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DC and AC conductivity properties of bovine dentine hydroxyapatite (BDHA)

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Abstract. Bovine dentine bio-waste may be used as a potential natural source of hydroxyapatite (BDHA), thus extraction of bovine dentin hydroxyapatite (BDHA) from bio-waste is significantly important to fabricate in a simple, economically and environmentally preferable. DC and AC conductivity properties of BDHA were investigated depending on sintering temperature (1000 ℃ - 1300 ℃) in air and vacuum (<10^{-2} mbar) ambient at room temperature. DC conductivity measurements performed between -1 and 1 V. AC conductivity measurements performed in the frequency range of 40 Hz – 100 kHz. DC conductivity results showed that dc conductivity values of the BDHA decrease with increasing sintering temperature in air ambient. It is not observed remarkable/systematic behavior for ac conductivity depending on sintering temperature.

1. Introduction

Recently, hydroxyapatite (HA) has widely used as a crucial biomaterials due to brilliant properties of biocompatibility and high osteogenic potential. Its crystallographic and chemical properties closely resemble those of bone and tooth minerals [1]. Therefore HA is commonly used in in several hard tissue orthopedic applications. There has been attracted attention in the last years for the investigation of the electrical properties of HA. Valerio and coworkers [2, 3], presented their reports showing constructive contribution of higher conductivity on bone healing for HA composite. Another study

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were performed by Rodrigues and coworkers on piezoelectric properties of the composites since it is well known, that one of the mechanism for bone growth is electrochemical process due to the generation of piezoelectric dipoles [4]. Electrical characterization is very informative tool to study for some process such as dehydration and dehydroxylation in HA [5] and investigate variations in microstructure [6].
Up to now, a small number of reports were presented on electrical characterization of HA. Because of the importance of electrical characterization of these materials, since positive contribution of higher conductivity on bone growing, there is a need to study of HA based materials. In this study dc and ac electrical properties of the BDHA were investigated as a function of sintering temperature, in air and in vacuum ambient, in dark and at room temperature.

2. Materials and experimental procedure

Extracted bovine teeth were obtained from local shop in Istanbul. The teeth were immersed in an antiseptic solution to avoid bad smell and possible contamination caught during preparation course. The teeth were cleaned slightly with continuous distilled water irrigation and then deproteinized in an alkali solution. After drying process, teeth calcined at 850^0C for nearly 4 h to remove any proteins and prions. Dentine was clearly peeled off from enamel. The obtained dentine samples were ground by a ball grinder for 4 h (Planetary Ball Mill PM 200 Retsch, Haan, Germany). Sheep dentine powders were sieved. The dentine powders were obtained nearly 100-150 μm. The prepared powders were pressed between hardened steel dies under the pressure of 350 MPa. The green bovine dentine compacts were sintered for 4 h at different temperatures (1000, 1100, 1200, and 1300^0C) (Nabertherm HT 16/17, Lilienthal, Germany). Four pellets were prepared in order to investigate sintering temperature effects on BDHA. The prepared pellets of the composites were presented in Table 1. Silver electrodes were deposited onto both sides of pellets of BDHA in high vacuum ambient ($<10^{-6}$ mbar) using thermal evaporation method to prepare the composites to measure current passing through the pellets. DC and ac conductivity measurements were done on the composites in air and in vacuum ambient ($\sim 2x10^{-2}$ mbar) at room temperature. The measurements were performed in dark ambient to prevent the current from light effects. Current passing through the composites were measured using Keithley model 6517B programmable electrometer and recorded by applying voltages between the -1 V and 1 V with 50 mV steps to the terminals of the pellets. AC conductivity measurements were done on the composites between the frequencies 40 Hz - 100 kHz using Keithley model 3330 LCZ meter. All the measurement system was computerized.

