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Rapid design and characterization of natural binary nanocomposite (nanocellulose/nano calcium fluoride) for teeth remineralization

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ABSTRACT

Background: Nanocellulose derived from agricultural waste offers sustainable potential for biomedical applications. *Aim*: To synthesize nanocellulose-based nanocomposite with nano calcium fluoride and evaluate its adhesion to dental surfaces. *Methodology*: Nanocellulose was synthesized from rice husk via chemical and ultrasonic treatment. The nanocellulose/nano calcium fluoride (NC/nCaF_2) nanocomposite was formed using ultrasound. Characterization was performed using Fourier Transform Infrared Spectroscopy, X-ray Diffraction, Scanning Electron Microscope and Transmission Electron Microscope. *Results*: Fourier Transform Infrared Spectroscopy confirmed cellulose integrity; X-ray Diffraction revealed 66.3% crystallinity. Scanning Electron Microscope and Transmission Electron Microscope showed structural features and successful nanoparticle integration. The nanocomposite adhered effectively to tooth surfaces, with thickness around 2 ± 2.5 µm. *Conclusion*: The NC/nCaF_2 nanocomposite demonstrated promising potential for tooth remineralization and biomedical applications.

KEYWORDS

rice husk, nanocellulose, nano calcium fluoride, nanocomposite, teeth adhesion

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1. INTRODUCTION

Dental caries is a complex disease that involves a continuous cycle of demineralization and remineralization, driven by bacterial activity. The application of remineralizing agents, such as fluoride-based and non-fluoridated compounds, has been central to combating this disease. Non-fluoridated systems include calcium phosphate products [1], CPP-ACP systems, self-assembling peptides like SAP11-4, and advanced techniques such as laser treatment and nanoparticles [2].

Nanoparticles are increasingly being explored for dental treatments due to their unique properties at the nanoscale, which influence their visual and physicochemical characteristics. Nanomedicine, a growing field, aims to develop effective health treatments. Additionally, natural biomaterials, valued for their biodegradability and support for cell growth, are gaining prominence in medical applications. The conversion of agricultural waste into cost-effective medical composites, such as cellulose and silicon-based fillers, is also emerging as a sustainable trend [3].

Rice husk, a common byproduct of rice milling, is primarily composed of silica and cellulose [4]. Cellulose, composed of β-D-glucopyranose units linked by β-1-4 bonds, has strong intra- and intermolecular hydrogen bonds that influence its physical properties. Sustainable nanocellulose, extracted from agricultural waste, offers excellent characteristics such as high strength, crystallinity, biodegradability, and cost-effectiveness [5].

Various techniques, including acid hydrolysis, electrospinning, and ultrasonication, are used to break down crystalline cellulose into nanoscale particles. Among these, acid hydrolysis is particularly popular because it produces single, well-defined crystals with high crystallinity [6]. This study aims to synthesize nanocellulose-based nanocomposites and evaluate their adhesion to dental surfaces, in line with the broader effort to utilize sustainable materials in advanced dental applications.

2. METHODOLOGY

2.1. Preparation of experimental materials

Preparation of nanocellulose: The synthesis of nanocellulose began with the washing of rice husk to remove contaminants, after which the husk was dried and ground into fine particles. The dewaxing process was performed using a Soxhlet extractor, followed by mixing the rice husk with a potassium hydroxide (KOH) solution to remove residual waxes. After filtration, the husk underwent a bleaching treatment using sodium chlorite, and further chemical treatments were applied. The cellulose was then subjected to acid hydrolysis using sulfuric acid to produce nanocellulose. The resulting suspension was dialyzed, centrifuged, and sonicated to obtain the nanocellulose suspension [7].

Preparation of nanocellulose/nano calcium fluoride nanocomposite: To prepare the NC/nCaF_2 nanocomposite, nano calcium fluoride (nCaF_2) was added to the nanocellulose solution. The mixture was subjected to ultrasonic waves for 10 min at 40°C. UV-Vis spectrophotometry was used to monitor the calcium fluoride content in the filtrate, with the optimal amount of nCaF_2 being approximately 0.315 g [7].

