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NANOSILICA COLLAGEN PRODUCTION USING NANOSILICA SYNTHESIS FROM RICE STUBBLE AND ITS APPLICATION IN SKIN CREAM

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ABSTRACT
The production of sustainable and environmentally friendly materials has become a top priority in recent years. Nanosilica is a material with a wide range of applications in many fields such as additives to construction materials, environmental remediation, drug delivery, used in paper production, and applications in cosmetics, etc. Interestingly, nanosilica is abundant in agricultural wastes such as rice husks, bagasse, and corn cobs. In this study, nanosilica is synthesized from rice stubble ash, an agricultural byproduct common in some Southeast Asian countries. The synthesized nanosilica is then modified with collagen, a biopolymer widely used in cosmetic formulations. This research aligns with the goals of sustainable development, particularly in significantly reducing waste generation (SDG 12.5), promoting eco-friendly production, and reusing agricultural byproducts to create valuable materials. The nanosilica particles are spherical with an average diameter of 20.08 ± 0.61 nm, according to scanning electron microscopy (SEM) images. Following collagen modification, the particles retain their spherical shape with a diameter of 30.76 ± 0.67 nm. Advanced physicochemical techniques such as Fourier Transform Infrared (FTIR), Brunauer-Emmett-Teller (BET) theory, and X-ray Diffraction (XRD) are used to confirm the synthesis and modification of nanosilica, and the results are encouraging. The collagen-modified nanosilica (nanosilica collagen) was incorporated into a skin cream formulation for further evaluation.
Keywords: Nanosilica, Collagen**,** Nanosilica Collagen, Rice Stubble
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INTRODUCTION

The advent of nanotechnology has driven notable advancements across various technological sectors, including the cosmetics industry. Research into the application of nanotechnology in cosmetics has focused extensively on enhancing product performance and ensuring safety. Following the COVID-19 pandemic, personal care products have become more important, and consumers are calling for safer, natural, and eco-friendly goods.[1,2] Nanosilica is a white powder with a stable, porous structure and is chemically stable. It disperses uniformly, preventing clumping. As an adsorbent and desorbent, nanosilica gradually releases nutrients to the skin, making it beneficial in cosmetic formulations. Moreover, it enhances the texture, performance, and longevity of cosmetic products. Due to its refractive properties and chemical stability even under ultraviolet exposure, nanosilica is used in sunscreens and skin-whitening products. It is also incorporated into lipsticks to improve appearance and ensure uniform pigment distribution.[3,4] The safety of nanosilica materials used in cosmetics and drug delivery systems is a primary concern, with numerous studies conducted and ongoing in this area. These studies show that only crystalline nanosilica particles, typically released from construction sites, are linked to the risk of silicosis. In contrast, amorphous nanosilica particles, which are synthesized for drug delivery and cosmetic applications, are considered safe for human health.[5] In this study, the synthesized nanosilica was in an amorphous form. Collagen is a highly valuable biopolymer in skincare, frequently incorporated into products such as moisturizers, body lotions, and sunscreens. Its film-forming ability helps to lock in moisture, improve water retention, and soften the skin.[6] In this study, nanosilica, sourced from rice stubble, an abundant agricultural waste in Vietnam-is modified with collagen to form a nano collagen material. This combines the beneficial properties of both nanosilica and collagen for use in cosmetics. Nanosilica serves as an effective nutrient delivery system in skincare products. The research process is summarized in Fig.-1. This study aims to help reduce agricultural

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waste and environmental pollution, in line with Sustainable Development Goal 12.5 (SDG 12.5) as called for by the United Nations.

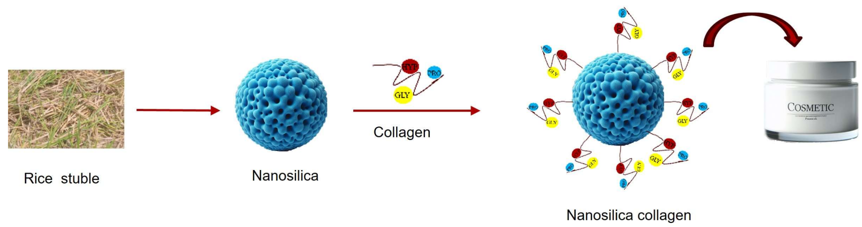

Fig.-1: The Synthesis Process of Collagen Nanosilica for Cosmetic Applications from Agricultural Waste Rice Stubble

EXPERIMENTAL

Material

Hydrochloric acid (HCl, 37%, China), sodium hydroxide (NaOH, ≥96%, Xilong), and distilled water were used in this study. Additionally, rice stubble from the Red River Delta served as the silica source. All chemicals used in the cosmetics, including almond oil, avocado oil, rice bran oil, shea butter, silky emulsifying wax, cetyl alcohol, dimethicone, and EHGP (ethylhexylglycerin and phenoxyethanol), were sourced from GreenLab, a leading supplier of high-quality cosmetic ingredients in Vietnam.

