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Novesar Jamarun
Department of Chemistry, University of Andalas, Limau Manis, Padang, West Sumatra, Indonesia, novesarjamarun@sci.unand.ac.id

Nabiila Ayyu Trycahyani
Department of Chemistry, University of Andalas, Limau Manis, Padang, West Sumatra, Indonesia, 2320412010_nabiila@student.unand.ac.id

Upita Septiani
Department of Chemistry, University of Andalas, Limau Manis, Padang, West Sumatra, Indonesia, upitaseptiani@sci.unand.ac.id

Vivi Sisca
Research Center for Chemistry, National Research and Innovation Agency, B.J. Habibie Science and Techno Park, Serpong, South Tangerang, Banten, 15314, Indonesia, sisca_vivi@yahoo.com

Muhammad Refki Novesar
Department of Tourism, Institut Seni Indonesia, Padang Panjang, 27128, West Sumatra, Indonesia, refki.novesar@gmail.com

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Novesar Jamarun, Nabiila Ayyu Trycahyani, Upita Septiani, Vivi Sisca, Muhammad Refki Novesar, and Sri Mulya

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RESEARCH ARTICLE

Utilization of Pensi Shell Waste as Hydroxyapatite Composite with Biopolymer from Shrimp Shell and PEG and Its Application

Novesar Jamarun [1,*], Nabiila Ayyu Trycahyani [1], Upita Septiani [1], Vivi Sisca [2], Muhammad Refki Novesar [3], Sri Mulya [1]

[1] Department of Chemistry, University of Andalas, Limau Manis, Padang, West Sumatra, Indonesia
[2] Research Center for Chemistry, National Research and Innovation Agency, B.J. Habibie Science and Techno Park, Serpong, South Tangerang, Banten, 15314, Indonesia
[3] Department of Tourism, Institut Seni Indonesia, Padang Panjang, 27128, West Sumatra, Indonesia

ABSTRACT

This study focuses on the synthesis, structural analysis and antibacterial properties of a composite comprising hydroxyapatite (HAp), polyethylene glycol (PEG) and chitosan (CTS). CTS was extracted from shrimp shells, and CaO for Hap was obtained from pensi shell. These composites were synthesized using the sol-gel method in situ by varying the concentrations of PEG (5, 10, 15, 20 and 25%) and CTS (1, 2, 3, 4, and 5%). The synthesized composites were characterized using FTIR, XRD, SEM-EDX, and TGA-DTA analyses, and their antimicrobial activities were also evaluated. The HAp-PEG-Chitosan composite with 5% chitosan and 10% PEG was identified as the best product as a white powder. FTIR analysis revealed the presence of specific functional groups: $PO4^{3-}$ and OH from HAp, N-H from chitosan, and C-H and C-C from PEG, confirming the successful formation of the composite. XRD analysis showed a hexagonal structure consistent with the hydroxyapatite diffraction standard (ICSD #157481) and a reduction in crystallinity due to polymer addition. SEM analysis demonstrated that needle-like HAp derived from pensi shells underwent surface changes, resulting in a more uniform texture when combined with PEG. EDX analysis confirmed the presence of Ca, P, C, and O elements, validating the successful synthesis of the HAp-polymer composite. TG-DTA analysis showed weight loss below 200°C, indicating the thermal stability of PEG at this temperature. The antimicrobial activity of the HAp-PEG-Chitosan composite was tested against *E. coli* and *S. aureus,* with CIP antibiotics used as a positive control for comparison.

Keywords: Antimicrobial, Chitosan, Hydroxyapatite, In situ method, Polyethylene glycol

Introduction

The bone defects due to trauma, pathological fractures, or degenerative disease, for example, osteoporosis and rheumatoid arthritis, constitute a major and persistent challenge in the practice of regenerative medicine. Despite the intrinsic regenerative potential of bone tissue, reconstruction of critical-sized defects, especially those resulting from deep infections or large tissue loss, frequently outstrips the natural healing ability and requires recourse to bone tissue engineering (BTE) strategies for restoration of functional and structural integrity.[1,2] Successful bone regeneration depends on the orchestration of a number of biological events, including the activation and proliferation of cells from the bone marrow, the presence of osteoinductive bioactive factors, and the presence of a three-dimensional porous scaffold that

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* Corresponding author.
E-mail addresses: novesarjamarun@sci.unand.ac.id (N. Jamarun), 2320412010_nabiila@student.unand.ac.id (N. A. Trycahyani), upitaseptiani@sci.unand.ac.id (U. Septiani), sisca_vivi@yahoo.com (V. Sisca), refki.novesar@gmail.com (M. R. Novesar), srimulya@adm.unand.ac.id (S. Mulya).

