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Fourier Transform Infrared (ftir) Spectroscopic Analysis of Hydroxyapatite Scaffold Obtained from Channa striata and Scomberomorus commersoni Fish Bone

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Abstract

This study aims to analyze the functional groups in hydroxyapatite (HAp) scaffolds synthesized from the bones of (Channa striata) and (Scomberomorus commerson) using Fourier Transform Infrared (FTIR) spectroscopy. Fish bone waste contains calcium and phosphate; to obtain HAp powder, the bones were cleaned and dried, then calcined at 800℃ for 3 hours to produce HAp powder. The HAp powder was mixed with polyvinyl alcohol (PVA) as a polymer and honeycomb (HCB) was used as a biopolymer in scaffold fabrication with concentration variations of 10%, 20%, and 30%, then formed into scaffolds using the freeze-drying method. FTIR characterization results showed absorption bands in functional groups characteristic of phosphate (PO_4^{3-}) and hydroxyl (OH^-) groups indicating the formation of HAp, as well as the presence of PVA and HCB groups without eliminating the main HAp groups. The novelty in this research is the utilization of fish bone waste and beeswax as bioceramic materials in the fabrication of scaffolds that can be used for bone tissue engineering.

Keywords: Hydroxyapatite; fish bone; scaffold; honeycomb; bioceramic

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INTRODUCTION

Bone defects often occur due to tumors or infections and other external factors. Natural bone tissue has the ability to heal itself [1,2]. Bone grafting is considered the most effective method, but it has limitations due to limited bone supply and risks of infection and complications, especially for bone defects exceeding the critical threshold. To address these issues, a method used is bone tissue engineering, which is considered an alternative treatment for bone defects [3]. Three-dimensional scaffolds are widely used to facilitate bone tissue regeneration. Scaffolds are made using various methods including microsphere sintering [4,5], powder metallurgy method, combustion synthesis method [6].

In the study by Sadeghzade et al. [9], common materials for scaffold fabrication include ceramics, polymers, and metals that can be used. Autograft bone is considered the standard in bone grafting due to its osteogenic, osteoinductive, and osteoconductive capabilities. Another solution for bone tissue repair can use Hydroxyapatite (HAp), which is a type of bioceramic material that resembles natural human bone in both structure and chemical composition [7]. HAp can form a strong bond with bone tissue to promote bone growth and has phase stability, serving as a scaffold substitute in bone healing with good performance [8].

Bones naturally contain 20% organic material, usually type I collagen, 70% inorganic material in the form of HAp, and 10% water content [10]. In 2017, Indonesia experienced an increase in marine fish catches that impacted the fish processing industry, reaching 256,614 tons, while freshwater production reached 21,407 tons. Fish bones, which are fishery waste, account for 15% of the fish's body weight. Naturally, bones consist of 70% inorganic minerals, 20% organic material, and 10% water (Rohmah et al., 2019). The use of natural biopolymers in HAp production is one of the biopolymers derived from plants or animals. Biopolymers have the advantage of biodegradability, which is suitable for bone regeneration [11]. Honeycomb (HCB) is one of the natural biopolymers used in HAp production for forming pores in scaffolds, because HCB has a regular cell structure regular (Hayasgi et al., 2019). In scaffold fabrication, the freeze drying method is used because it can create pores ranging from micro to macro sizes uniformly [12]. This method does not damage the polymer structure during removal [13].

The PVA used acts as a binding material between HAp and HCB [14]. HCB is used as a natural biopolymer capable of forming pores in the scaffold. This study will also analyze the effect of HCB variation on scaffold characterization. The HCB variations used in this study are 10%, 20%, and 30% of the total sample weight for each HAp and HCB mixture, with a PVA to HAp ratio of 1:1 [15,16]. The scaffold manufacturing process is carried out using freeze-drying to produce a good porous structure without damaging the polymer structure in the scaffold [17]. The finished scaffold will then be characterized using Fourier Transform Infrared Spectroscopy (FTIR).

