CHARACTERIZATION OF HYDROXYAPATITE FROM CHANNA STRIATA AND SCOMBEROMORUS COMMERSO FISH BONE BY HEAT TREATMENT

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ABSTRACT

Hydroxyapatite (HAp) biomaterials have been successfully obtained from Channa striata and Scomberomorus commerso fish bone. Heat treatment was chosen as the method to get it. Heat treatment was done at 400°C, 600°C, and 800°C. The X-ray diffractometer (XRD) was used to determine the crystalline phase and crystallography properties. Based on XRD data and JCPDS 09-0432, all samples showed the phase of HAp. However, the β-TCP phase has been found in samples from Scomberomorus commerso, which calcined at 600°C and 800°C. The crystallite size of HAp from Channa striata fish bone increased as followed by higher temperature, and Scomberomorus commerso has the same crystallite size on samples that calcined at 600°C and 800°C. The microstrain of both samples has the same trend: the higher the temperature, the lower the microstrain. The temperature also affected the crystallinity sample; the higher the calcined temperature, the improved crystallinity. Fourier Transform Infrared (FTIR) data determined the functional groups of samples. The functional groups that appeared were OH− and PO43−. The mass of samples before calcined differ from after calcined, where the mass after calcined is smaller than before. The higher the temperature we used, the more decreased yield percentage we got.

Keywords: Hydroxyapatite; Fish Bone; Channa striata; Scomberomorus commerso; Heat treatment

INTRODUCTION

Hydroxyapatite (HAp, Ca10(PO4)6(OH)2) is an inorganic mineral components containing calcium and phosphate. This substance has a similar chemical structure to human bones and teeth. Through this similarity, HAp as a biomaterial and bioceramic is often used for biomedical applications, particularly in orthopedic, odontology, and as an implants of coating material. HAp is made up of 70% bone, 20% collagen, and the other 10% of water (Mohd Pu’ad, et al., 2020).

HAp can be obtained from natural materials, especially organic waste which contains high calcium such as eggshell (Mohd Pu’ad, et al., 2021), bovine bones, pig bones, fish bones and scales (Horta, et al. 2023). Other sources of calcium can also be obtained from pigeon bone (Sharifianjazi, F., et al., 2021) and Turkey femur-bone (Esmaeilkhani, et al., 2019). Fish bones become interesting source of calcium for Hydroxyapatite as they are abundant and easy to obtain. Other research have been successfully extracted fish bones into hydroxyapatite, such as carp fish bones (Hammood, et al., 2019; Callob, et al., 2023), rabbitfish (Fendi, et al. 2023), sardinella longiceps (Surya, et al., 2021), and selayang fish (Afiqah et al., 2020).

A variety of methods have been used to get HAp, such as precipitation (Rizkayanti and Yusuf, 2019), microwave irradiation (Castro, et al. 2022), reflux method (Cahyanto, et al. 2017), heat treatment (Lolo, et al. 2022), interfacial reaction with multiple emulsion, Solid-state syntesis (Khalid and Chaudhry, 2020) cationic surfactant template (Yusuf, et al. 2019), sol-gel (Charlena, et al. 2022), etc, Heat treatment known as the simplest and cheapest method because there is no need to mix additional sources to obtain HAp (Sunil & Jagannatham, 2016).

The variation of temperature has been used to obtain HAp with heat treatment. The HAp was successfully obtained in Turkey Femur-Bone Waste after heated at 850°C – 1150°C (Esmaeilkhani, et al. 2019). The carp fish bones do the same after calcined at 900°C – 1100°C (Hammood, et al. 2019). However, lower temperatures enable the ability to obtain HAp from the fish bone. Characteristics of HAp (in major fraction) were observed when Sheelavati (roho labio) fish calcined starting at temperature 600°, and so does the milkfish bone source (Sunil & Jagannatham, 2016; Lolo, et al. 2022). The lower temperature has been used to get HAp from Lates calcarifer fish bone. The calcined
started at 200°C and observed the characteristics of HAp (Pal, et al. 2017).

This work studied the potency of two kinds of fish bone - Channa striata and Scomberomorus commerson fish bones – as the natural sources of HAp. Heat treatment was used as the method. The temperature of heat treatment were 400°C, 600°C, and 800°C. All samples were heated in the same heater and had the same duration. The X-ray diffractometer (XRD) and The Fourier Transform Infrared (FTIR) were chosen to determine the crystalline phase, crystallography properties, and functional groups of the samples.