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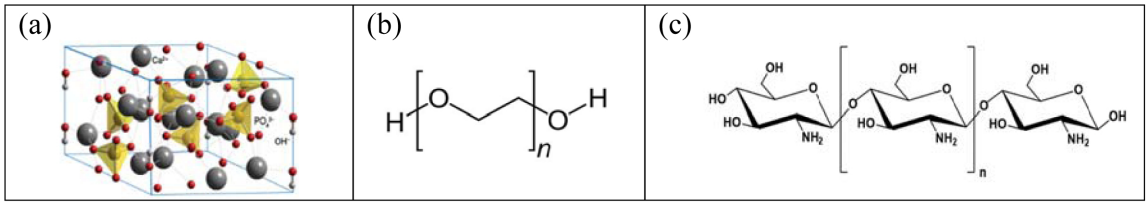

Fig. 1. Molecular structure of (a) HAp (b) PEG (c) CTS.

closely resembles the structure of the native extracellular matrix (ECM), thereby offering mechanical support and tissue guidance.

Hydroxyapatite (HAp), a naturally occurring apatite with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, boasts a Ca:P molar ratio of 1.67 and a molecular weight of 1004.6 g/mol. Its hexagonal crystal structure Fig. 1(a)[3] characterized by $\alpha = \beta = 90^{\circ}$ and $\gamma = 120^{\circ}$ in the P63/m space group, makes it an ideal component of bone tissue, comprising approximately 70% of bone composition. HAp, one of the major inorganic constituents of bone mineral, has garnered great interest for scaffold development based on its exemplary biocompatibility, bioactivity, and osteoconductivity.[4,5] The chemical similarity of HAp to the bone's mineral phase promotes good biologic integration. Yet, while possessing these admirable biological characteristics, the intrinsic brittleness and insufficient mechanical strength of pure HAp restrict its application, especially in load-bearing systems that demand high mechanical performance.[6] Efforts to overcome these drawbacks have included utilizing naturally occurring hydroxyapatite from eggshells and bovine bones. Besides reducing the cost of production, this process enables the development of scaffolds with improved porosity, an interconnected microstructure, and better biofunctionality.[7]

Recent advances have increasingly focused on the development of HAp-based composite materials incorporating biopolymers to enhance mechanical and biological performance synergistically. Among them, chitosan, a natural polysaccharide derived from the exoskeletons of shellfish, emerges as a promising candidate due to its superior biocompatibility, biodegradability, antimicrobial properties, hemostatic properties, and wound-healing-promoting ability.[8,9] The addition of HAp to CTS Fig. 1(c)[10] has been found to enhance scaffold flexibility, cell adhesion, and bioactivity. Further, the incorporation of PEG, a synthetic hydrophilic polymer, was reported to significantly improve the mechanical stability, thermal stability, and osteointegration activity of hydroxyapatite (HAp) composites via the improvement of scaffold hydrophilicity and cell–material interactions.[11]

Yet, though their predecessors investigated HAp-chitosan composites with the addition of antimicrobial agents like ciprofloxacin, the mechanical degradation that results from pharmaceutical incorporation and specific biopolymer inclusion continues to be a major drawback.[9] The attainment of a compromise between mechanical strength and antibacterial activity remains one of the biggest hurdles to the clinical translation of such materials. Furthermore, the majority of reported approaches have concentrated on synthetic starting materials, with no regard for the potential advantages of exploiting sustainable, renewable resources for the construction of scaffolds, which could enhance material innovation as well as environmental sustainability.

