under constant stirring until complete dissolution. Distilled water was then added, and the mixture was heated to 80 °C to evaporate the organic solvent. In parallel, a 1% microcrystalline cellulose (MCC) dispersion in acetone was prepared and subjected to

ultrasonic treatment to achieve uniform dispersion. An aliquot of this dispersion was incorporated into the CMC solution under manual stirring to obtain a CMC–MCC mixture. Subsequently, a 5% citric acid solution was added dropwise, and the system was heated to 70 °C with stirring until homogenization. The resulting gel was allowed to rest for 24 h and then dried at 50 °C for 24 h to obtain the final hydrogel.

Hydrogels were obtained as transparent, slightly rough films with moderate flexibility. The presence of MCC reduced the overall flexibility compared to pure CMC hydrogels but enhanced mechanical strength and water retention capacity.

Swelling Behavior

The CMC-MCC hydrogels exhibited a swelling ratio exceeding 105%, indicating a highly hydrophilic structure with improved water uptake capacity. The incorporation of MCC increased the internal porosity and available hydrophilic sites for water absorption. The results provide insight into the influence of microcrystalline cellulose incorporation on the hydrogel network and its hydration dynamics. Figure 1.

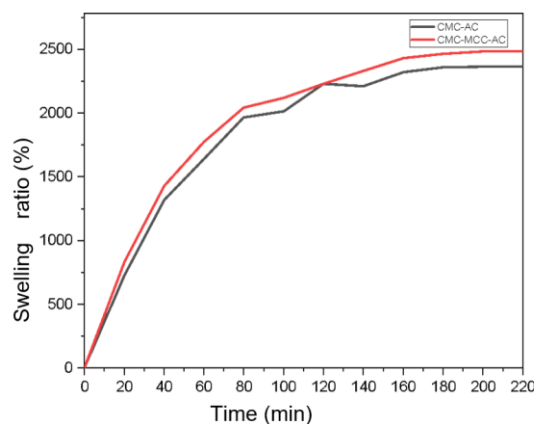


Figure1: Physicochemical characterization of CMC-based hydrogels and their performance as matrices for metallic nanoparticle synthesis. Swelling kinetics (swelling ratio, %) as a function of time for CMC-AC and CMC-MCC-AC hydrogels.

FTIR Analysis

FTIR spectra confirmed the formation of ester linkages between citric acid and the hydroxyl groups of cellulose, evidenced by the appearance of characteristic bands around 1720 cm^{-1} (C=O stretching of ester) and 3400 cm^{-1} (O–H stretching). Additional peaks associated with cellulose (C–O–C and C–O stretching) verified the successful incorporation of MCC into the hydrogel network. Figure 2.

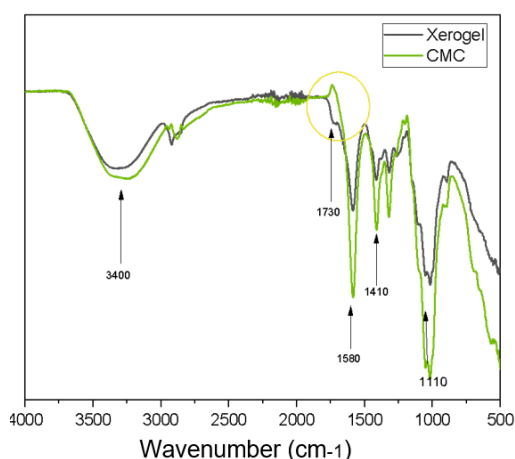


Figure 2: Comparative FTIR spectra of CMC and xerogel, highlighting changes in characteristic bands: O–H stretching ($\sim 3400\text{ cm}^{-1}$) and bands associated with carbonyl/carboxylate groups (~ 1730 , 1580 , 1410 cm^{-1}) and C–O–C/C–O vibrations ($\sim 1110\text{ cm}^{-1}$), indicating interactions/chemical modifications after xerogel formation.

UV–Vis and XRD Analysis

UV–Vis spectra of the hydrogels loaded with metallic salts and natural extracts showed distinct surface plasmon resonance bands around $420\text{--}440\text{ nm}$ for Ag nanoparticles and $520\text{--}540\text{ nm}$ for Au nanoparticles, confirming their formation within the hydrogel matrix. Figure 3 and Figure 4.