3. RESULTS

Identification of the prepared nanocellulose and nanocellulose/nano calcium fluoride nanocomposite: The Fourier Transform InfraRed (FTIR) analysis of nanocellulose show peaks corresponding to O-H stretching at 3398 cm^{-1} and C-H stretching at 2900 cm^{-1} (Figure 1A). The absence of the 1740 cm^{-1} peak, linked to acetyl and ester groups. Structural features, such as the C-O-C bond at 1161 cm^{-1} and C-H stretching at 1097 cm^{-1}, remained intact. Furthermore, the prominence of the glycosidic bond-associated signal at 893 cm^{-1}. Regardless, the nanocellulose/nano calcium fluoride (NC/nCaF_2) composite the FTIR spectrum showed physical interactions between the two materials, with no new chemical bonds formed. The O-H bond appeared at 3645 cm^{-1}, and the C-H stretching was observed at 2889 cm^{-1} and 2357 cm^{-1}, while the C-O bond was at 1103 cm^{-1} (Figure 1A).

The X-ray diffraction (XRD) analysis revealed a crystallinity index of 66.3%, with characteristic peaks at 16°, 22.5°, and 35.4° corresponding to the crystallographic planes (110), (200), and (004), respectively (Figure 1B).

Scanning Electron Microscopy (SEM) analysis of nanocellulose demonstrated nanoscale dimensions, with fibers ranging from 18 to 60 nm. Nanocrystalline cellulose, distributed along the fibers, ranged from 17 to 40 nm, consistent with similar rice husk-derived cellulose reports [8]. Regarding, NC/nCaF_2 composite the SEM analysis showed significant structural changes, including irregular sapphire-like features up to 33.61 nm (Figure 1C). This result also supported by finding Transmission electron microscopy (TEM) which shows rod-like crystalline regions (Figure 1D).

Characterization of Tooth-Decorated NC-NCaF_2: SEM imaging revealed that the nanocomposite effectively adhered to the tooth surface. The study also determined that the thickness of the NC/nCaF_2 nanocomposite was approximately 2 ± 2.5 μm (Figure 1E).

4. DISCUSSION

Characterization of nanocellulose and nanocellulose/nano calcium fluoride nanocomposite: The FTIR analysis of nanocellulose confirmed the effective chemical treatment to remove lignin and hemicellulose, absence of the 1740 cm^{-1} peak indicated successful removal of acetyl and ester groups.