Under the given context, the present study aims to prepare novel hydroxyapatite-polyethylene glycol-chitosan composites via an in situ process using sustainable biomaterials, namely calcium oxide (CaO) from clam shells and chitosan from shrimp shells. This hybrid reinforcement strategy can enhance the antibacterial properties of this material due to the presence of chitosan, which has antibacterial capabilities. The addition of PEG Fig. 2(b)[10] can also increase the solubility of the material, potentially affecting the degradation rate and release of beneficial ions. This compositional change can influence the HAp energy band gap, which typically ranges from 4.50 to 5.40 eV, as well as the Ca:P molar ratio, which is ideally 1.67 for stoichiometric HAp.[12] Thus, the HAp-PEG-CTS composite can become a material that is not only antibacterial but also has optical and chemical properties that can be tailored for biomedical applications.[13,14] In addition, the use of marine-derived biowastes as starting materials not only maintains the spirit of green chemistry but also answers the current need for environmentally friendly biomaterials in biomedical uses. Thus, this work introduces a new and sustainable approach to the development of next-generation scaffolds for bone tissue engineering, aiming at the integration of biological functionality, mechanical properties, and sustainability.

Materials and methods

Materials

The primary materials employed in this study were CaO as a precursor in the synthesis of HAp, which was obtained from pensi shell waste collected from lake areas in West Sumatra, Indonesia, along with $(NH_4)_2HPO_4$ (Merck), PEG 6000 (Merck), and shrimp shells. Shrimp shells were used as a raw material for chitosan extraction, derived from chitin, and were sourced from shrimp shell waste gathered at local fish markets. The synthesis of the composite also involved HNO_3 65% (Merck) as a solvent for CaO, CH_3COOH 98% (Merck) as a solvent for chitosan, and NH_4OH (Merck) to adjust the pH of the composite mixture. For antibacterial activity testing, the materials used included Mueller-Hinton Agar (MHA), Nutrient Agar (NA), NaCl, ethanol, spiritus, and bacterial isolates of *E. coli* and *S. aureus*.

Synthesis of hydroxyapatite with sol gel method

The synthesis of HAp was carried out using the sol-gel method. Initially, 4.2 grams of CaO powder were dissolved in HNO_3 2 M. Once fully dissolved, a solution of $(NH_4)_2HPO_4$ was gradually added to the CaO solution under continuous stirring, while maintaining the pH at 10 to ensure optimal HAp formation. As a white sol began to form, the temperature was increased to 70˚C, and the mixture was stirred continuously for approximately 5 hours until the sol gradually thickened. The resulting mixture was then aged for 24 hours, followed by a drying process in an oven at 110˚C. [9]

Synthesis of HAp-PEG-Chitosan with in situ method

The synthesis of the HAp-PEG-CTS composite was carried out by sequentially adding the three main components to ensure accurate composition and homogeneous distribution. Initially, CaO powder, serving as the precursor for HAp, was weighed and dissolved in 2 M nitric acid. The dissolved CaO solution was then slowly combined with $(NH_4)_2HPO_4$ while continuously adjusting the pH to 10 using ammonium hydroxide. Once the white HAp sol formed at pH 10, PEG was added, followed by further pH adjustment and constant stirring at a stable temperature. Subsequently, chitosan—previously dissolved in 2% acetic acid was added to the mixture, and the pH was once again adjusted to 10. The resulting mixture was stirred for 3 hours at 70˚C until it gradually thickened. Afterwards, the mixture was cooled, filtered, and dried in an oven at 70˚C for 24 hours. The composite was synthesized using various concentrations of PEG (5, 10, 15, 20, and 25%) and chitosan (1, 2, 3, 4, and 5%) to determine the optimal composite formulation. [15,16]

