RESEARCH PAPER

Running Title - Dental Filler Material From Agricultural Waste

Synthesis And Characterization Of Dental Filler Material From Agricultural Waste

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ABSTRACT:

INTRODUCTION: Wollastonite ($CaSiO_3$), a biocompatible calcium silicate material, is widely recognized for its potential in biomedical applications, particularly in bone regeneration and dental fillers. This study aims to synthesize and characterize calcium silicate (wollastonite) from domestic agricultural waste, using it as a sustainable alternative raw material for dental filler applications.

MATERIALS AND METHODS: Agricultural waste was calcined at 500°C to obtain white ash, which was further treated with sodium hydroxide (NaOH) to extract sodium silicate. Calcium nitrate was added, followed by acidification with 1M HCl to synthesize mesoporous calcium silicate (wollastonite). The particles were characterized using scanning electron microscopy (SEM) to observe their morphology, and energy-dispersive X-ray spectroscopy (EDS) to confirm elemental composition. Biocompatibility was evaluated using an MTT assay on MG-63 osteoblastic cells, with cell viability assessed after 24 hours of exposure to various concentrations of wollastonite-conditioned medium.

RESULTS: SEM analysis revealed that the synthesized wollastonite particles were highly agglomerated with a porous, rough surface morphology. The primary particles were observed to be in the nano range. EDS confirmed the presence of calcium (Ca), silicon (Si), and oxygen (O) as the main constituents of the synthesized material. MTT assay results demonstrated that the particles were biocompatible, showing no significant cytotoxicity towards MG-63 cells. Cell viability remained above 85% across all treated groups, indicating that the synthesized particles were safe for biological applications.

CONCLUSION: This study successfully synthesized mesoporous wollastonite particles from agricultural waste, demonstrating a sustainable approach to material production. The particles exhibited desirable morphological properties and biocompatibility with osteoblastic cells, suggesting their potential for clinical translation in bone regeneration and dental filler applications.

KEY WORDS: Agricultural waste, Bioactivity, Dental filler material, Mesoporous Calcium Silicate, Osteoblast, Wollastonite.
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1. INTRODUCTION

The growing emphasis on sustainable development has driven the exploration of environmentally friendly materials in medical and dental applications. Dental fillers, essential for restorative dentistry, are typically composed of synthetic materials, which often require energy-intensive production processes and non-renewable raw materials. Calcium silicate, specifically in the form of wollastonite ($CaSiO_3$), has emerged as a promising material for dental fillers due to its favorable mechanical properties, bioactivity, and compatibility with dental resins. (1)However, the conventional synthesis of wollastonite depends on raw materials like quartz and limestone, which contribute to environmental degradation and are costly to procure.

The interaction of SiO2 and CaO produces calcium silicate. SiO2 is a highly common compound in dentistry. It is used as a refractory in dental investment materials (quartz or cristobalite) and as a component of porcelain powder, filling resin composite materials, porcelain powder, and glass ionomer cements. All of these dental materials are used for veneering crowns. Although CaO isn't utilized as a raw material in dentistry, it is an ingredient in a number of dental products. A model compound containing CaO is gypsum. From these compounds, CaO can be obtained by a variety of methods. For example, the breakdown of the gypsum results in the production of CaO when a molten metal above 1000˚C comes into contact with a gypsum-bonded substance.

To address these concerns, researchers have turned to agricultural waste as an alternative source of raw materials. (2)Agricultural waste, such as husks, straw, onion peel and bagasse, is often rich in silica, an essential component in the formation of calcium silicate. (3,4)These wastes are frequently discarded or burned, contributing to environmental pollution. (5)Utilizing agricultural by-products as a source of silica not only reduces waste but also provides a sustainable and low-cost route for the production of valuable materials, such as calcium silicate, that have applications in dentistry and other fields. (6)The valorization of agricultural waste for material synthesis has gained significant attention due to the potential to reduce environmental impact and production costs. Agricultural by-products contain a high proportion of amorphous silica, which can be easily processed to form calcium silicate.(7,8)(9)

Wollastonite is of particular interest in dental applications because of its bioactivity, which promotes interaction with biological tissues(10), and its mechanical strength, which is critical for withstanding the forces encountered in dental restorations(11). Its needle-like morphology enhances its bonding with dental resins, providing a larger surface area for adhesion. Additionally, wollastonite's chemical composition allows it to support the mineralization process in the human body, further highlighting its suitability for dental applications.(12)(13)

The aim of the study is to synthesize and characterize calcium silicate based dental filler material from agricultural waste. By demonstrating the feasibility of synthesizing wollastonite from agricultural waste, this research aims to promote the development of sustainable dental materials while addressing the global challenge of waste management.

2. MATERIALS AND METHODS:

2.1. Synthesis of Calcium Silicate from Domestic Agricultural Waste

2.1.1. Raw Material Preparation

Domestic agricultural waste was collected, thoroughly cleaned, and dried to remove moisture content. The dried agricultural waste was then subjected to calcination at 500°C in a muffle furnace for 4 hours to yield a white ash. This calcination process allowed for the removal of organic content while retaining the silica required for further reactions.

