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# Effects of sintering temperature on electrical properties of sheep enamel hydroxyapatite

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**Abstract.** Bioceramics, especially calcium phosphate based bioceramics, whose examples are hydroxyapatite, and calcium phosphate powders have been widely used in the biomedical engineering applications. Hydroxyapatite (HA) is one of the most promising biomaterials, which are derived from natural sources, chemical method, animal like dental enamel and corals. The influence of sintering temperature on the electrical properties (i.e. DC conductivity, AC conductivity) of samples of sintered sheep enamel (SSSE) was studied in air and in vacuum ambient at room temperature. The sheep enamel were sintered at varying temperatures between 1000°C and 1300°C. DC conductivity results revealed that while dc conductivity of the SSSE decreases with increasing the sintering temperature in air ambient the values increased with increasing the sintering temperature in vacuum ambient. AC conductivity measurements were performed in the frequency range of 40 Hz - $10^5$ Hz. The results showed that ac conductivity values decrease with increasing the sintering temperature.

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## 1. Introduction

With increased living standards, an increase in injuries due to bone deterioration has also been observed. Therefore, it is very important to heal these injuries as soon as possible. To resolve this problem, surgical operation usually performed. But after that, ıt can take a very long time for bone to recover its mechanical properties in the first place [1]. Enamel is the most highly mineralized skeletal tissue present in the body. Mature enamel is consisted of 95-97 % carbonated Hydroxyapatite (HA) and 1% organic material [2]. HA is the most attractive as replacement of the inorganic material of bones. There has been attracted attention in the last years for the investigation of the electrical properties of HA material. Valerio and coworkers [3, 4], showed the positive effects of higher conductivity on bone healing for HA. Rodrigues et al. have examined piezoelectric properties because one of the mechanisms for bone growth is electrochemical process due to the generation of piezoelectric dipoles [5]. Electrical characterization is the powerful technique for investigation for some process such as dehydration and dehydroxylation in HA [6] and microstructure [7]. To date, a few study were reported on electrical characterization of SSSE. Because of the importance of electrical characterization of these materials there is a need to study of HA based material. In this study dc and ac electrical properties of the SSSE 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 sheep teeth were obtained from local shop in Istanbul. The teeth were immersed in an antiseptic solution to avoid odour 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, calcined at 850 $^0$C for nearly 4 h to remove any proteins and prions. Enamel was clearly peeled off from dentine. The obtained enamel samples were ground by a ball grinder for 4 h (Planetary Ball Mill PM 200 Retsch, Haan, Germany). Sheep enamel powders were sieved. The enamel powders were obtained nearly 100-150 μm. The prepared powders were pressed between hardened steel dies under the pressure of 350 MPa. The green sheep enamel compacts were sintered for 4 h at different temperatures (1000, 1100, 1200, and 1300 $^0$C) (Nabertherm HT 16/17, Lilienthal, Germany). In order to prepare the pellets of SSSE for dc and ac electrical measurements circular-shaped silver electrodes on both surfaces of the SSSE were coated in high vacuum ambient ($<10^{-6}$ mbar) using thermal evaporation method. DC and ac conductivity measurements were performed in aluminum chamber in air and in vacuum ambient (~$2x10^{-2}$ mbar) in dark at room temperature. During the dc conductivity measurements sweep voltage between -1 and 1 volt was applied to the SSSE and current passing through to SSSE was measured using Keithley model 6517B programmable electrometer. AC conductivity measurements were performed between the frequencies 40 Hz - $1x10^5$ Hz using a Keithley model 3330 LCZ meter. All the measurement system was computerized.

## 3. Results and discussion

In order to determine effects of sintering temperature on dc and ac conductivity properties of the SSSE, electrical measurements were performed. Electrical conductivity measurements were done on the sheep enamel sintered at different temperatures between 1000 -1300 $^o$C in air and in vacuum ambient ($<10^{-2}$ mbar) in dark at room temperature. In order to calculate conductivity of the SSSE, current values were recorded between the applied voltages of -1 and 1 V. Current-Voltage (I-V) curves of the SSSE at indicated temperatures in air and in vacuum ambient were given in Figure 1. I-V curves of the SSSE showed ohmic behavior in air and in vacuum ambient. By inspecting the Fig. 1 hysteresis were observed in I-V curves except the SSSE at 1000$^o$C and 1100$^o$C in air ambient. The hysteresis may arises from deep traps as previously reported [7, 8]. This processes need longer time constant for charging and discharging process. Temperature, voltage sweeps speed and deep trap level may affects the hysteresis.