Antibacterial activity analysis

For the antibacterial test, the paper disc diffusion method was used. Mueller Hinton Agar medium (9.5grams) was dissolved in 250 mL of distilled water. Then, the mixture was stirred and heated using a hotplate. The media and tools were sterilized. 10 mL of the media solution was compacted into a petri dish. Suspensions of *E. coli* and *S. aureus* bacteria were prepared by dissolving them in 0.9% physiological NaCl with the same turbidity level as the McFarland standard of 0.5. Next, each bacterium was rubbed onto the surface of the MHA using a cotton bud. Paper discs containing 0.0010 grams of selected HAp-PEG-CTS were placed on the media. Ciprofloxacin and HAp served as the positive and negative controls, respectively. Then, the media was incubated at 37˚C for 24 h. The inhibition zone formed was measured. [15]

Characterization

The synthesized HAp-PEG-CTS composite was characterized using several analytical techniques. The identification of chemical bonds and functional groups was carried out using Fourier Transform Infrared Spectroscopy (FTIR). The crystallinity of hydroxyapatite (HAp) was determined by X-ray Diffraction (XRD) analysis. Morphological and elemental analyses were conducted using Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDX). In addition, the thermal properties of the polymer components were examined through Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA).

Results and discussion

XRD analysis

XRD analysis was conducted on the composite materials HAp-PEG 10% and HAp-PEG-CTS 5%. The figure shows that the peak patterns of the HAp-PEG 10% and HAp-PEG-CTS 5% composites are similar to those of standard HAp (ICSD #154781), confirming the successful formation of HAp in both composite samples. The X-ray diffraction peaks of the synthesized hydroxyapatite are observed at 2θ values of 25.9˚, 31.8˚, 32.9˚, and 39.9˚, corresponding to the (002), (211), (300), and (310) crystal planes of a hexagonal structure. The addition of PEG and

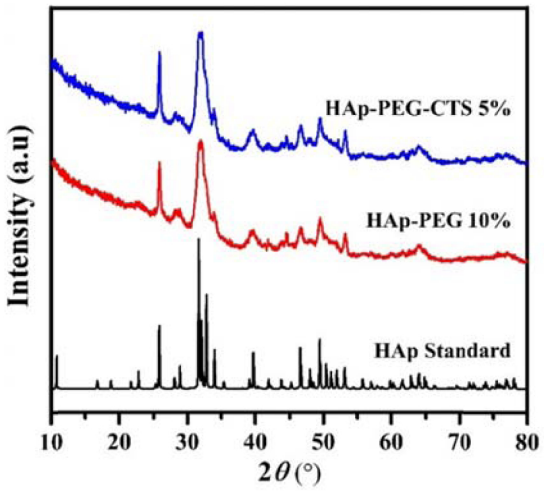

Fig. 2. XRD pattern of composite HAp-PEG 10% and HAp-PEG-CTS 5%.

chitosan polymers did not significantly affect the structure of hydroxyapatite, but a slight peak broadening was observed in both composite samples. The presence of PEG in the composite was confirmed by the emergence of new peaks at 2θ value of 23° and 26° in the HAp-PEG 10% and HAp-PEG-CTS 5% composites, while the peak at 2θ of 19° was related to the presence of chitosan in the HAp-PEG-CTS 5% composite, although the intensity was very low due to the low concentration of the polymer used. Further analysis of the crystal size showed that the crystal size of the HAp-PEG 10% composite was 14.64 nm, and that of the HAp-PEG-CTS 5% composite was 13.13 nm, indicating that the addition of polymers affected the crystal size. This suggests that the addition of organic compounds, namely PEG and chitosan, can inhibit the growth of hydroxyapatite in the composite, resulting in a decrease in crystal size and peak broadening. [17,18]

Fig. 2 illustrates the XRD pattern obtained in this analysis.