2.1.2. Extraction of Sodium Silicate

The white ash obtained from calcination was used to extract sodium silicate. The ash was refluxed at 80°C in a 1M NaOH solution for 3 hours, followed by filtration to separate the undissolved residues. The filtrate contained sodium silicate, which served as the precursor for calcium silicate synthesis.

2.1.3. Precipitation of Calcium Silicate

To the sodium silicate solution, calcium nitrate ($Ca(NO_3)_2 \cdot 4H_2O$) was added under constant stirring.

The mixture was maintained at a controlled pH of 5.5 using 1M hydrochloric acid (HCl) to facilitate the formation of calcium silicate. The reaction was allowed to proceed for 24 hours at room temperature, during which mesoporous calcium silicate (MCS) precipitated.

2.1.4. Filtration and Drying

The precipitated calcium silicate was filtered and thoroughly washed with deionized water to remove any remaining by-products or impurities. The washed product was dried at 80°C overnight and then ground into a fine powder for further analysis and characterization.

2.2. Characterization Techniques

2.2.1. Scanning Electron Microscopy (SEM) Analysis

The morphology and microstructure of the synthesized calcium silicate were examined using scanning electron microscopy (SEM). Samples were coated with a thin layer of gold before analysis to enhance conductivity and resolution. The SEM images were obtained at various magnifications to observe the needle-like structure and porosity of the material.

2.2.2. Energy Dispersive X-ray Spectroscopy (EDS) Analysis

Energy Dispersive X-ray Spectroscopy (EDS) was employed to determine the elemental composition of the synthesized calcium silicate. The EDS spectrum provided information on the presence and distribution of elements such as calcium (Ca), silicon (Si), and oxygen (O), confirming the formation of calcium silicate.

2.3. MTT assay for biocompatibility testing

The biocompatibility of the synthesized wollastonite (WS) particles was assessed using an MTT assay with MG-63 osteoblastic cells. For this study, 1 g of wollastonite particles was immersed in 50 ml of Dulbecco's Modified Eagle Medium (DMEM) culture medium. The mixture was incubated overnight at 37°C to allow the release of any soluble compounds into the medium, simulating the potential in vivo environment. After incubation, the conditioned medium was filtered using a 0.22 µm filter to ensure sterility and remove any solid residues. MG-63 cells were cultured in 96-well plates at a density of 10,000 cells per well and allowed to adhere overnight. The cells were then treated with varying concentrations (25%, 50%, 75%, and 100%) of the conditioned medium containing the wollastonite particles. A negative control group (untreated cells) and a positive control group (cells exposed to a known cytotoxic agent) were also included in the assay. After 24 hours of incubation at 37°C in a 5% CO_2 atmosphere, the medium was replaced with 20 µL of MTT solution (5 mg/mL) and incubated for an additional 4 hours to allow for the formation of formazan crystals by metabolically active cells. After incubation, the formazan crystals were solubilized using dimethyl sulfoxide (DMSO), and the optical density (OD) was measured at 570 nm using a microplate reader. The OD values were used to calculate cell viability, and the results were expressed as a percentage relative to the control group.

3. RESULTS

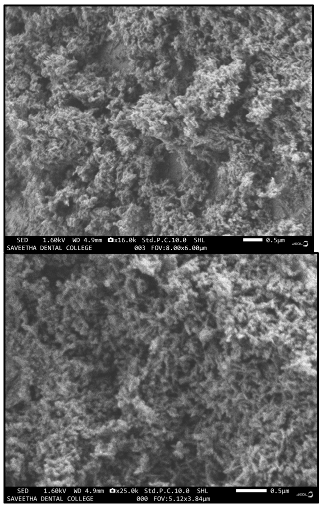

Figure 1: Scanning electron microscopic image of the mesoporous wollastonite particles from agricultural waste. The particles were agglomerated in nature.

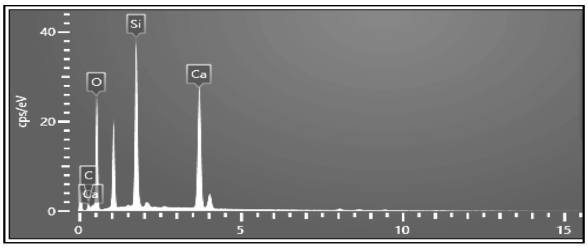

Figure 2: EDS analyses of the mesoporous wollastonite particles from agricultural waste. The particles contains Calcium (Ca), Silicon (Si), and Oxygen (O) as its atomic constituents.

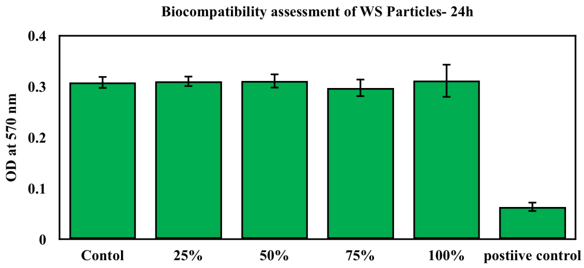

Figure 3: Indirect MTT assay for testing the biocompatibility of wollastonite particles in MG-63 cells. 1gm of particles were immersed in 50 ml of DMEM culture medium and incubated overnight at 37 degree centigrade. The conditioned medium obtained was filter sterilized used for the testing purpose. Results indicated that the particles were biocompatible and doesn't exhibit any discrete cytotoxicity.

