Journal of
Biomedical Science and Bioengineering

Review of the temperature and holding time effects on hydroxyapatite fabrication from the natural sources

Akhlis Rahman Sari Nurhidayat[1*], A.P. Bayuseno[1,2], Rifky Ismail[1,2], Rilo Berdin Taqriban[1,2]

[1]Department of Mechanical Engineering, Faculty of Engineering, Universitas Diponegoro, Indonesia
[2]Center for Biomechanics Biomaterials Biomechatronics and Biosignal processing (CBIOM3S), Universitas Diponegoro, Indonesia
Email : akhlisrahman22@gmail.com

ABSTRACT. Biomaterial development is currently being carried out to help people who have daily needs. Hydroxyapatite has biocompatibility properties and suitables for the use as a biomaterial. Hydroxyapatite can be found in natural sources sometimes as waste. One of the hydroxyapatite fabrication methods is calcination process. Calcination and sintering are used to obtain the desired Ca/P ratio of the hydroxyapatite. This paper reviews several research which have been published by researchers to withdraw the connection during calcination process, with respect to the temperature and holding time effects on hydroxyapatite fabrication from the natural organism. The effect of temperature and holding time determines the yield of Ca/P ratio which affects the resulting mechanical properties. Choosing the right temperature and holding time will produce Ca/P which meets the standard.

Keywords: Calcination, Ca/P, Hydroxyapatite, Mechanical properties, Sintering.
Article History: Received 23 March 2021; Accepted 17 April 2021; Available online: 20 April 2021
DOI: 10.14710/ jbiomes.2021.v1i1.27-31

1. INTRODUCTION

The use of biomaterials at this time helps the daily activities of people who have limitations caused by disease. Biomaterials are expected to be able to replace the same structure and function as damaged organs. Ceramic material is a material commonly used in biomaterials. Ceramics that are easy to process in various forms are very suitable for biomaterial needs, especially for implants. One of the biomaterials that continues to be improved in its process and use is hydroxyapatite (HAp). Hydroxyapatite can be used as a bone strengthener, tooth [1], drug delivery [2], or coating for implants.

Hydroxyapatite which will be used as an implant in the body must have biocompatibility that has no side effects in the body. It is should be nontoxic, noncarcinogenic, nonallergic, noninflammatory, biocompatible, and biofunctional for its lifetime in the host. HAp can be obtained by synthesizing or extracting. Natural HAP can be found in bio-waste consisting of animal antlers [3], mammal bones [4–6], fish bones [4, 7–10], clamshells [4, 11–14], and eggshells [4, 15–17].

Several methods have been developed in the synthesis of HAp including mechanochemical synthesis [18–19], precipitation methods [20–23], microwave processing [24], solid-state [25], and wet chemical methods [23, 26–27]. One method used in the extraction of HAp from natural sources is calcination [8, 10, 12, 16, 17]. Calcination temperature will affect the formation of HAp powder. Upon completion of the sintering process carried out in the formation of the HAp pellet. The influence of temperature will have an impact on the mechanical properties that will be obtained [28–32]. For more specific uses, HAp will be compatible with other materials.

HAp has calcium and phosphate by replacing the recommended ones. Each material and process will produce a different Ca/P ratio. Selection of temperature and holding time during calcination will affect the ratio. The ratio value of Ca/P already has a set of standards. Effect of other elements on the Hap is likely to affect the Ca/P value.

Its mechanical properties will also be affected by the sintering process. Sintering temperature will produce different results depending on the source of the material used. These parameters will be adjusted to which the HAp will be used, for example, implanted HAp into fractured human bone as bone graft. HAp properties correspond to the function and mechanical properties of the fractured bone that will be implanted.

In this paper, review effect of temperature used in the calcination and sintering processes will be determined. This paper discusses the HAp product sourced from natural materials which always available and often found in the environment, sometimes as waste.

2. HYDROXYAPATITE SOURCES AND FABRICATION

Hydroxyapatite can be extracted from bio-waste in the nearby environment. The use of bio-waste as the main source of natural hydroxyapatite is intended to support the movement to reduce waste and upscaling the economic value of the waste itself. Table 1 shows some bio-waste and the methods used.