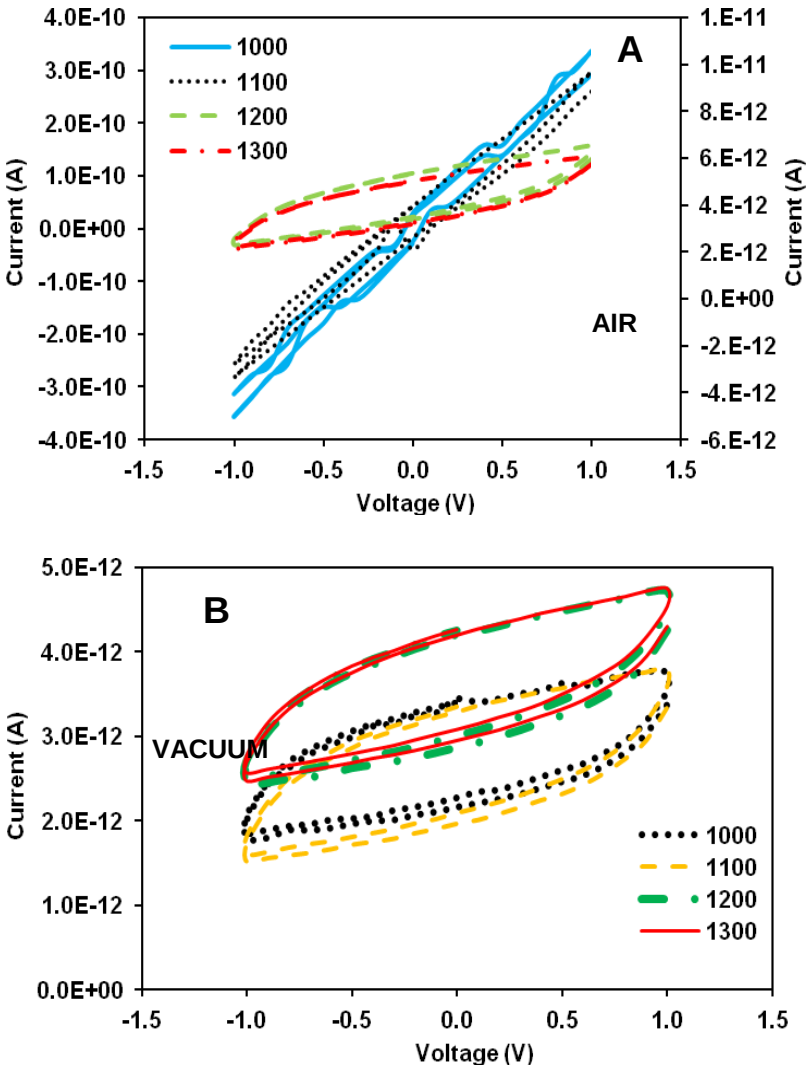


**Figure 1.** Current-Voltage (I-V) curves of the sheep enamel at indicated sintering temepratures obtained in dark (a) in air ambient (the SSSE at 1000 °C uses right-axis, and rest of the sheep enamel use left-axis) (b) in vacuum ambient.

DC conductivity values of the SSSE were calculated using the slopes of the I-V curves. The calculated d.c. conductivity values of the sheep enamel as a function of sintering temperatures in air and in vacuum ambient were presented in Figure 2. DC conductivity value was calculated as $9.77 \times 10^{-11}$ S/m for the composite sintered at 1000 $^{o}$C and increased to $3.01 \times 10^{-10}$ S/m for the sheep enamel sintered at 1300 $^{o}$C in vacuum ambient. By examining Fig. 2 effects of sintering temperature can be seen easily. First observation is that since the scale of vertical axis is logarithmic scale it can be concluded that sintering temperature affects strongly the dc conductivity. Another observation is the opposite behavior of dc conductivity in air and in vacuum ambient depending on sintering temperature. While dc conductivity decreases with increasing sintering temperature in air it increases with increasing sintering temperature in vacuum. The decrease in conductivity in air may arise from oxygen absorption. The real effect of the sintering temperature on dc conductivity of the sheep enamel can be obtained by eliminating another effects other than temperature such as humidity and oxygen. The conductivity of the sheep enamel increased with increasing sintering temperature in vacuum ambient. The behavior may be attributed to

decrease in resistance in two ways: i) increasing sintering temperature decrease the particle size and that results the decrease in grain boundary resistance, ii) probably change in molecular structure with increasing sintering temperature that results the decrease in bulk resistance.

