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Optimization of Process Parameters for Porous Artificial Bone

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Abstract- In this study, porous artificial bone from hydroxyapatite was fabricated using garbonyl diamide space fille. In porous bioceramics applications has low mechanical properties, therefore new developments are required on production of porous ceramics. İn this study, calcium-phosphate in hydroxyapatite from (Nanotech Co. in Eskişehir) and carbonyl diamide balls in 50% rati were used to prepare the powder. To optimize the fabrication, sintering temperatures (950 ℃, 1050 ℃, 1150 ℃) and time (1-5h) wereoptimse to enhance the mechnical properties of porou ceramics. Afte sintering, pore size and microstructure of the samples were analyzed with scanning microsopy. Porous samples (> 600µm) were tested with compression test. From the results compressive strenght were changged between 1.2 MPa to 11MPa depends on the process parameters.
Keyword- artificial bone, hydroxyapatite, sintering, porous, comprerssion

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I. INTRODUCTION

In the biomaterial area calcium-phosphate hydroxyapatite is an important interesting gain, similar to human bone and different researches are carried out. In the human body, hydroxyapatite bioceramic is used in various regions: orthopedics, prosthetic coating, dental floss and filler, cranial skull, back bone in surgery, bone fixations and many other biomaterials. Among the most interesting bioceramics are calcium-phosphate Hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$, the elemental composition and the apatite contained in it have a great similarity with the minerals in the inner structure of the bone and teeth [1, 2].

Calcium-phosphate hydroxyapatite is one of the most important properties of high porous bioceramic production with high biocompatibility, slow degradation, mineralization in human bone, chemical and biological structure, and it is in the production of bio-medical material [3]. Hydroxyapatite bioceramic weaknesses of the mechanical properties of the higher level and biological properties to reach a higher degree of hydroxyapatite high-porosity bioceramic production studies are increasingly important [4-6]

Calcium phosphate hydroxyapatite lattice structure is hexagonal rhombic shape, cell boutu; $a = b = 9.432^o\,A$ ve $c = 6.881^o A'\,dur$. Calcium phospot has the most valuable ratio $Ca\,/\,P\, =\, 10/6$, the calculated density is $3.219\ gr/cm^3$. The mechanical properties of hydroxyapatite bioceramic are shown in Table 1 below [7].

Table 1. Mechanical properties of hydroxyapatite and porous bone [7,12]

	HaP	Porous Bone
Elastic modulus (GPa)	4.0-117	0.05-0.5
Compressive strength (MPa)	147	0.1-16
Density (theory, g/cm^3)	3.16	1-2.5

The bone has a 75% porous mesh structure in the trabecular region. The size of the pores in the cortical part of the human bones is 10 nm to 50 μm, pore sizes of trabecular range from 50 to 500 μm [7]. High porosity bioceramics has shown better support for new bone formation and biocompatibility. Porous diameter of bone tissue mineralization should be at least 100 µm. The bioceramic pore size in bone tissue engineering demonstrates better biocompatibility with a diameter of at least 300µm. Macro-sized porous bioceramics have better biocompatibility than intense bioceramics, increased the development of porous and porous biosynthetic bone filling materials used to fill bone defects not exposed to load and external forces [8].

II. MATERIALS AND METHOD

In this study, the production section of the calcium-phosphate hydroxyapatite high porous bioceramics was carried out in three steps. In the first step, the hydroxyapatite powder was mixed in PVA 6000 pure water, the mixture was dried in a vacuum oven for 20 hours, then passed through a 100 µm sieve. In the second step mixes hydroxyapatite powder and 50% carbon diamide balls (average ball size 1.98 mm), the mixture was pressed in a tubular shape at a pressure of 595 MPa in a metallic mold. In the third step at the heat treatment stage, the samples were sintered at 950℃, 1050℃, 1150 ℃ and 1-5 hours. Density and compressive strength results were

compared depends on temperature and time. As shown in the production diagram Fig. 1 and Fig.2 gives the process diagram and sintering regime of the samples.