Table 1
Summary of materials and methods using Calcination to extract Hap

Source	Method	°C	H	Ca/P	Product	ref
Bovine bone	Calcination	850	-	-	HAp	[29]
	Calcination	600	-	-	HAp	[33]
	Calcination	900	-	-	HAp	[33]
	Calcination	1200	-	-	HAp	[33]
	Calcination	700	3	-	HAp	[34]
	Calcination	900	3	-	HAp	[34]
	Calcination	1100	3	-	HAp	[34]
	Calcination	750	4	-	HAp	[24]
Porcine Bone	Calcination	600	-	-	HAp	[33]
	Calcination	900	-	-	HAp	[33]
	Calcination	900	-	-	HAp	[33]
Caprine Bone	Calcination	900	2	2.2	HAp	[35]
	Calcination	1000	2	1.9	HAp TCP	[35]
	Calcination	1100	2	1.8	HAp TCP	[35]
	Calcination	1200	2	1.8	HAp TCP	[35]
	Calcination	1300	2	1.7	HAp TCP	[35]
Egg Shell	Calcination	700	2	-	HAp	[16]
	Calcination	900	3	-	HAp	[17]
Clam Shell	Calcination	900	5	1.67	HAp	[12]
	Calcination	1000	5	2.18	HAp TTCP	[12]
Fish Bone	Calcination	600	-	1.86	HAp	[36]
	Calcination	950	-	1.84	HAp	[36]
	Calcination	700	1	-	HAp	[8]
	Calcination	700	2	1.83	HAp	[10]
	Calcination	700	2	1.8	HAp	[10]
	Calcination	700	2	1.82	HAp	[10]
	Calcination	700	2	1.69	HAp	[10]
	Calcination	600	2	1.53	HAp	[37]
	Calcination	600	-	1.87	HAp	[36]
	Calcination	950	-	1.89	HAp	[36]
	Calcination	600	2	1.51	HAp	[37]
	Calcination	600	2	1.55	HAp	[37]
	Calcination	600	2	1.42	HAp	[37]
Fish Scale	Calcination	200	1	-	HAp	[22]
	Calcination	400	1	-	HAp	[22]
	Calcination	800	1	-	HAp	[22]
	Calcination	1000	1	-	HAp	[22]
	Calcination	1200	1	1.71	HAp TCP	[22]

H = Time (Hour)

The trace elements in the hydroxyapatite powder will produce different ratios for each source. Mass spectrometry testing will show all the elements contained in the powder other than Ca and P. The temperature and holding time given also affect the results of the ratio.

The elements contained in raw materials can affect the resulting Ca/P value. Other elements which could be found in HAp are Al, Na, Mg, and Sr. Other elements such as Na and Mg can substitute Ca [38]. Na and Mg can help enhance the growth/development of bones and teeth.

The research conducted by Vidhya, et. al [39] showed that the Mg content in eggshells is 0.65% At. Meanwhile, the research conducted by Nam, et. al. [10] using catfish as HAp raw material informs that the Mg content in HAp by calcination process at 700 °C was 2.03 mg. In the research of Goto and Sasaki [37] and Nam, et. al. [10] using tuna fish resulted in different amounts of Mg. This can be caused by the environment or the habitat the tuna lived in.

Using the substitution equation, the molar ratio of (Ca + Mg + 0.5Na + 0.5K)/P is not much different from the Ca/P molar ratio. However, the increasing of molar ratio occurred in Goto and Sasaki [37] from 1.55 to 1.63 while in Nam, et. al. [10] from 1.69 to 1.71 which are both using tuna as raw material.

3. CHARACTERISTICS OF THE HYDROXYAPATITE FROM DISTINCT FABRICATION

The characteristics of HA from the calcination process are known through the Ca/P ratio that complies with the standard 1.67 molars. In the study of Goto and Sasaki [37] in Table 1 using fish as a material at a temperature of 600 °C Ca/P values below the specified stoichiometry. While research conducted by Nam, et.al [10] using different fish bones with temperature calcination of 700 °C with a holding time of 2 hours produces a range of 1.69-1.83 Ca/P. Such results indicate the potential for a fishbone temperature is at 600 -700 °C with a holding time of 2 hours.

The study from Alif, et.al [12] which used clamshell as material needs 5 hours holding time to get a Ca/P of 1.67 following the required standards. Calcination temperature and holding time that will determine the value of calcium and phosphate needed in the HAp need further synthesis to get results that are under the standards.

In Figure 1, some Ca/P values of animal bones are calculated concerning the temperature used in the heat agreement. In the bovine study of Ramesh, et.al [40] compared to Niakan, et. al [5] the Ca / P value produced has a large enough value. At a temperature of 1000 °C Ca/P values produced by Ramesh, et.al more values are temporarily standardized in Niakan, et. al is much higher. The decrease in Ca / P values in bovine [5] at temperatures of 800 °C to 1000 °C is stable, in contrast to bovine [40] which decreases dramatically at 1000 °C.