FTIR analysis

FTIR (Fourier-Transform Infrared) analysis was employed to identify the functional groups within the sample using an FTIR spectrometer. Fig. 3 shows the FTIR spectrum of successfully synthesized composites, namely HAp-PEG composites with PEG concentrations of 5%, 10%, 15%, 20%, and 25%, and HAp-PEG-CTS composites with chitosan concentrations of 1%, 2%, 3%, 4%, and 5%. Based on the results, all composites exhibit specific functional groups from each composite component, including PO_4^{3-} at wavenumbers of 1017, 1016, 1014, and 1013 cm^{-1} and OH at wavenumbers of 3206 and 3205 cm^{-1}, indicating the presence of HAp. C-H bending is observed at wavenumbers of 1340, 1337, 1332, 1331, 1330, 1329, and 1328 cm^{-1}, representing the polymer PEG, and the N-H functional group specific to chitosan is identified at wavenumbers of 1637 and 1636 cm^{-1}. [13]

The appearance of C = O, CH, and C = C absorption bands in the FTIR spectrum indicates the presence of organic compounds within the catalyst, consistent with the morphological analysis. These FTIR spectrum results align with previous studies. [12,19] Further supporting the chemical composition of the catalyst. Notably, the FTIR spectrum findings also complement the XRD peak results, emphasizing the consistency between various characterization techniques used in this study. This demonstrates the successful application of FTIR in elucidating the chemical changes in the catalyst, both before and after its utilization in the esterification reaction.

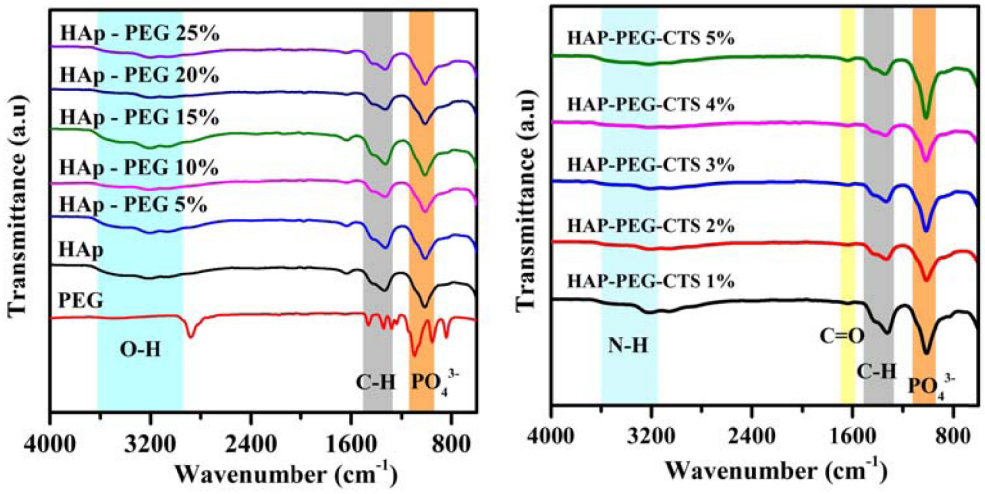

Fig. 3. FTIR spectra of composite HAp-PEG and HAp-PEG-CTS.

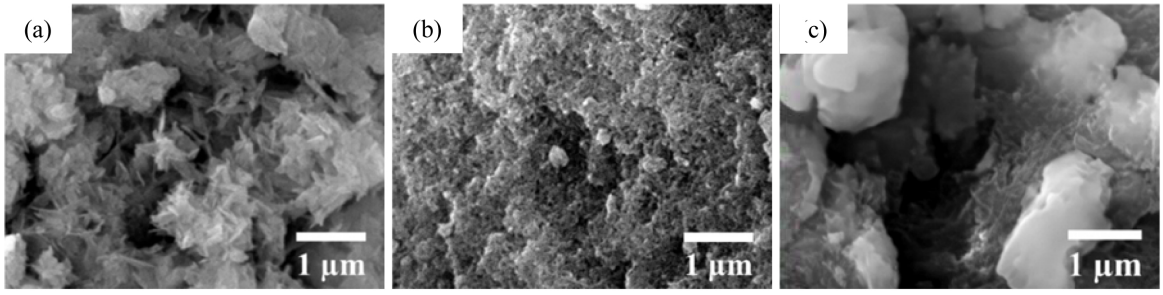

Fig. 4. SEM images of (a) HAp from pensi shell, composite (b) HAp-PEG 10% and (b) HAp-PEG-CTS 5%.

Table 1. Elements composition of HAp Pensi, HAp-PEG 10% and HAp-PEG-CTS 5%.