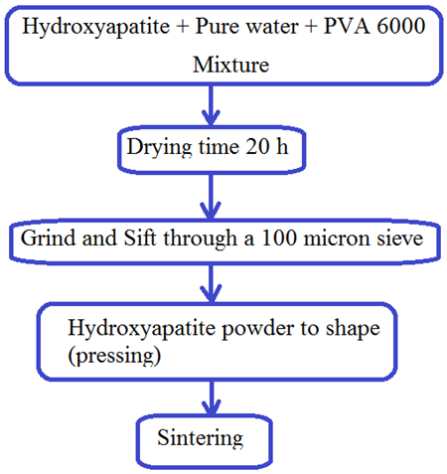

Fig. 1 Production diagram.

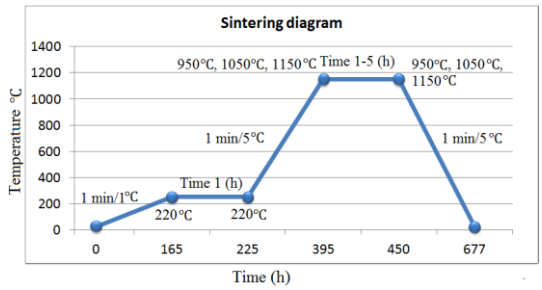

Fig. 2 Sintering regime of samples.

III. RESULTS AND DISCUSSION

Dimensions and weight of the samples obtained were measured and the density of the samples was calculated. The results of the test and the density of the samples show in Figure 2. As given the best conditions are 1150º C for 2h. Hydroxyapatite porous bioceramic production method in the comparing the results most optimal temperature and sintering time were determined. In future studies reinforced hydroxyapatite composite in different additives makes use of porous bioceramic production. Table 2 summarise the the results for 950 ℃, 1050 ℃, 1150 ℃ and 1-5 hours.

Table 2. The test results with temperature and time.

	950 ℃	1050 ℃	1150 ℃
1 saat	1.277 MPa	1.492 MPa	3.36 MPa
2 saat	1.766 MPa	2 MPa	4.998 MPa
3 saat	2.445 MPa	2.285 MPa	4.085 MPa
4 saat	-	1.7533 MPa	2.46 MPa
5 saat	-	1.662 MPa	2.16 MPa

Samples at 1150 ℃ temperature and 2 hours showed the best compressive strength, samples obtained in 3-5 hours, resistance to compression test each time it was reduced in the previous hour. Samples obtained are at 950 ℃ and 1050 ℃ temperature showed less durability in pressure testing than 1150 ℃ to the obtained samples. At the same time, the samples are obtained at 950 ℃ and 1050 ℃ for 4 - 5 hours a large number of samples were destroyed. Density shows in Figure 3, the samples at 1150 ℃ temperature have shown similarity diagram, samples at 950 ℃ and 1050 ℃ temperatures up to 3 hours, but samples obtained in 4 and 5 hours density calculation can't be done because it is ruined.

Samples at the forming stage at press values 295 MPa, 443 MPa and 595 MPa on 10 pieces best compressive strength it has been identified, Figure 4 shows the porosity of the samples. Comparing the porosity of samples, samples obtained at a pressure of 595 MPa showed minimal porosity, then the samples obtained under pressure of 295 MPa and 443 MPa. After forming of the samples and sintering the porosity are shown in Figure 4 and compression test shown Table 3.