METHODS

Research Design

This research is an experimental study conducted in the laboratory involving HAp synthesis, scaffold fabrication, and characterization using Fourier Transform Infrared Spectroscopy (FTIR). This study was carried out at the Instrument Laboratory and final project lab, Faculty of Science and Technology, University of Jambi. This research uses a quantitative analysis approach.

Research Target/Subject

The target of this research is Hydroxyapatite (HAp) obtained from the bones (Channa striata) and (Scomberomorus commerson) as well as composite materials such as Polyvinyl Alcohol (PVA) and Honeycomb (beeswax). Channa striata and Scomberomorus commerson fish bones are waste materials that have good calcium and phosphorus content, making them potential sources for HAp production. The research samples consist of 3 scaffold variations, namely HAp-10, HAp-20, and HAp-30. Each sample uses the same variations. The selection technique is carried out based on the research objectives to examine the effect of honeycomb concentration variations on HAp functional groups. The materials used in this study are Channa striata and Scomberomorus commerson, aquades, acetone, PVA, and beeswax.

Research Procedure

The fish bones that have been obtained are then cleaned with water, with the tail section cut first. Next, they are boiled in boiling water; bony bones are boiled for 1 hour and Scomberomorus commerson bones for 15 minutes. The purpose of boiling is to remove any remaining meat on the bones and to make the bones softer for easier cleaning. The bones are cleaned, soaked in acetone for 30 hours to remove fat. The bones are then sun-dried for 4 days, dried in an oven at 100℃, and ground into powder.

Fish bone powder is then calcined at 800 ℃ for 3 hours to obtain pure HAp, and then sieved using a 250 mesh sieve. To make beeswax, prepare 500 grams of honeycomb, dissolve it in distilled water at 70℃ for 10 minutes, then filter the boiled solution to obtain beeswax from the honeycomb. To make the HAp solution, prepare fish bone HAp powder and PVA each weighing 2.55 grams, and dissolve them in 20 ml of distilled water at 95℃ for 1 hour. Scaffold preparation uses beeswax as a biopolymer with variations of 10%, 20%, and 30%. The prepared HAp and PVA solution is mixed with beeswax at different variations and homogenized for 30 minutes at 90℃ until it forms a gel. The finished sample is then frozen using freeze drying at -51℃ for 5 hours to produce a scaffold.

Instruments, and Data Collection Techniques

The type of data in this study is in the form of FTIR functional group characterization, which is useful for analyzing chemical compounds by identifying functional groups. The instruments used in this research are furnace, oven, hot plate stirrer, freeze drying, and Fourier Transform Infrared Spectroscopy (FTIR). Data collection techniques for characterization need to be prepared; the dried scaffold is then characterized using the FTIR instrument.

Data analysis technique

Data collection using FTIR was carried out to determine the functional groups of the HAp-PVA-honeycomb scaffold. FTIR analysis provides information about the bonds present in the HAp scaffold, such as phosphate bonds (PO_4^{3-}), hydroxyl (OH^-), as well as the functional groups from PVA and honeycomb. The HAp sample that had been made into a scaffold was then directly subjected to FTIR characterization. The FTIR results consist of absorption peaks, which were compared with FTIR reference data.

RESULTS AND DISCUSSION

Table 1. Group content in hydroxyapatite Channa striata fish bone

Wavenumber (cm^{-1})					
G.HAp/PVA /HCB 10%	**G.HAp/PVA /HCB 20%**	**G.HAp/PVA /HCB 30%**	**Literature**	**Functional Group**	**Reference**
568,20	568,92	568,92	500-611	PO_4^{3-}	(Suci et al., 2020)
820,68	811,25	832,29	794,67-1303,88471-1415	PO_4^{3-}	(Siregar dan Sulistyowati., 2019) (Purba et al., 2024)
1057,21	1056,48	1047,05	794,67-1303,881000-1150	PO_4^{3-}	(Siregar dan Sulistyowati., 2019) (Karisma et al., 2023)
1262,54	1262,54	1266,16	794,67-1303,88	PO_4^{3-}	(Siregar et al., 2018)
1423,61	1412	1423,61	1400-1550	CO_3^{2-}	Asmi, 2014
1700,04	1702,94	1710,19	1489-2156	CO_3^{2-}	(Utami et al., 2025)
2910,24	2908,78	2900,08	1967,39-4004,22	OH^-	Siregar et al., 2018
3290,42	3296,22	3321,62	1967,39-4004,223039-3749	OH^-	(Utami et al., 2025)(Purba et al., 2024)