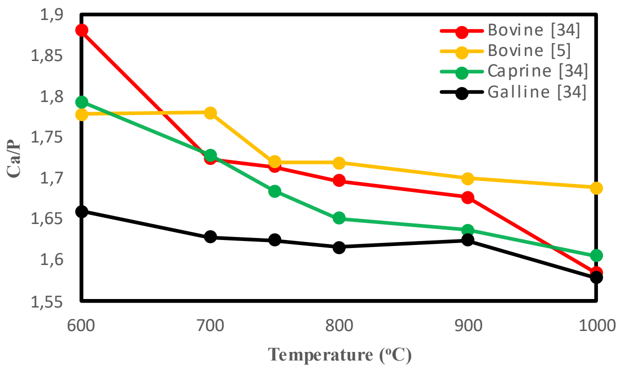

Fig 1. Effect of temperature on Ca / P values

Increasing the temperature used in the calcination will cause the formation of β-HAp which affects the Ca / P ratio value. The higher calcination temperature used, the higher possibility of TCP will form. It will affect the total HAp mass, because the more TCP forms, the least HAp will forms [41]. It was known that β-TCP appears at 1000 ºC calcinaction temperature, which was reported in the study of Barua, et. al [35].

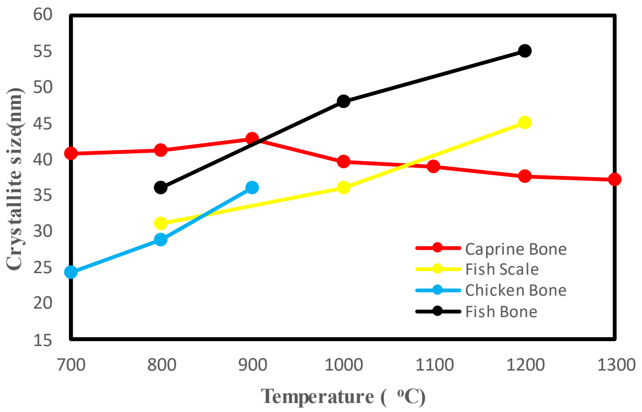

Fig 2. Comparison of crystallite sizes from several HA sources

The temperature calcination will also be the crystallite size shown in Figure 2 between caprine bone [35], fish scale [22], chicken bone [43], and fishbone [44]. An increase in temperature will increase the size of crystallite [22, 42]. The crystal size will also affect the mechanical properties of the product generated.

Figure 3**.** presents the ratio of relative density (%) to the increase in sintering temperature used. An increase in relative density occurs with an increase in temperature. In the study of Ramesh, et.al [16] relative density reaches a maximum value at a temperature of 1350 ºC. The use of eggshells [15, 16] and fishbone [8] in the relative values obtained can reach a maximum value at a temperature of 1250 ºC.

While in calcinated caprine bone [35] the maximum value is around 90%. In the research of Piccirillo, et. al [8] the sintering process is done after the calcination process which makes the value of the resulting relative density above 95%. This will cause the porosity of HA to be smaller with increasing sintering temperature [46].