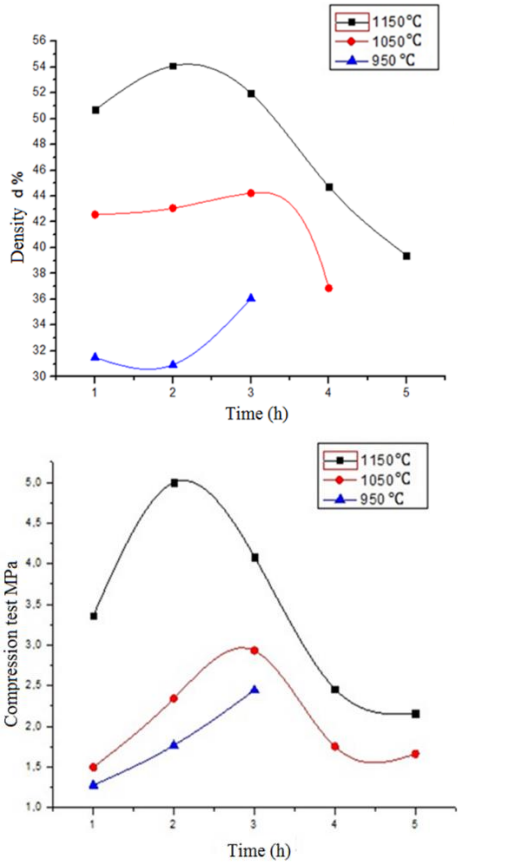

Fig. 3 a) Samples density (%), b) Compression test obtained samples.

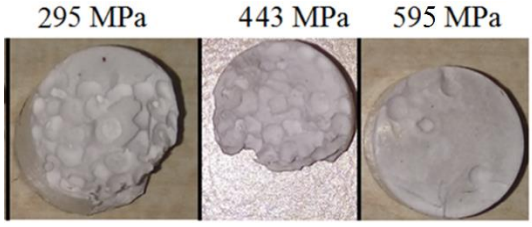

Figure 4. Images of the samples for various shaping load

Table 3. The results of compression test after forming and sintering.

Forming stage, sample press	Compression test
295 MPa	11.56 MPa
443 MPa	6.88 MPa
595 MPa	4.99 MPa

Figure 5 shows the samples obtained at the stage of formation at various pressure loads, after formatting the samples were synthesized at 1150 ℃ temperature and 2 time (h). The obtained sample with a press of 295 MPa showed the best results in the compression test 11.56 MPa shows, other samples showed less result. Due to this the subsequent samples were pressed 295 MPa and the porosity is examined.

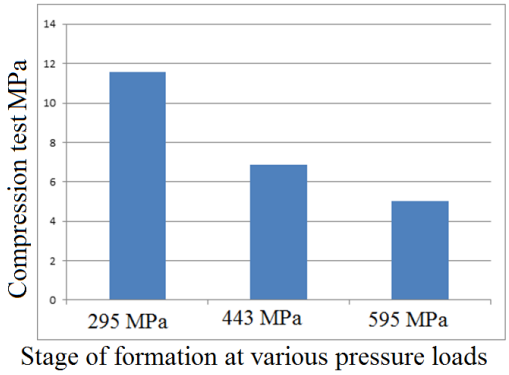

Figure 5. The compressive behaviour with various shaping loads.

In this section, samples hydroxyapatite porous bioceramic samples the pore size was measured. By dividing the samples into the middle and under the microscope, pore sizes were measured. Samples obtained at a temperature 1150 ℃ and 1-5 hours, the average pore size was calculated and the result was 1.085 mm, about 50 - 45% pore size decreased. Obtained samples of different time (h) sintered the pore structure showed similarity. The samples pores is shown in Fig.6.

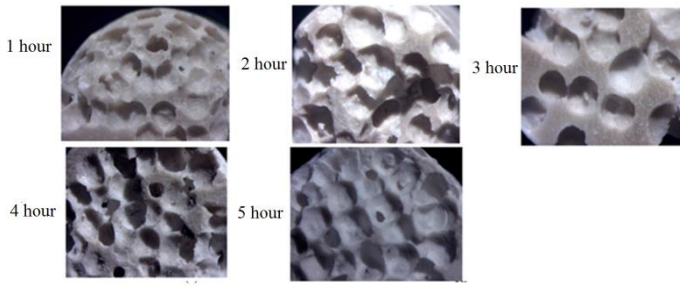

Fig. 6. Samples pores

IV. CONCLUSION

- Hydroxyapatite high porous bioceramics production was determined in the process sintering optimumit at 1150 ℃ temperatures and 2 time (h).
- The press value for shaping the samples was optimal at 295 MPa and the resulting sample was obtained with a compressive strength of 11 MPa, minimum compressive strength it at 950 ℃ and 1 time (h) at the stage of formation sample applied press 595 MPa.
- Samples obtained at a temperature of 1150 ℃ pore size decreased by about 50 - 45%.

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