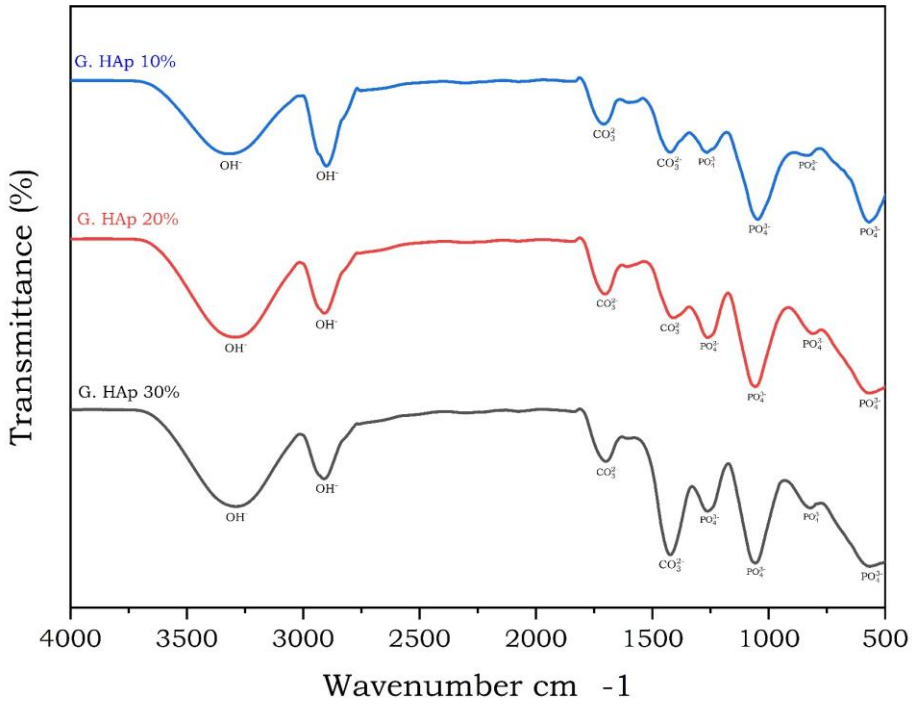

Figure 1. FTIR spectrum of hydroxyapatite (HAp) and Honeycomb (HCB)

FTIR characterization tests were carried out on HAp samples from Channa striata. FTIR characterization was performed in the wavelength range of 4000 cm-1 – 500 cm-1. Based on Figure 1, it shows that all samples have formed OH^-, (PO_4^{3-}), and ($\boldsymbol{CO_3^{2-}}$), functional groups, which are the characterizations of HAp functional groups [18]. At the wavenumbers of 568.20-568.92 cm-1, 820.68-832.29 cm-1, 1057.21-1047.05 cm-1, and

1262.54-1266.16 cm-1, there are (PO_4^{3-}) functional groups which represent stretching vibrations (Suci et al., 2020; Siregar and Sulistyowati, 2019; Purba et al., 2024; Karisma et al., 2023; Siregar et al., 2018). At the wavenumbers of 1423.61-1412 cm-1 and 1700.04-1710.94 cm-1 there are ($\boldsymbol{CO_3^{2-}}$), functional groups (Asmi, 2014; Utami et al., 2025). At the wavenumbers of 2910.24-2900.08 cm-1 and 3290.42-3321.62 cm-1 are $\boldsymbol{OH^-}$ functional groups, which are one of the components of hydroxyapatite, namely hydroxyl (Siregar et al., 2018; Utami et al., 2025; Purba et al., 2024).