Sample	Ca [at%]	P [at%]	C [at%]	O [at%]	N [at%]	Ratio Ca/P
HAp Pensi	15.15	11.85	11.31	61.68	–	1.28
HAp-PEG 10%	21.98	15,47	9.90	52.66	–	1.42
HAp-PEG-CTS 5%	18.62	10.08	10.97	54.40	5.93	1.85

SEM analysis

SEM (Scanning Electron Microscopy) analysis was carried out to assess the surface morphology of the Hap and the composite materials HAp-PEG 10% and HAp-PEG-CTS 5%, as depicted in Fig. 4. EDX (Energy Dispersive X-ray Spectroscopy) analysis was carried out to perform elemental composition analysis. SEM analysis shows the needle-like morphology of HAp synthesized from clam shells Fig. 4(a). However, with the addition of the PEG polymer, the needle-like shape of HAp undergoes agglomeration and changes to a more uniform and evenly distributed surface structure Fig. 4(b). Furthermore, the formation of the HAp-PEG-CTS 5% composite, with the addition of the chitosan polymer, results in morphological changes, forming dense clumps Fig. 4(c). EDX analysis also reveals the presence of elements Ca, P, O, C, and N, which are components of HAp, PEG, and chitosan Table 1. The Ca/P ratio obtained is 1.87, indicating that the HAp composite with PEG and chitosan polymers has been successfully synthesized. [20]

TG-DTA analysis

TG-DTA (Thermogravimetric-Differential Thermal Analysis) is used to analyze the thermal stability and decomposition behavior of HAp composites combined with PEG and chitosan (CTS). TG-DTA was conducted on the composite HAp-PEG.

In the curve of the HAp-PEG composite Fig. 5, a weight loss is observed at 122°C due to the release of residual OH groups in the composite and at 150–287°C due to the decomposition of organic compounds, specifically PEG. At temperatures >300°C,

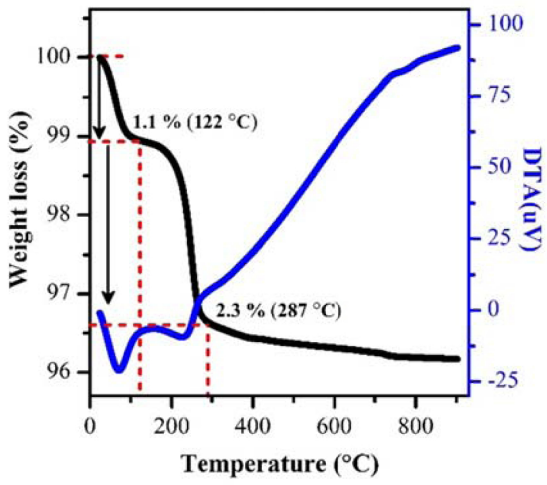

Fig. 5. The curve TG-DTA of Composite HAp-PEG.

no further weight loss occurs, indicating that the remaining compound (HAp) exhibits high thermal stability. Additionally, the DTA curve shows an endothermic reaction below 100°C and an exothermic reaction above 200°C. The DTA graph continues to rise after 300°C, and the TGA graph shows no further weight loss, indicating the thermal stability of the remaining compound, HAp. [7,13]

Antibacterial activity analysis

Fig. 6 illustrates the antibacterial activity of HAp, the HAp-PEG-CTS 5% composite, and the antibiotic CIP. HAp is used as a negative control, CIP as a positive control, and the HAp-PEG-CTS 5% composite as the test substance to evaluate antibacterial activity against *E. coli* and *S. aureus*.

Based on Fig. 6 and Table 2, HAp shows no inhibition zone against both types of bacteria, indicating its low q activity. The HAp-PEG-CTS-5% composite demonstrates an inhibition zone of 2 mm against E.*coli* and 5 mm against S. *aureus*. The formation of the inhibition zone indicates the antibacterial activity of the composite.