Preparation of Amorphous Nanosilica from Rice Stubble Ash

The rice husks are first cleaned with water, left to dry in the open air, and then heat-treated in a furnace at 650°C for 10 hours. After heat treatment, the resulting product is a light gray ash. Three grams of the ash are dissolved in a 3N NaOH solution and stirred in a magnetic stirrer bath at 100°C for 2 hours. Following dissolution, the mixture is filtered through filter paper to obtain a yellowish-transparent sodium silicate solution.[7,8] The sodium silicate solution is then neutralized with a 2.5N HCl solution and stirred in a magnetic stirrer bath. During neutralization, the pH is adjusted to a range of 6.5–7 until precipitation occurs, at which point stirring is stopped.[7,9] Following neutralization, the material is separated by centrifugation and then cleaned with deionized water and ethanol until the pH is neutral. The final solid is freeze-dried for 12 hours and then further dried for 20 hours to obtain the nanosilica product.[9] The synthesis process of amorphous nanosilica is shown in Fig.-2.

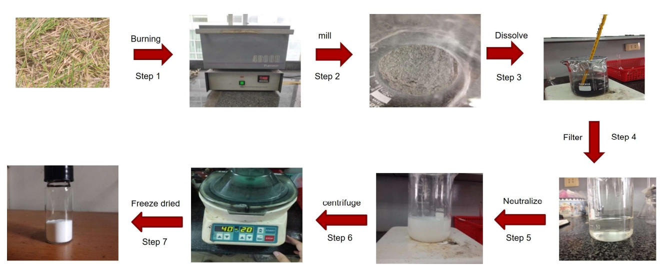

Fig.-2: Detailed Description of the Synthesis Process of Amorphous Nanosilica from Rice Stubble

Modification of Nanosilica with Collagen

The modification of nanosilica with collagen follows a similar procedure to the synthesis of nanosilica from rice stubble. However, after step 5, 0.4 g of collagen is added, and the mixture is stirred for 48 hours. The resulting collagen-modified mixture is then washed with deionized water and subjected to centrifugation. The product is then freeze-dried for 12 hours, followed by an additional 20 hours of drying.

Incorpatation of Nanosilica Collagen in Cosmetics

The nanosilica collagen product was incorporated into the formulation of a skincare cream through the following steps. In the preparation of the oil phase (mixture A), 2 g of almond oil, 2 g of avocado oil, 2 g of rice bran oil, 5 g of shea butter, 3 g of silky emulsifying wax, and 2 g of cetyl alcohol were combined in a 500 mL beaker. The mixture was heated to 50–60°C while stirring with a magnetic stirrer until the wax was fully dissolved. For the preparation of the water phase (mixture B), 77.5 g of double-distilled water, 5 g of dimethicone, and 1 g of nanocollagen were mixed in another 500 mL beaker. The solution was stirred until completely dissolved, then heated to 50–60°C to match the temperature of mixture A. Mixture B was gradually added to mixture A while continuously stirred. Heating was then discontinued, and the mixture was allowed to cool to 45°C. At this stage, EHGP, a combination of ethylhexylglycerin and phenoxyethanol, was added. EHGP provides enhanced preservation efficacy compared to conventional agents and requires only a minimal amount to ensure consumer safety.[10] The mixture was stirred further until it formed a consistent, milky-white cream texture. Finally, the pH of the mixture was adjusted to a range of 5.5–6.5 to complete the formulation. The final product was a nanocollagen W/O emulsion-based skin care cream, as shown in Fig.-3.

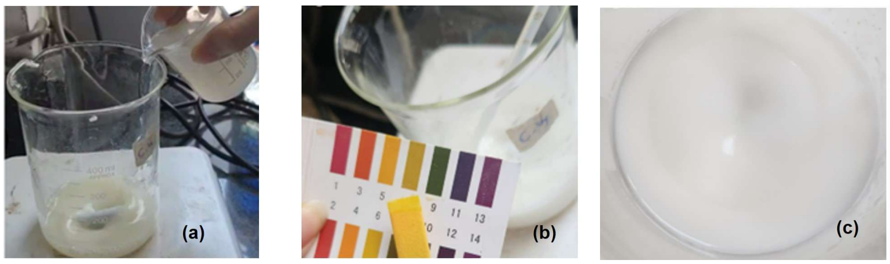

Fig.-3: The Process of Preparing Nanocollagen Skin Care Cream: (a) Emulsification System During the Addition of Solution B into A, (b) pH Testing of the Product, (c) the Final Nanosilica-Collagen Cream Product

Detection Method

FTIR 6300 (Jasco) was used to record the FTIR spectra of samples. Scanning electron microscopy (HITACHI Regulus 8100) was used to observe the morphology of the sample. Sizes and structures of nanosilica and nanosilica collagen were assessed by XRD (PAN Empyrean). Surface area & pore size were analyzed by NOVA 4200e (Quantachrome instrument).