The sharp (PO_4^{3-}) peak indicates increasing crystallinity growth. Good crystallinity corresponds to good HAp quality [23]. According to the research by Siregar et al. [22], the presence of the ($\boldsymbol{CO_3^{2-}}$), group is the result of a reaction between HAp and CO2 present during synthesis and heat treatment. The presence of the ($\boldsymbol{CO_3^{2-}}$), functional group cannot be considered harmful because human bones naturally contain ($\boldsymbol{CO_3^{2-}}$), as a substitution for (PO_4^{3-}) following the equation $\boldsymbol{Ca_{10}(CO_3)_X(PO_4)_{6-\left(\frac{2}{3}\right)X}(OH)_2}$, commonly called Carbonated Hydroxyapatite. However, because the presence of ($\boldsymbol{CO_3^{2-}}$), in this synthesis process is uncontrolled, it is categorized as an impurity. Based on Table 1, it shows that there is no significant change in the samples.

Table 2. Group content in hydroxyapatite from Scomberomorus commerson

Wavenumber cm-1					
T.HAp/PVA/HCB 10%	**T.HAp/PVA/HCB 20%**	**T.HAp/PVA/HCB 30%**	**Literature**	**Functional Group**	**Reference**
565,29	562,39	563,84	560-603	PO_4^{3-}	(Ramadhnona et al., 2023)
993,36	1041,25	1041,25	794,67-1303,881000-1150	PO_4^{3-}	(Siregar dan Sulistyowati, 2019) (Dewantoro et al., 2018)
1259,63	1259,63	1261,81	794,67-1303,88	PO_4^{3-}	(Siregar et al., 2019) (Siregar dan Sulistyowati, 2019)
1429,41	1410,55	1419,98	794,67-1303,88	CO_3^{2-}	(Siregar et al., 2019)
1714,55	1710,19	1712,37	1820-16001712	C=O	(Mentari et al., 2018)(Rohania et al.,2025)
2895	2910,24	2900,80	3700-2600	OH·	(Rahayu dan Kurniawidi, 2018)
3337,58	3304,93	3317,99	1630-3436	H_2O	(Purba et al., 2024)

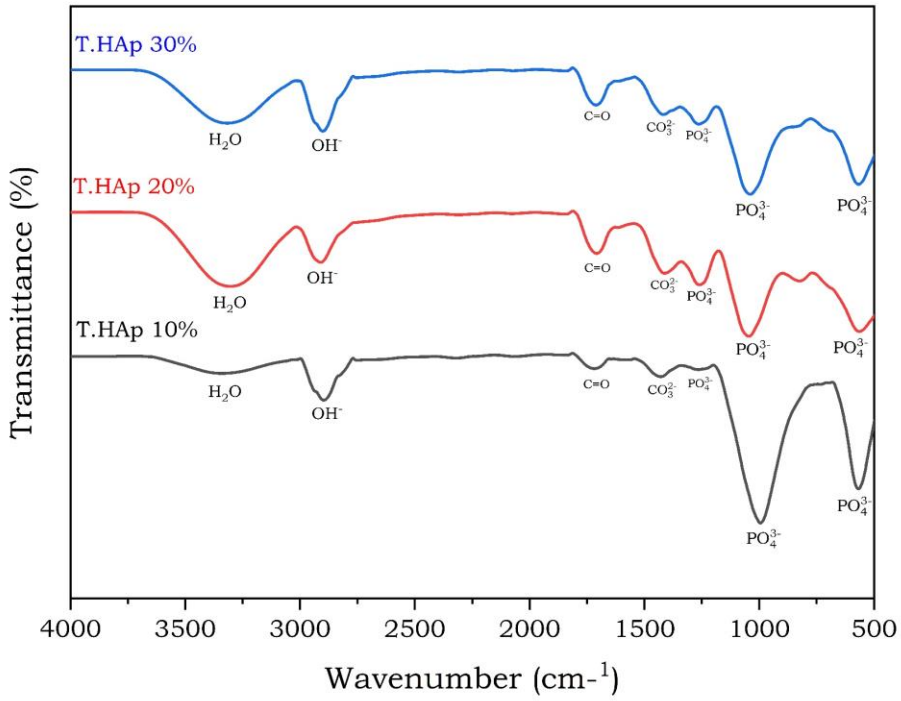

Figure 2. FTIR spectrum of hydroxyapatite (HAp) and Honeycomb (HCB)