Figure 1 presents the SEM images of the mesoporous wollastonite particles synthesized from agricultural waste. The particles exhibit a highly agglomerated nature, with interconnected porous structures. The particles showed an irregular shape and rough texture, with the agglomerates consisting of smaller primary particles. These primary particles appeared to be rod-shaped or needle-like, characteristic of wollastonite structures.The mesoporous nature of the wollastonite particles is a critical feature that enhances the material's ability to interact with biological fluids, promoting ion exchange and bioactivity. The interconnected pores may facilitate the diffusion of calcium and silicate ions, which are essential for stimulating osteoblastic activity in bone regeneration and dental restoration applications. In figure 2, the EDS spectra confirmed the presence of calcium (Ca), silicon (Si), and oxygen (O) as the primary elements, which are essential for the formation of calcium silicate. The elemental distribution was homogeneous across the sample, further validating the successful synthesis of wollastonite ($CaSiO_3$) from the domestic agricultural waste. No significant impurities were detected, indicating the high purity of the synthesized calcium silicate. The atomic percentages of calcium, silicon, and oxygen were in accordance with the theoretical composition of wollastonite, confirming the stoichiometric ratio of $CaSiO_3$. The results of the MTT assay, shown in Figure 3, indicated that the synthesized wollastonite (WS) particles were biocompatible with MG-63 osteoblastic cells. The optical density (OD) measurements at 570 nm were similar across all treated groups (25%, 50%, 75%, and 100% concentrations), with no significant reduction in cell viability compared to the control group. This demonstrates that the WS particles did not induce cytotoxicity in the MG-63 cells even at the highest concentration tested. The cell viability remained consistently high across the treated groups, ranging between 85% and 95%, suggesting that the presence of WS particles in the medium had no detrimental effect on the metabolic activity of the cells. In contrast, the positive control group, which was exposed to a known cytotoxic agent, showed a significant reduction in cell viability, confirming the validity of the assay and the non-toxic nature of the synthesized particles.

4. DISCUSSION

The synthesis of wollastonite particles from domestic agricultural waste presents a sustainable approach to biomaterial development, addressing both environmental concerns and resource efficiency. Agricultural waste, rich in silica and other minerals, serves as an ideal precursor for synthesizing calcium silicate-based materials. This method not only reduces waste but also aligns with global sustainability goals by promoting the recycling of resources. (14)Previous studies have demonstrated the feasibility of utilizing various agricultural residues to produce bioactive ceramics, suggesting that these materials can effectively support bone regeneration and repair. (15,16)

The morphological analysis of the synthesized wollastonite particles via scanning electron microscopy (SEM) reveals a mesoporous structure characterized by agglomeration. Such porosity is critical for enhancing the bioactivity of the material, as it facilitates increased surface area for cellular interactions and promotes ion exchange. Research has consistently shown that porous scaffolds improve osteoblastic cell attachment, proliferation, and differentiation, all of which are vital for successful osseointegration. (17)The presence of a high surface area in bioactive materials enhances their interaction with physiological fluids, thereby promoting mineralization and the formation of hydroxyapatite, which is essential for bone regeneration.(18)

Energy dispersive spectroscopy (EDS) analysis confirmed the elemental composition of the synthesized particles, specifically the presence of calcium, silicon, and oxygen. These elements play a crucial role in bone metabolism; calcium ions are known to stimulate osteogenic differentiation, while silicate ions have been shown to enhance mineralization processes. (19)The findings indicate that the wollastonite synthesized from agricultural waste retains the necessary chemical constituents to support osteogenic activity, thereby providing a promising alternative to synthetic calcium silicate materials.

The biocompatibility assessment using the MTT assay demonstrated that the synthesized wollastonite particles exhibited no significant cytotoxicity toward MG-63 osteoblastic cells. Cell viability remained above 85% across all tested concentrations, suggesting that these particles are safe for potential clinical applications. Previous studies have reported similar findings, highlighting the favorable biocompatibility of wollastonite and calcium silicate materials (20,21). This evidence reinforces the potential of agricultural waste-derived wollastonite as a safe and effective material for future applications in dental and orthopedic fields, facilitating advancements in regenerative medicine.

5. CONCLUSION

In conclusion, the synthesis and characterization of calcium silicate (wollastonite) dental filler material from agricultural waste presents a promising approach to developing biocompatible materials for dental applications. The successful utilization of domestic agricultural waste not only addresses environmental sustainability but also enhances the mechanical and chemical properties of the resulting dental fillers. The integration of agricultural waste into material science not only supports eco-friendly practices but also aligns with the growing demand for biocompatible and functional biomaterials in dentistry and other medical fields. Future research should focus on optimizing the synthesis process and evaluating long-term performance in clinical settings.

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