RESULTS AND DISCUSSION

Structural Confirmation

The infrared spectroscopy results shown in Figure 4 reveal the following: In Fig.-4(a), collagen displays peaks at 3400 and 3300 cm^{-1}, indicating hydrogen bonding between the N-H group and the carbonyl group of the peptide chain. The absorption band between 1700–1600 cm^{-1} corresponds to the N-H bond of primary amines in the collagen structure. The band shifts slightly to a lower wavenumber, around 1580–1490 cm^{-1} for secondary amines.[6, 11,12] In Fig.-4(b), the absorption band between 3600–3200 cm^{-1} is attributed to the stretching vibration of the OH group. A strong and broad peak at 1082 cm^{-1} suggests the presence of asymmetric Si-O-Si bonds. The peaks at 958 and 802 cm^{-1} correspond to the symmetrical vibrations of Si-O-Si bonds, while the peak at 467 cm^{-1} is related to the bending vibration of Si-O-Si.[13,14,15] When nanosilica is modified with collagen, the FTIR spectrum in Fig.-4(c) reveals a prominent peak at 1645 cm^{-1}, characteristic of amide I, and another at 1546 cm^{-1}, which corresponds to amide II. These peaks confirm the successful attachment of collagen to nanosilica.[6, 11,12]

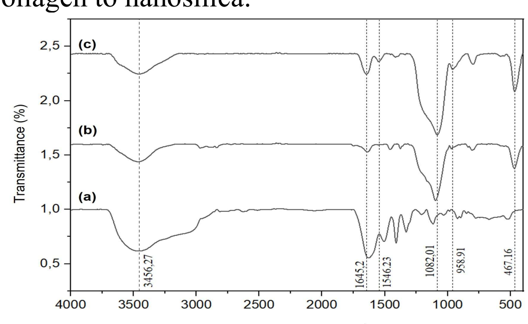

Fig.-4: FTIR Spectra of (a) Collagen, (b) Nanosilica, (c) Nanosilica Collagen

Morphology and Particle Size

SEM imaging analysis revealed the size and morphology of nanosilica particles and nanosilica modified with collagen as follows. The nanosilica particles were spherical, with an average size of 20.08 ± 0.61 nm. Similarly, the nanosilica-collagen particles retained a spherical shape but exhibited a slightly larger average size of 30.76 ± 0.67 nm. Literature suggests that nanoparticle size has a significant impact on their distribution kinetics within biological systems. Particles approximately 20 nm in size are excreted through the kidneys more rapidly than those measuring 100 nm.[16,17] Consequently, the nanosilica-collagen particles comply with safety size requirements, allowing efficient nutrient delivery to skin cells while ensuring rapid excretion, thereby minimizing any potential health risks to users.

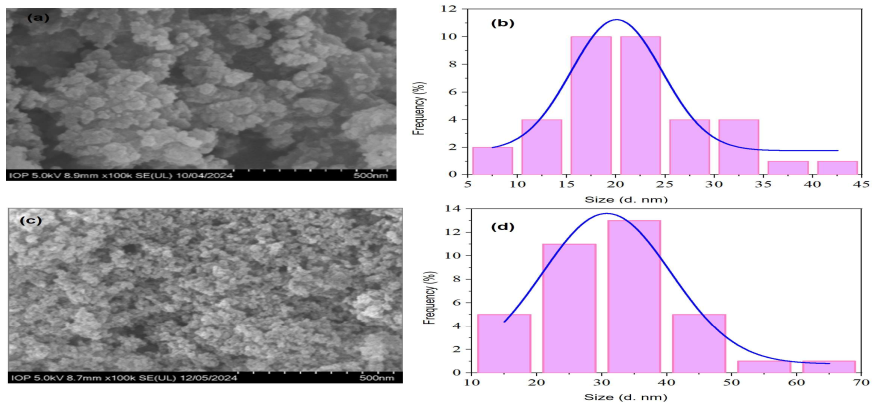

Fig.-5: SEM Images and Particle Size of (a, b) Nanosilica and (c, d) Nanosilica Collagen

X-ray Diffraction

Figure-6(a) shows a diffraction peak at 2θ = 22.78°, indicating the presence of an amorphous nanosilica structure. Following modification by collagen and XRD analysis (Fig.-6b), diffraction peaks at 22.4° and 24.3° are observed, confirming that the modification process did not change the amorphous nature of the nanosilica.[9,17,18] With its amorphous structure, nanosilica-collagen is safe for use in cosmetics.[19,20]