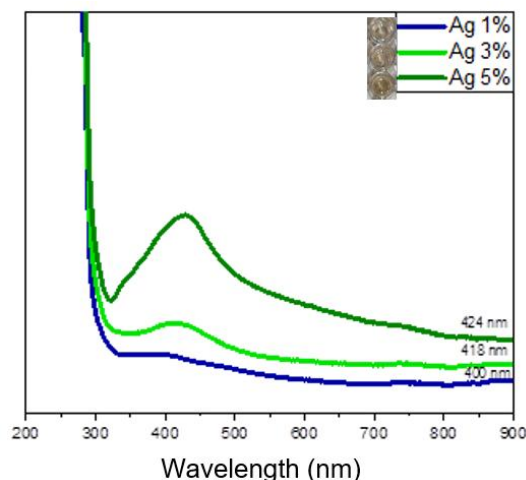


Figure 3: UV–Vis spectra of NPsAg synthesized at different loadings (1, 3, and 5%), showing the localized surface plasmon resonance (LSPR) band at $\sim 400\text{--}424\text{ nm}$.

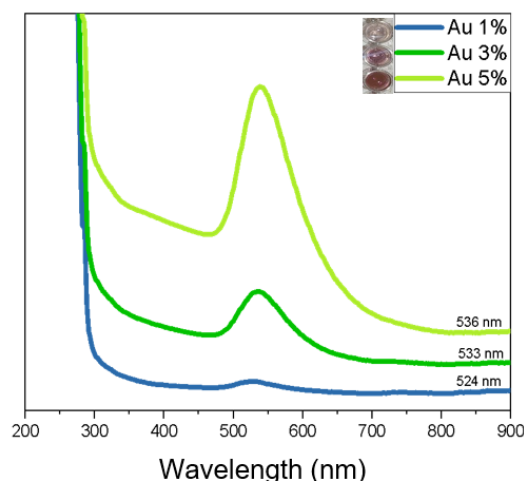


Figure 4: UV–Vis spectra of NPsAu (1, 3, and 5%), with the LSPR band at $\sim 524\text{--}536\text{ nm}$

X-Ray analysis

Upon analyzing the nanoparticles at the three evaluated metal loadings, only the samples containing 3% metallic content exhibited well-defined diffraction reflections. In the case of NPsAg at 3%, characteristic Bragg reflections were observed at $2\theta = 32.3^\circ$ and 37.0° , corresponding to the (111) and (101) crystallographic planes, respectively, thereby confirming the presence of metallic silver

(Ag⁰). Similarly, for NPsAu, diffraction peaks located at $2\theta = 38.1^\circ$ and 44.3° were assigned to the (111) and (200) planes, respectively, which are characteristic of metallic gold (Au⁰) nanoparticles. Figure 5.

These results demonstrate that the green synthesis process successfully generated well-dispersed metallic nanoparticles stabilized by the hydrogel matrix.

Antimicrobial Activity

Hydrogels containing metallic nanoparticles exhibited enhanced antimicrobial activity compared with commercial nanoparticles. The silver nanoparticles synthesized using the cellulose-based hydrogels exhibited clear antimicrobial activity against both *Staphylococcus aureus* and *Escherichia coli*, demonstrating their effective bactericidal potential. In contrast, the gold nanoparticles prepared under the same conditions did not show detectable antimicrobial activity against

either microorganism. These results highlight the distinct biological behavior of silver nanoparticles and confirm the suitability of the hydrogel matrix for generating bioactive Ag-based nanomaterials.

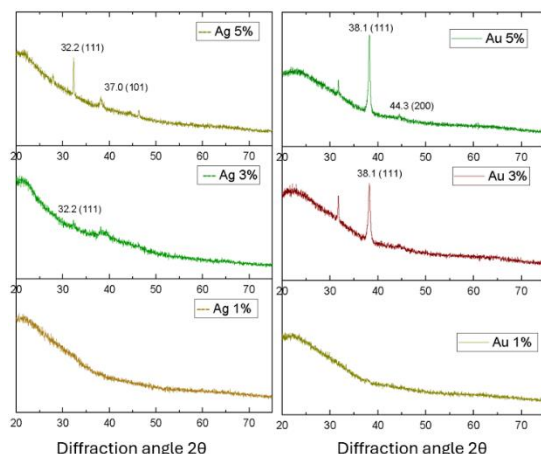


Figure 5: X-ray diffraction (XRD) patterns of silver (Ag) and gold (Au) nanoparticles synthesized at different metal loadings (1, 3, and 5%).

Conclusions

Nanocomposite hydrogels based on CMC and MCC crosslinked with citric acid were successfully developed and functionalized with natural extracts from Moringa, Paramenteira, and orange. These extracts enabled the green synthesis of silver and gold nanoparticles directly within the hydrogel matrix. The resulting materials demonstrated excellent water absorption, structural integrity, and antimicrobial activity.

The integration of renewable cellulose derivatives and bioactive extracts provides an eco-friendly pathway for producing multifunctional biomaterials suitable for biomedical and environmental applications.

Acknowledgments

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