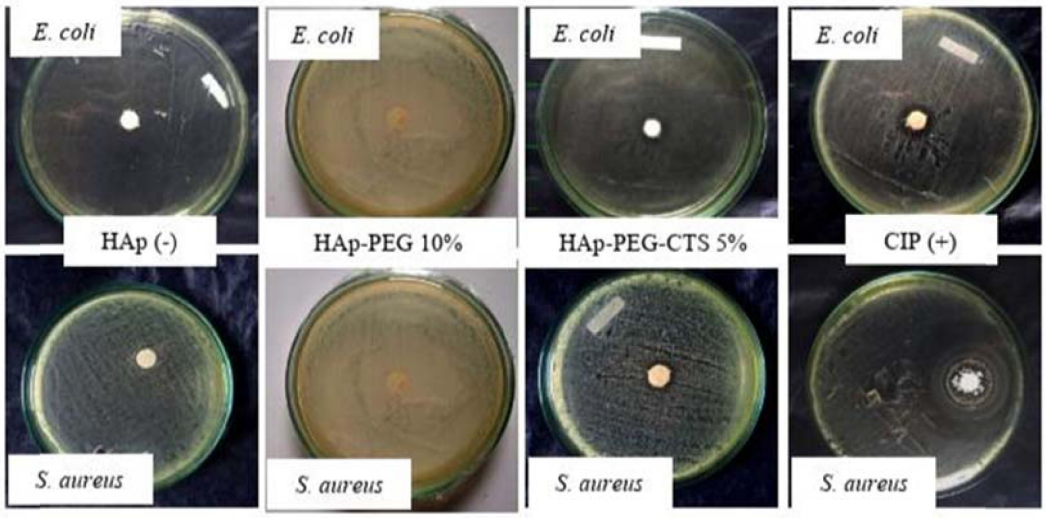

Fig. 6. The antibacterial activity of the negative control (HAp), the HAp-PEG-CTS 5% composite, and the positive control (Ciprofloxacin).

Table 2. Inhibition zone of HAp, HAp-PEG-10%, HAp-PEG-CTS 5% and CIP.

	Diameter Zona Inhibisi (mm)	
Material	*E. coli*	*S. aureus*
HAp	–	–
HAp-PEG 10%	–	–
HAp-PEG-CTS 5%	2 mm	5 mm
CIP	10 mm	20 mm

Compared to HAp (–), the addition of chitosan in HAp-PEG-CTS-5% enhances antibacterial activity due to the interaction between NH_3^- groups of chitosan and anionic components of the bacterial cell membrane, increasing membrane permeability and causing membrane lysis. In the positive control (CIP), an inhibition zone of 20 mm against *E. coli* and 30 mm against *S. aureus* was observed, indicating that CIP antibiotics exhibit susceptibility to both bacteria. [20]

The differences in the composite's antibacterial sensitivity towards the two bacteria are influenced by the structural differences in bacterial cell walls. *S.aureus* (gram-positive) has a thick peptidoglycan layer containing negatively charged teichoic acid, whereas *E. coli* (gram-negative) has a lipopolysaccharide layer, an outer membrane, lipoproteins, and peptidoglycan.

Conclusion

This research can contribute to the development of applications related to the health sector, because the development of biomaterials in this study has antibacterial and biocompatible properties for bone tissue engineering or medical devices. The synthesis of HAp-PEG-CTS composites with variations in polymers (PEG and chitosan) was successfully conducted. This is evidenced by FTIR analysis showing specific functional groups of each component (PO_4^{3-}, OH, C-O-C, N-H, and C-H), EDX analysis confirming the presence of elements Ca, O, P, C, and N with a Ca/P ratio of 1.87, as well as the similarity of the X-ray diffraction pattern with the ICSD #157481 standard. Additionally, thermal analysis revealed weight loss (150-300˚C) from the polymers, while HAp demonstrated good thermal stability. The effect of polymer addition on the resulting composites is reflected in increased mass with higher polymer variations, as well as changes in morphology and crystallinity. The antibacterial activity of the HAp-PEG-CTS composite showed an inhibition zone of 2 mm against *E. coli* and 5 mm against *S. aureus*, indicating that the composite possesses antibacterial properties.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images that are not ours have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.

- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Andalas.