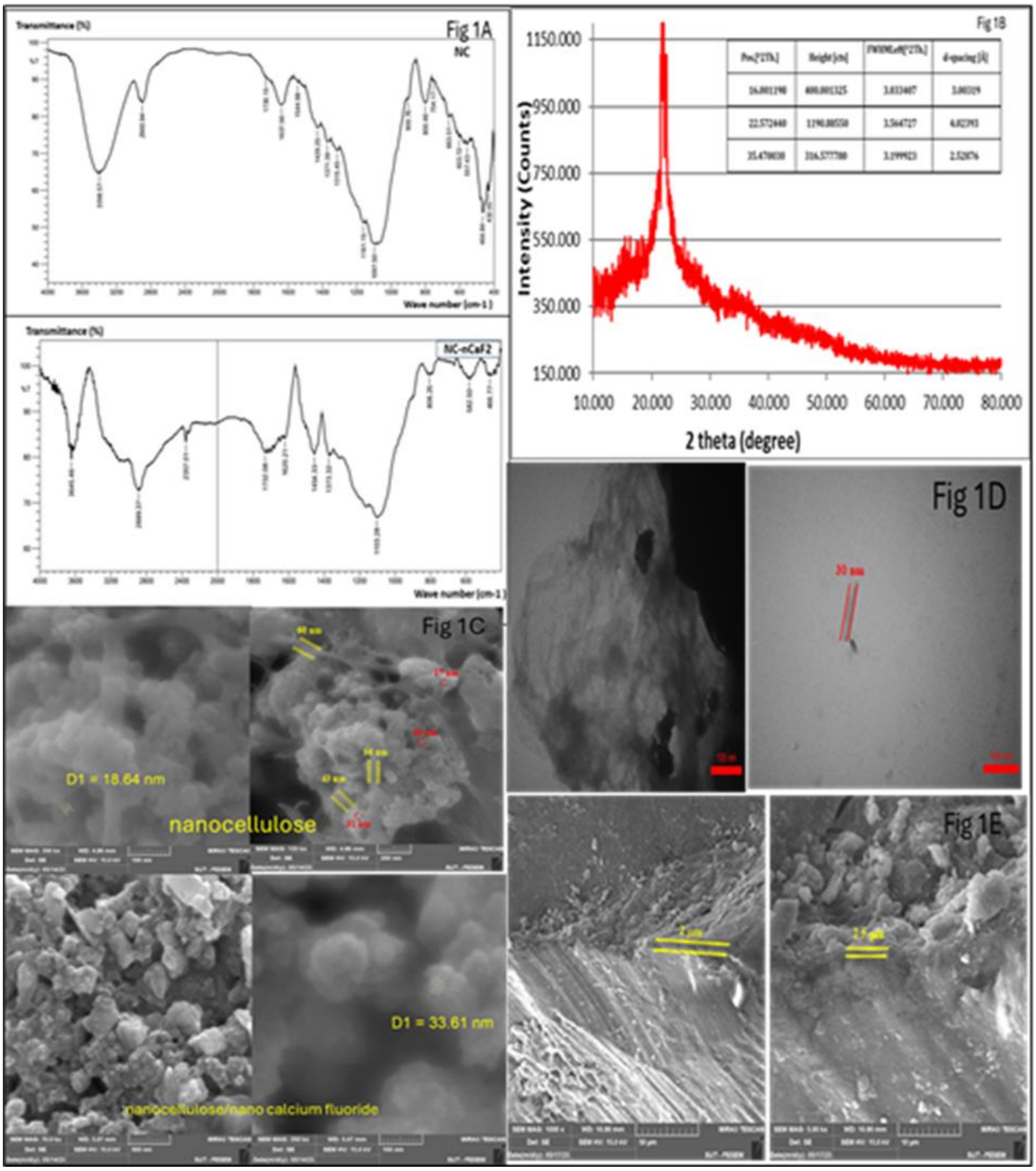

Figure 1. Fourier Transform InfraRed (FTIR) of nanocellulose and NC/nCaF$_2$ (**A**), XRD pattern of nanocellulose (**B**), SEM of nanocellulose and nanocellulose/nano calcium fluoride (**C**), TEM of nanocellulose (**D**), SEM of tooth decorated NC- nCaF$_2$ (**E**).

The presence of the C-O-C bond at 1161 cm^{-1} and C-H stretching at 1097 cm^{-1} confirming that acid hydrolysis did not alter the cellulose structure. the prominence of the glycosidic bond reflected the increased cellulose concentration after treatment, consistent with previous studies by Chen *et al*. [9] and Wang *et al*. [10]. The FTIR of the nanocellulose/nano calcium fluoride (NC/nCaF$_2$) show no new chemical bonds formed, confirming the incorporation of calcium fluoride nanoparticles into the nanocellulose matrix without altering its chemical structure (Figure 1A).

The XRD analysis revealed a crystallinity index of 66.3%, This high crystallinity, attributed to hydrogen bonding within the cellulose structure, was further enhanced by the removal of non-crystalline components, corroborating findings from previous study by Wang *et al*. [10] (Figure 1B).

SEM analysis of nanocellulose demonstrated nanoscale dimensions, confirming the effective preparation method. Regarding, NC/nCaF_2 composite the SEM analysis showed significant structural changes demonstrating successful nanoparticle coating on the nanocellulose surface. The size and morphology changes confirmed the formation of the NC/nCaF_2 composite, aligning with the desired structural modifications (Figure 1C). TEM analysis further validated the structural changes, showing rod-like crystalline regions indicative of the effective separation of crystalline and amorphous regions, supporting the high crystallinity of the nanocellulose (Figure 1D).

Characterization of tooth-decorated NC-NCaF_2: The characterization of the tooth-decorated NC/nCaF_2 nanocomposites was performed to evaluate their potential for enhancing remineralization on tooth surfaces.

The SEM shows that the nanocomposite effectively adhered to the tooth surface, indicating successful bonding between the material and the enamel. This strong adhesion is significant as it suggests the potential for the nanocomposite to aid in remineralization. Furthermore, the thickness of the NC/nCaF_2 nanocomposite was approximately 2 ± 2.5 μm, which provides important insight into the material's performance and physical properties in dental applications. These findings confirm that the NC/nCaF_2 nanocomposite could be a promising agent for enhancing tooth remineralization, as demonstrated by the SEM images (Figure 1E).

5. CONCLUSION

This study successfully synthesized nanocellulose (NC) from rice husk, confirming its nanofiber and nanocrystal structures with a crystallite index of 66.3%. A binary nanocomposite (NC/nCaF_2) was then formed using an ultrasound-assisted method by incorporating nano calcium fluoride into NC. The SEM results demonstrated effective adhesion of the NC/nCaF_2 nanocomposite to the tooth surface, highlighting its potential for enhancing remineralization. With a thickness of approximately 2 ± 2.5 μm, the nanocomposite presents promising applications in materials science, biomedicine, and dental treatments.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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