Table 1. Prepared Bovine Dentine Hydroxyapatite (BDHA)

Label of the Bovine Dentine Hydroxyapatite	BDHA 1000	BDHA 1100	BDHA 1200	BDHA 1300
Sintering Temperature [^{o}C]	1000	1100	1200	1300

3. Results and discussion

Electrical measurements were performed in order to determine effects of sintering temperature on electrical properties of the BDHA. Electrical conductivity measurements were done in air and in vacuum ambient ($<10^{-2}$ mbar) in dark at room temperature on the BDHA sintered at the temperatures of 1000^oC - 1100^oC, 1200^oC, and 1300^oC. Conductivity of the BDHA were calculated using the slope of current - voltage (I-V) curves between the applied voltages of -1 and 1V. Figure 1 shows current - voltage (I-V) curves of the BDHA in air and in vacuum ambient sintered at indicated temperatures. I-V curves of the BDHA revealed ohmic behavior in air and in vacuum ambient. As can be seen from the Figure 1 hysteresis was observed in I-V curves obtained in air and in vacuum ambient. The hysteresis may arise from deep traps as previously reported [7, 8]. These processes need longer time constant for charging and discharging process. Temperature, voltage sweeps speed and deep trap level may affect the hysteresis.

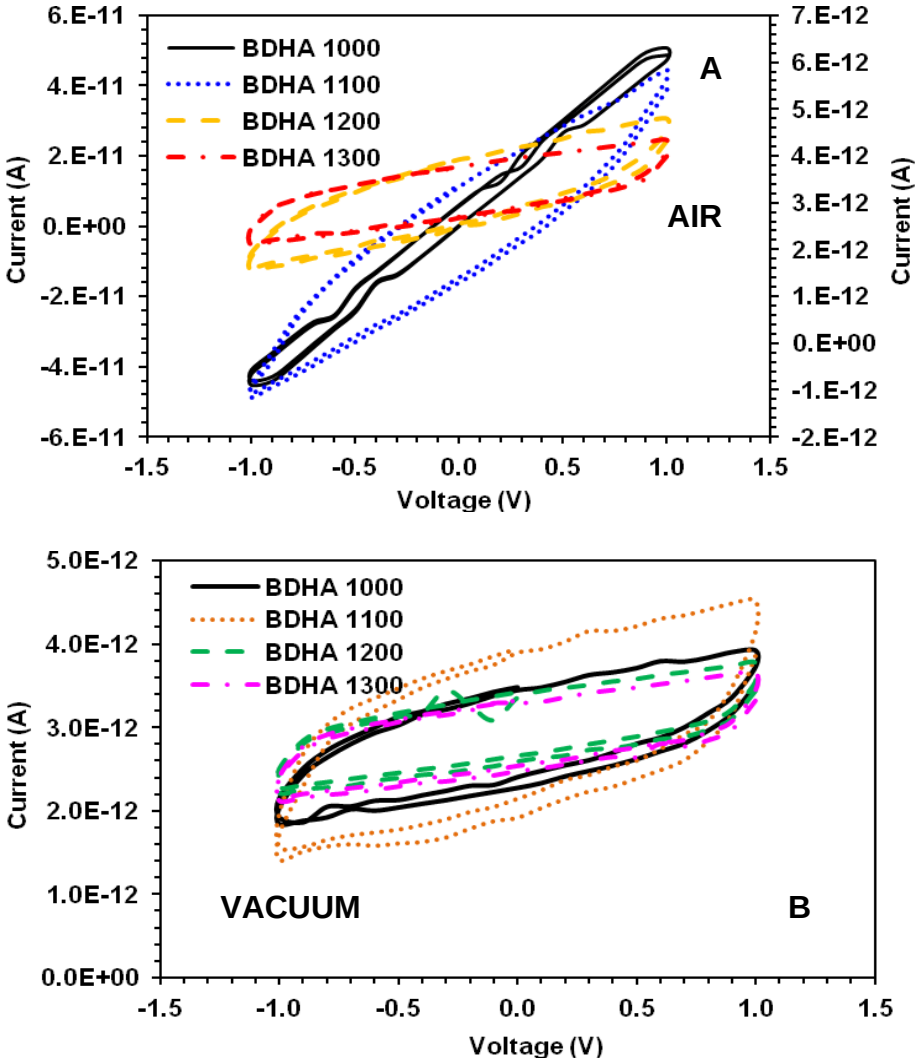

Figure 1. Current-Voltage (I-V) curves of the BDHA obtained in a dark (a) in air ambient (BDHA 1000 uses left-axis, and the rest of the BDHA use right-axis) (b) in vacuum ambient