Authors' contributions statement

N.A.T: Data curation; Formal analysis; investigation; Methodology; Writing-Original Draft; Writing-Review & Editing. N.J: Supervision; conceptualization; validation; Visualization and review. U.S: Supervision; validation; Visualization. V.S: Writing-Review and editing. M.R.N: Review and editing. S.M: Review and editing.

Data availability

Data are available upon reasonable request and will be provided by the corresponding author.

Funding statement

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استخدام مخلفات قشور الجمبري كمركب هيدروكسي أباتيت مع بوليمر حيوي من قشور الجمبري وبولي إيثيلين جلايكول وتطبيقاته

نوفيسار جاماروون[1]، نبيلا أيّو تريتشاياني[1]، أوبيتا سبتيني[1]، فيفي سيسكا[2]، محمد رفقي نوفيسار[3]، سري موليا[1]

[1]قسم الكيمياء، جامعة أندالاس، ليماو مانيس، بادانغ، سومطرة الغربية، إندونيسيا.
[2]مركز أبحاث الكيمياء، الهيئة الوطنية للبحث والابتكار، حديقة العلوم والتكنولوجيا بي. جي. حبيبي، سيربونغ، جنوب تانغيرانغ، بانتن 15314، إندونيسيا.
[3]قسم السياحة، المعهد الإندونيسي للفنون، بادانغ بانجانغ 27128، سومطرة الغربية، إندونيسيا.

الخلاصة

تركز هذه الدراسة على تركيب وتحليل البنية والخواص المضادة للبكتيريا لمركب يتكون من هيدروكسي أباتيت (HAp) وبولي إيثيلين جليكول (PEG) وكيتوزان (CTS). تم استخلاص الكيتوزان من قشور الجمبري، بينما تم استخلاص أكسيد الكالسيوم (CaO) من قشور بنسي للحصول على هيدروكسي أباتيت. تم تركيب هذه المركبات باستخدام طريقة سول-جل في الموقع، وذلك بتغيير تركيزات بولي إيثيلين جليكول (5، 10، 15، 20، و25%) والكيتوزان (1، 2، 3، 4، و5%). تم توصيف المركبات المُصنّعة باستخدام تحليلات FTIR وXRD وSEM-EDX وTGA-DTA، كما تم تقييم نشاطها المضاد للميكروبات. وقد تبين أن مركب HAp-PEG-Chitosan، الذي يحتوي على 5% كيتوزان و10% بولي إيثيلين جليكول، هو المنتج الأفضل، حيث ظهر على شكل مسحوق أبيض. كشف تحليل FTIR عن وجود مجموعات وظيفية محددة: $PO4^{3-}$ وOH من هيدروكسي أباتيت، وN-H من الكيتوزان، وC-H وC-C من البولي إيثيلين جلايكول (PEG)، مما يؤكد نجاح تكوين المركب. وأظهر تحليل XRD بنية سداسية متوافقة مع معيار حيود هيدروكسي أباتيت (ICSD #157481) وانخفاضًا في التبلور نتيجة إضافة البوليمر. وأظهر تحليل SEM أن هيدروكسي أباتيت الإبري الشكل، المستخلص من أصداف بنسي، قد خضع لتغيرات سطحية، مما أدى إلى نسيج أكثر تجانسًا عند دمجه مع PEG. وأكد تحليل EDX وجود عناصر الكالسيوم والفوسفور والكربون والأكسجين، مما يثبت نجاح تصنيع مركب هيدروكسي أباتيت-البوليمر. وأظهر تحليل TG-DTA فقدانًا في الوزن عند درجة حرارة أقل من 200 درجة مئوية، مما يشير إلى الثبات الحراري لـ PEG عند هذه الدرجة. تم اختبار النشاط المضاد للميكروبات لمركب هيدروكسي أباتيت-PEG-كيتوزان ضد بكتيريا الإشريكية القولونية والمكورات العنقودية الذهبية، مع استخدام مضاد CIP كعنصر تحكم إيجابي للمقارنة.

الكلمات المفتاحية: الكيتوزان، هيدروكسي أباتيت، طريقة في الموقع، بولي إيثيلين جليكول.