FTIR characterization testing was conducted on Scomberomorus commerson HAp samples. FTIR characterization was carried out in the wavelength range of 4000 cm-1 – 500 cm-1. Based on Figure 2, it shows that all samples formed the functional groups OH^-, $\mathbf{PO_4^{3-}}$, $\mathbf{CO_3^{2-}}$, C=O, and H2O, which are characteristic functional groups of HAp [18]. At the wavenumbers 565.29-563.84 cm-1, 993.36-1041.25 cm-1, 1259.63-1261.81 cm-1, the functional groups of phosphate $\mathbf{PO_4^{3-}}$were observed (Ramadhona et al., 2023; Siregar and Sulistyowati, 2019; Dewantoro et al., 2019 and Siregar et al., 2019). At the wavenumbers 1429.41-1419.98 cm-1, the functional group $\mathbf{CO_3^{2-}}$ was present [20]. At the wavenumbers 1714-1712.37 cm-1, the functional group of C=O was observed [18,24]. At the wavenumbers 2895-2900.80 cm-1, the functional group $\boldsymbol{OH^-}$ was detected [23]. At the wavenumbers 3337.58-3317.99 cm-1, the functional group H2O was identified [21].

In the functional group C=O, the stretching vibration originates from PVA in the sample (Najim et al., 2022). According to Dewantoro et al. (2018), the phosphate group bond (PO_4^{3-}) exhibits the strongest stretching in the wave number range of 1000-1150 cm-1 and medium stretching at a wave number of 960 cm-1. The $\boldsymbol{H_2}0$ group appears due to the hygroscopic nature of CaO, which is highly reactive with H2O gas in the air, resulting in the formation of Ca(OH)2 [21]. According to Supangat and Cahyaningrum [25], $\mathbf{CO_3^{2-}}$is classified as an impurity that exists before heat treatment or as an impurity present in the hydroxyapatite base material. However, the inorganic composition of human bone contains carbonate, so the presence of the carbonate phase is not harmful to the human body. Based on Table 1, it is shown that there is no significant change in the sample.

CONCLUSIONS

Based on the FTIR characterization results of hydroxyapatite (HAp) scaffolds derived from Channa striata bones and Scomberomorus commerson bones, it can be concluded that the calcination process of fish bones at 800°C for 3 hours successfully produced hydroxyapatite powder with functional groups indicated by the appearance of phosphate (PO_4^{3-}) and hydroxyl (OH^-) absorption bands. The addition of honeycomb (HCB) with concentrations of 10, 20, 30% to the scaffolds of both Channa striata and Scomberomorus commerson bones did not eliminate the main functional groups of hydroxyapatite, so HAp still dominates the chemical structure of the scaffold. The FTIR spectrum shows the presence of functional groups from PVA and HCB, such as OH^- and C=O, indicating interactions among HAp, PVA, and HCB in the scaffold. The FTIR characterization results indicate that HAp/PVA/HCB scaffolds derived from fish bone waste have functional groups similar to those in human bone.

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AUTHOR CONTRIBUTIONS

In this study, Anggraini, R.M contributed to the conceptualization of the research, enabling it to run smoothly; Anggraini, R.M also developed the methodology for this study; Anjelina, R operated the software; Pujaningsih, F.B provided validation for this research; Afrianto, M.F conducted the analysis; Samsidar carried out the investigation; Resta, I.L provided resources for the research; Maulana, L.Z curated the data; Deswardani, F was responsible for writing and preparing the manuscript; Anjelina, R handled the review writing and editing; Deswardani, F managed visualization; Maulana, L.Z supervised the research; Anggraini, R.M managed project administration; Anggraini, R.M & Deswardani, F as a funding acquisition in the research conducted.

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