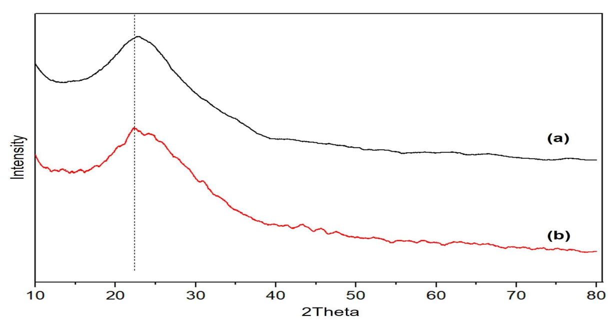

Fig.-6: XRD Patterns of (a) Nanosilica, (b) Nanosilica Collagen

The Adsorption-Desorption Isotherm Curve (BET)

The result indicates that the material has a porous structure (Fig.-7). The analysis of the surface area and pore structure reveals the following results: an adsorption surface area of 141.357 m²/g, a pore volume of 0.422 cc/g with a diameter of 9.88 Å, and a desorption surface area of 110.912 m²/g, with a pore volume of 0.415 cc/g and a diameter of 11.532 Å.[21,22]

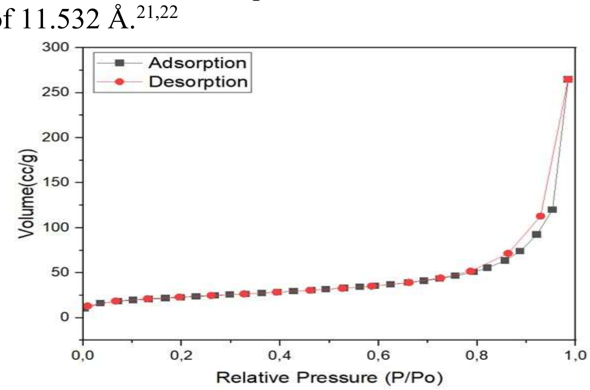

Fig.-7: N_2 Adsorption-Desorption Isotherms Graph of Nanosilica

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The Moisture Evaluation of Skin before and after using Nanosilica Collagen Skin Cream
The evaluation revealed that the skin care cream with nanosilica-collagen exhibited comparable moisturizing effects to those of a globally recognized nano-collagen skin care product (Fig.-8).

Fig.-8: Skin Moisture Measurement Results: a) Before using the Product, b) 10 Minutes after using the Cream Sample Containing Collagen, c) 10 minutes after using the Cream Sample Containing Nanosilica-Collagen, d) 10 minutes after using the Premium Branded Nano-Collagen Cream Sample

The Skin Test Results After Using Nanosilica Collagen Skin Cream
Digital microscope images revealed that the skin was slightly red before using the skin care cream, with visible creases and a few dry patches. After 7 days of using the nanosilica-collagen cream, the redness had diminished, and the skin appeared softer and healthier, although there was no significant change in the creases (Fig.-9). The microbiological test results for total yeast and mold, following ISO 16212:2017 standards, revealed a count of less than 10 CFU/g, indicating no bacterial contamination on the plate.

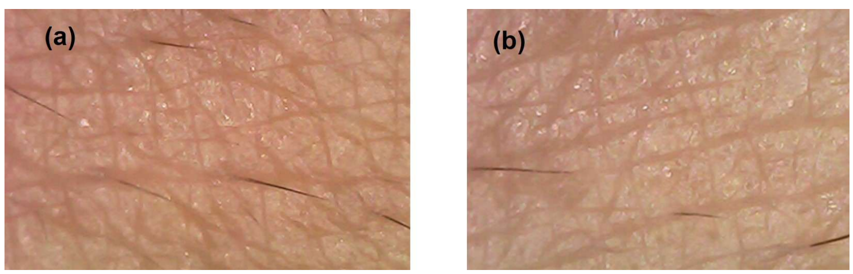

Fig.-9: The Skin Test Results After Using Nanosilica Collagen Skin Cream

CONCLUSION

Nanosilica is a versatile material with wide-ranging applications. This study successfully synthesized nanosilica from rice stubble, a byproduct of rice plants after harvesting. XRD, SEM, and BET analyses revealed that the material has an amorphous structure with a diameter of approximately 20.08 ± 0.61 nm. After modification with collagen, the nanosilica particles measure approximately 30.76 ± 0.67 nm in size and maintain their amorphous structure, ensuring their safety for cosmetic use. This research is expected to contribute to the expanding field of biogenic material synthesis, offering potential solutions for reducing environmental pollution, promoting the development of agriculture and industry, and aligning with the United Nations Sustainable Development Goal 12.5 (SDG 12.5).

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHORS CONTRIBUTION

The present work is related to *SDG-12:Responsible Consumption and Production.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:
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