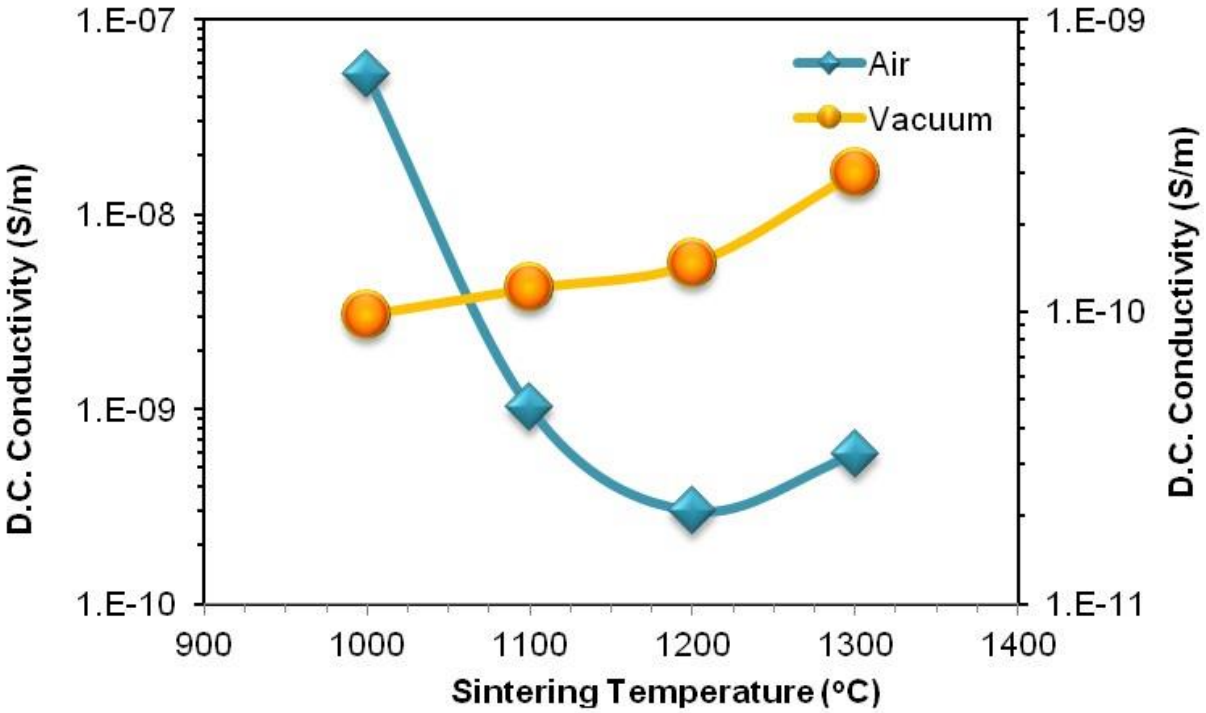


**Figure 2.** Variation of D.C. conductivity values as a function of sintering temperature in air and in vacuum.

AC conductivity measurements were also performed in air and vacuum ambient in the frequency range of 40 Hz - $10^5$ Hz. AC conductivity values were decreased from $1.21 \times 10^{-6}$ S/m to $3.69 \times 10^{-7}$ S/m as sintering temperature increased from 1000 ºC to 1300 ºC at 10 kHz in air ambient. The values were decreased from $6.06 \times 10^{-7}$ S/m to $2.46 \times 10^{-7}$ S/m as sintering temperature increased from 1000 ºC to 1300 ºC at 10 kHz in vacuum ambient. It can be concluded that ac conductivity values decreases with increasing sintering temperature.

## 4. Conclusions

The present study of the influence of sintering temperature on the electrical properties of the calcinated sheep enamel, prepared from derived sheep teeth, observed that sintering temperature of the sheep enamel affected strongly the dc and ac conductivity of the SSSE. From conductivity measurements it can be concluded that, basically, dc conductivity of the sheep enamel directly proportional and ac conductivity of the sheep enamel inversely proportional to sintering temperature.

## 5. Acknowledgements

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## 6. References

[1] F N Oktar 2006 *Mater. Lett.* **60** 2207-2210

[2] L C Palmer, C J Newcomb, S R Kaltz, E D Spoerke and S I Stupp 2008 *Chem. Tech.* **108(11)** 4754-4783

[3] A P M Schainberg, P Valerio, F N Oktar, M F Leite, A M Goes 2006 *20th European Conference on Biomaterials Proceedings* Nantes
[4] A P M Shainberg, P. Valerio, F N Oktar 2012 *Adv. Mater. Sci. Eng.* **2012** 1-10
[5] R Rodriguez, D Rangel, G Fonseca, M Gonzalez, S Vargas 2016 *Res. Phys.* **6** 925-32
[6] J P Gittings, C R Bowen, A C E Dent, I G Turner, F R Baxter, J B Chaudhuri *Acta. Biomater.* **5** 743-54
[7] C C Silva, M P F, Graça, M A Valente, A S B Sombra 2006 *J. Non. Cryst. Solids.* **352** 1490-94
[8] R Bayrak, F Dumludag, H T Akcay, I Degirmencioglu 2013 *Spectrochım Acta. A.* **105** 550-56
[9] P D Hooper, M I Newton, G McHale, M R Willis 1992 *Semicond. Sci. Technol.* **12** 455-59