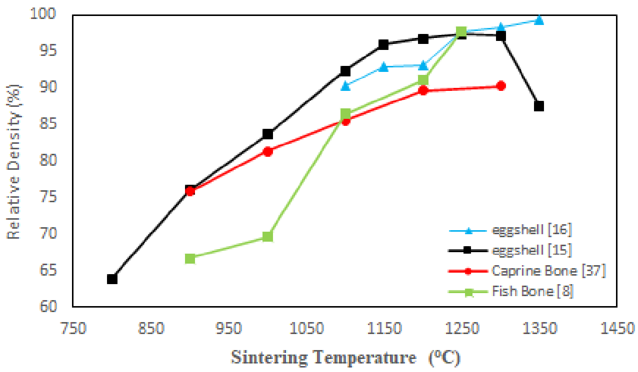

Fig 3. Comparison of relative density to heat treatment

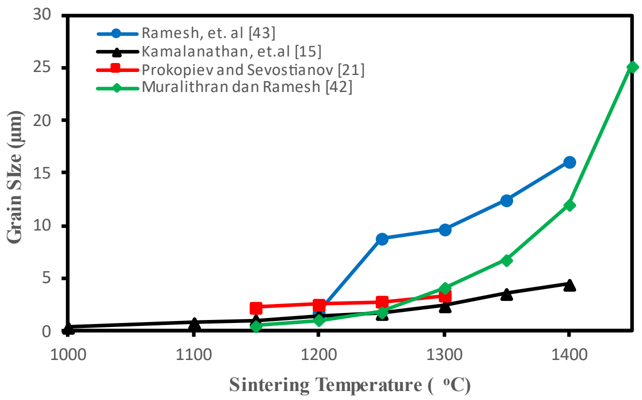

Fig 4. Comparison of grain size and sintering temperature

Similarly, the relationship between sintering temperature and relative density, from 4 studies [15, 21, 42, 43] the value is almost the same at 1200 ºC at the grain size shown in Figure 4. The increase in sintering temperature in grain size is obtained. When connected between the value of relative density with the value of grain size has a different value at vulnerable sintering temperatures of 1200 ºC and 1250 ºC. By heating above 1300 ºC, research by Muralithran and Ramesh [45] experienced a large grain size enlargement. In contrast to Prokopiev and Sevostianov [21], the increase in sintering temperature does not significantly increase grain size. HA unit cells did not experience the size change reported in Sunil, et al [9].

Figure 4. Comparison of grain size and increasing the sintering temperature will change the mechanical properties of the HAp itself. The effect of grain size enlargement will have an impact on the value of hardness and fracture toughness that HAp has. Figure 5 shows the effect of sintering temperature on HAp hardness. Some studies experience maximum hardness values at temperatures of 1250 ºC [15, 16, 42] and 1300 ºC [48].

In contrast to other studies, in the ark clamshell [49] the resulting value is not too large at an increase in temperature. The influence of grain size during the sintering process affects the fracture toughness value presented in Figure 6. Increasing sintering temperature will increase the fracture that occurs [50]. The optimal fracture toughness value is obtained at a sintering temperature of 1250 ºC.

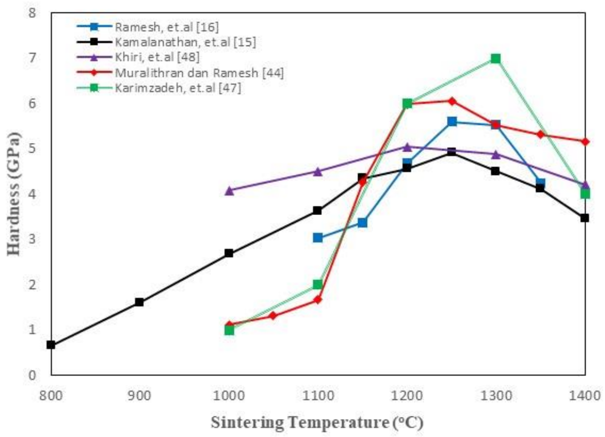

Fig 5. Effect of sintering temperature on the value of hardness

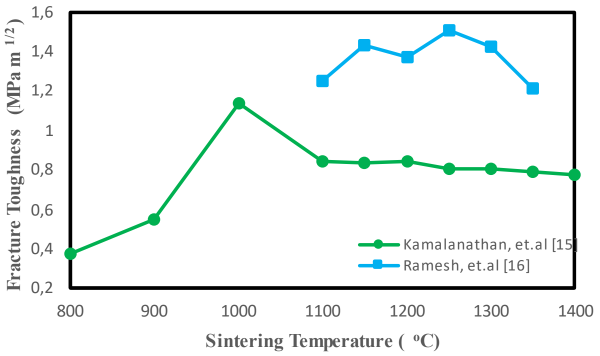

Fig 6. Effect of sintering temperature on fracture toughness values

The hardness of the Hap will be adjusted according to its use. Hardness of human cortical bone in a study by Mirzaali, et. al. [51] was confirmed at 455 ± 78 MPa. Based on this results, it is necessary to know the predicted activities of the patients who will use the HAp product as bone graft. The load and direction of the bone movements also need to be predicted, whether compression, torsion, or tensions.

4. Conclusion

The calcination process for each base material has an uncertain time and temperature to obtain a standardized Ca/P. Increase in sintering temperature will affect the mechanical properties of HA. Selection of temperature calcination and sintering is optimized for the use of HA. an increase in sintering temperature will affect the mechanical properties of HA. The choice of temperature calcination and sintering is optimized for the use of HA.

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