Figure 2 shows the calculated d.c. conductivity values of the BDHA as a function of sintering temperatures in air and in vacuum ambient. DC conductivity value was calculated as 8.33×10^{-9} S/m for the BDHA1000 and decreased to 1.21×10^{-10} S/m for the BDHA 1300 in air ambient. In contrast to the behavior in air the values were fluctuated depending on sintering temperatures in the temperatures range of 1000°C - 1300°C. The values took place 6.53×10^{-11} S/m and 1.34×10^{-10} S/m. By inspecting Fig. 2 effects of sintering temperature can be seen easily. It might be said that since the scale of vertical axis is logarithmic scale dc conductivity values were strongly affected from sintering temperature. The effect of the sintering temperature on dc conductivity of the BDHA can be obtained more clearly by eliminating effects other than temperature such as humidity and oxygen. Because of this fact the measurements were also performed in vacuum ambient. The conductivity of the BDHA tends to decrease with increasing sintering temperature in vacuum ambient. The behavior may be explained as decrease in resistance in two ways: i) decreasing the particle size with increasing sintering temperature that results the decrease in grain boundary resistance, ii) increasing sintering temperature may small changes in molecular structure that results the decrease in bulk resistance.

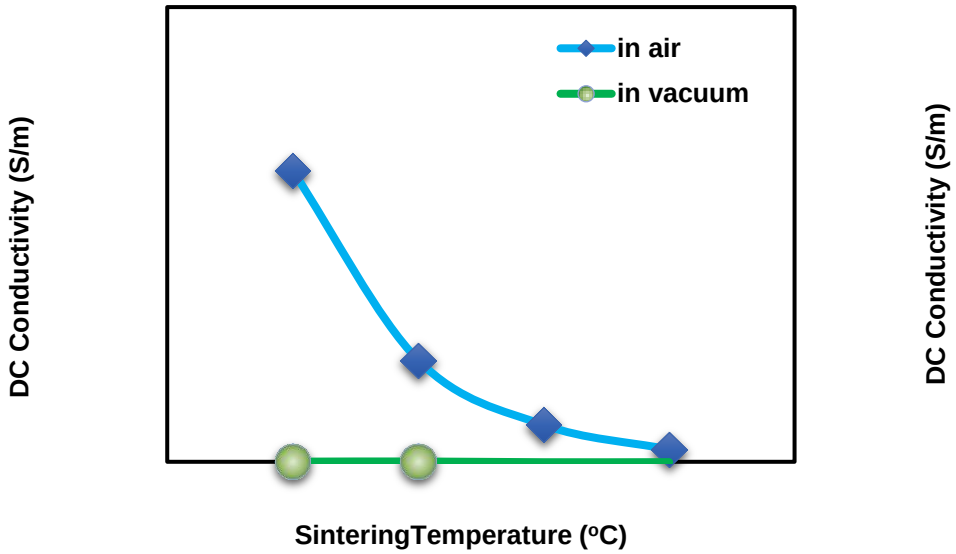

Figure 2. Variation of d.c conductivity values as a function of sintering temperature in air and in vacuum (dc conductivity in air uses left-axis, dc conductivity in vacuum use right-axis)

AC measurements were also done on the BDHA in air and vacuum ambient in the frequency range of 40 Hz – 100 kHz. AC conductivity values of the composites took placed between 5.90×10^{-7} S/m and 9.97×10^{-7} S/m at 10 kHz in air ambient. The values were decreased to the range of 5.88×10^{-7} S/m - 6.64×10^{-7} S/m at 10 kHz in vacuum ambient. Although there are some changes in ac conductivity depending on sintering temperatures but it is not observed remarkable/systematic behavior for ac conductivity depending on sintering temperature. This point needs to investigate in more detail such as ac conductivity variation as a function of frequency at elevated ambient temperatures in vacuum. The measurements are also underway.

4. Conclusions

This study shows the influence of varying sintering temperature on the DC and AC conductivity properties of the BDHA (prepared from extracted bovine dentine). It was investigated that sintering temperature of the bovine dentine were affected powerfully the dc conductivity of the samples of BHDA. From dc conductivity measurements it can be concluded that, although there is fluctuation in dc conductivity values of the samples in vacuum but in general it tends to decrease with increasing sintering temperature. It is not observed remarkable/systematic behavior for ac conductivity depending on sintering temperature.

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