METHOD

Channa striata and Scomberomorus commerson fish bones used as biogenic material were taken in Jambi, Indonesia. The fish bones were washed with water first and left in boiling water at 100°C for 10 minutes. Then, the leftover meat was cleaned with the aid of a brush. After that, the cleaned fish bones were placed in acetone for 48 hours. To obtain the dried fish bones, they were dried in direct sunlight for three days and then dried in an oven at 100°C for 3 hours. Completely dried samples were blended in a blender and then crushed with mortar to obtain powder samples and smaller particle sizes. Further, samples were put in the furnace at 400°C, 600°C, and 800°C for 3 hours. In this research, Channa striata samples were given the symbol G, while Scomberomorus commerson samples were given the symbol T.

RESULT AND DISCUSSION

The XRD determined the samples' crystalline phase and crystallography properties. Figure 2 shows the diffraction pattern resulting from the HAp samples. HAp phase has been found in all samples based on JCPDS 09-0432. Another characterization was done using FTIR (BRUKER Alpha II sample compartment RT-DLaTGS) to identify the functional groups of the samples, such as OH− and PO3−. The FTIR data was recorded in the range of 400 – 4000 cm−1. The sample was measured directly from the powder. The detector of FTIR is DTGS, and the measurement mode is attenuated total reflectant (ATR).

The samples were characterized using XRD (XPERT PRO PANalytical PW3040/60) to determine the crystalline phase and crystallography properties. The XRD data were recorded using CuKα radiation at λ =0,154 nm in the range 2θ: 10° – 100°. The XRD data was identified with the help of the Joint Committee on Powder Diffraction Standards (JCPDS) 09-0432. Another characterization was done using FTIR (BRUKER Alpha II sample compartment RT-DLaTGS) to identify the functional groups of the samples, such as OH− and PO3−. The FTIR data was recorded in the range of 400 – 4000 cm−1. The sample was measured directly from the powder. The detector of FTIR is DTGS, and the measurement mode is attenuated total reflectant (ATR).

Figure 1. Schematic of methods to fabricate and characterize HAp

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Figure 2(a) shows that different temperatures affected the amount of HAp phase. Samples heated at 800°C had the greatest peak of HAp, with three main peaks: (211), (112), and (300). It means the higher the temperature we used, the more HAp phase formed. The peak became sharper as the temperature increased. The width of the lines becomes narrower, indicating an increase in the crystallinity degree (Ahmed, et al. 2015). The broader peak at samples
were heated at 400°C and 600°C, meaning their crystallinity degree was smaller than those at 800°C.

The crystallography properties - such as crystalline size, microstrain, crystallinity, and lattice

![Figure 2](image1.png)  
**Figure 2.** (a) XRD patterns of HAp powders from Channa striata fish bone; XRD pattern of different sources of HAp after heat treatment at temperatures: (b) 400°C; (c) 600°C; (d) 800°C.

**Table 1.** Crystallography properties of HAp based on XRD data

<table>
<thead>
<tr>
<th>Samples</th>
<th>Crystallite size (nm)</th>
<th>Microstrain</th>
<th>Crystallinity (%)</th>
<th>Lattice parameter (Å)</th>
<th>c/a</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>a</td>
<td></td>
</tr>
<tr>
<td>G400</td>
<td>19.76</td>
<td>0.44</td>
<td>-</td>
<td>9.38</td>
<td>6.86</td>
</tr>
<tr>
<td>G600</td>
<td>26.35</td>
<td>0.33</td>
<td>59.5</td>
<td>9.39</td>
<td>6.89</td>
</tr>
<tr>
<td>G800</td>
<td>52.70</td>
<td>0.17</td>
<td>88.9</td>
<td>9.40</td>
<td>6.89</td>
</tr>
<tr>
<td>T400</td>
<td>19.76</td>
<td>0.44</td>
<td>-</td>
<td>9.38</td>
<td>6.87</td>
</tr>
<tr>
<td>T600</td>
<td>52.70</td>
<td>0.17</td>
<td>47.6</td>
<td>9.37</td>
<td>6.89</td>
</tr>
<tr>
<td>T800</td>
<td>52.70</td>
<td>0.17</td>
<td>75.3</td>
<td>9.41</td>
<td>6.87</td>
</tr>
</tbody>
</table>
Figure 3. FTIR spectra of HAp powders from Channa striata and Scomberomorus commerson fish bones.

Table 2. Characteristic functional groups of HAp based on FTIR data

<table>
<thead>
<tr>
<th>Samples</th>
<th>Wavenumber (cm⁻¹)</th>
<th>((\text{OH}^-))</th>
<th>((\text{PO}_4^{3-}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>G400</td>
<td>-</td>
<td>560</td>
<td>595</td>
</tr>
<tr>
<td>G600</td>
<td>632</td>
<td>3570</td>
<td>560</td>
</tr>
<tr>
<td>G800</td>
<td>632</td>
<td>3570</td>
<td>560</td>
</tr>
<tr>
<td>T400</td>
<td>-</td>
<td>558</td>
<td>599</td>
</tr>
<tr>
<td>T600</td>
<td>636</td>
<td>3570</td>
<td>560</td>
</tr>
<tr>
<td>T800</td>
<td>632</td>
<td>3570</td>
<td>556</td>
</tr>
</tbody>
</table>

Table 3. Yield percentage of samples after heat treatment

<table>
<thead>
<tr>
<th>Samples</th>
<th>Before heat treatment (g)</th>
<th>After heat treatment (g)</th>
<th>Yield Percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G400</td>
<td>9.2</td>
<td>6.1</td>
<td>66.6</td>
</tr>
<tr>
<td>G600</td>
<td>24.0</td>
<td>15.1</td>
<td>63.0</td>
</tr>
<tr>
<td>G800</td>
<td>20.5</td>
<td>12.5</td>
<td>61.1</td>
</tr>
<tr>
<td>T400</td>
<td>57.5</td>
<td>26.6</td>
<td>46.3</td>
</tr>
<tr>
<td>T600</td>
<td>42.3</td>
<td>17.3</td>
<td>41.0</td>
</tr>
<tr>
<td>T800</td>
<td>42.3</td>
<td>18.3</td>
<td>43.3</td>
</tr>
</tbody>
</table>

crystal that appeared as a distortion or dislocation. The low microstrain showed a few defect of crystal (Anggraini, et al. 2021).

HAp has parameter lattice \(a = 9.41 \text{ Å}\) and \(c = 6.88 \text{ Å}\) (based on JCPDS 09-0432). Lattice parameters \(a\) and \(c\) of both samples were almost the same. The different calcined temperatures did not affect the lattice parameter of HAp from fish bones. However, the temperature affected on crystallinity,
morphology, and crystal size of HAp (Sunil & Jagnanatham, 2016).

Figure 3 presents the FTIR spectra of HAp after heat treatment in different temperatures and from different sources. The hydroxyl (OH) and phosphate (PO$_4^{3-}$) functional groups appeared on samples. As shown in Table 2, the bands at 632-636 cm$^{-1}$ and 3570 cm$^{-1}$ represent OH$^-$ groups. Those bands were stretching vibration of low-intensity O-H groups (Shi, 2018). The samples exhibited transmittance of the functional groups of PO$_4^{3-}$ at 556-560 cm$^{-1}$, 595-601 cm$^{-1}$, and 1014-1026 cm$^{-1}$. The bands at 556-569 and 595-601 represent the bending mode of P-O groups (Sari and Yusuf, 2018; Pal, 2017), while 1014-1026 cm$^{-1}$ and 1088-1090 cm$^{-1}$ represents the stretching mode of P-O groups (Sari, et al. 2021; Siddiqi and Azhar, 2020).

Table 3 shows the yield of samples after the calcined process. The samples from Channa striata fish bones had a higher percentage than Scomberomorus commerson fish bones. Also, the higher the temperature we used, the more decreased yield percentage we got. It happened not only in the sample from Channa striata but also from Scomberomorus commerson.

**CONCLUSION**

Hydroxyapatite (HAp) has successfully produced from Channa striata and Scomberomorus commerson fish bones with heat treatment method. The characteristics of HAp, including crystalline phase, crystallographic properties, and functional groups, were influenced by the temperatures. The samples from Channa striata fish bones has pure HAp phase based on XRD data. However, a few β-TCP phase has found in samples from Scomberomorus commerson fish bones. The heat treatment did not affect lattice parameters, but changed the crystallite size, the microstrain, and the crystallinity. The higher temperature, the higher crystallite size and crystallinity and the lower the microstrain. Based on FTIR data, the functional group HAp has found, such as hydroxyl (OH) and phosphate (PO$_4^{3-}$). The temperatures also affected the mass of sample after calcined.

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