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Nano Hidroksiapatit-Magnezyum Oksit Biyokompozitlerin Mikroyapısal ve Mekanik Özellikleri

Merve TURAN[1], Nermin DEMİRKOL[2,*]

[1] *Kocaeli Üniversitesi, Mühendislik Fakültesi, Biyomedikal Mühendisliği Bölümü, Kocaeli.*

[2]*Kocaeli Üniversitesi, Değirmendere Ali Özbay MYO, Seramik,Cam&Çinicilik, Kocaeli.*

e-posta: [1]merveeturan28@gmail.com, [2,]nermin.demirkol@kocaeli.edu.tr ORCID ID: [1]http://orcid.org/0000-0002-9531-594X, [2]http://orcid.org/0000-0001-9088-023X*

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Anahtar kelimeler
Hidroksiapatit, Nano,Biyokompozit, Magnezyum Oksit, Mekanik Özellikler, Mikroyapı

Öz

Hidroksiapatit (HA) ideal biyoaktif malzemedir. Ayrıca, HA iyi biyouyumluluk ve mükemmel osteoiletkenlik göstermektedir. Kimyasal bileşimi ve yapısı, doğal kemik mineralli matrise çok benzemektedir. Nano hidroksiapatitin tane boyutunun kemikteki apatit minerallerine çok benzemesi nedeniyle nano hidroksiapatit üzerindeki araştırmalar büyük önem kazanmıştır. Bu çalışmada, ticari nano hidroksiapatitin mekanik özelliklerini arttırmak için nano magnezyum oksit (nMgO) takviye malzemesi olarak kullanılmıştır. Ağ.% 80 nHA-ağ.% 20 nMgO (nMH1) ve ağ.% 70 nHA-ağ.% 30 nMgO (nMH2) toksik olmayan bağlayıcı içeren kompozitler pres kullanılarak şekillendirildi. Kompozitler 1000 **ve** 1100°C'de 2 saat süre ile sinterlendi, sonra nano kompozitlerin mikroyapıları ve mekanik özellikleri araştırıldı. Fiziksel ve mekanik özellikler yoğunluk, pişme küçülmesi, basma mukavemeti ve Vickers mikrosertlik (HV) ölçümleri ile tanımlandı. Yapısal karakterizasyon X-ışını kırınımı (XRD) ve taramalı elektron mikroskobu (SEM) ile ortaya kondu. Nano kompozitlerin mekanik özellikleri artan sıcaklık ve MgO ilavesi ile arttı.

Microstructural and Mechanical Properties of Nano Hydroxyapatite-Magnesium Oxide Biocomposites

Keywords
Hydroxyapatite, Nano, Biocomposite, Magnesium Oxide, Mechanical Properties, Microstructure

Abstract

Hydroxyapatite (HA) is an ideal bioactive material. Moreover, HA shows good biocompatibility and excellent osteoconductivity. Its chemical composition and structure are very similar to the mineralized matrix of natural bone. Due to the resemblance of grain sizes of nano hydroxyapatite to that of apatite minerals in bone, the researches of nano hydroxyapatite have become to gain great importance. In this study, in order to improve the mechanical properties of commercial nano hydroxyapatite (nHA), nano magnesium oxide (nMgO) was used as a reinforcement material. 80 wt% nHA-20 wt% MgO (nMH1) and 70 wt% nHA -30 wt% MgO (nMH2) nano composites including nontoxic binder were shaped by using press. The composites were sintered at 1000 and 1100°C for 2 hrs, then microstructures and mechanical properties of nano composites were investigated. The physical and mechanical properties were determined by measuring density, firing shrinkage, compression strength and Vickers microhardness (HV). Structural characterization was carried out with X-ray diffraction (XRD) and scanning electron microscopy (SEM). Mechanical properties of nano composites were increased with increasing temperature and increasing MgO addition.

14-16 Ekim 2019 tarihleri arasında düzenlenen X. Uluslararası Katılımlı Seramik Kongresi'nde sunulan bildirilerden seçilen çalışmadır.

1. Introduction

Hydroxyapatite [HA] is the main inorganic component of hard tissues in human body showing excellent biological properties and it is a vital material which has found many applications in the biomedical and clinical fields (Demirkol 2012 and Esmaeilkhanian et al. 2019). It has various exceptional properties like bioactivity, biocompatibility, osteoconductivity, slow biodegradability and non-toxicity (Kumar et al. 2011). Its stoichiometry is represented by the formula $Ca_{10}(PO_4)_6(OH)$. It is comprised of calcium and phosphorus present in the ratio (Ca/P) of 1.67 (Kantharia et al. 2014). Over the past few years, the impact of nanotechnology on implant field has begun to rise in a significant manner (Kumar et al. 2020). In recent years, nano-sized hydroxyapatite with appropriate stoichiometry, morphology and purity have stimulated great interest in scientific biomedical research (Shojai et al. 2013). Due to the low mechanical properties of HA, composite biomaterials became more popular. They can improve the mechanical and biological properties of ceramic scaffolds for bone tissue engineering application (Mondal et al. 2018). In order to improve the HA properties, there are many different methods. It can be doped with different metal ions such as Mg^{2+}, Zn^{2+}, Sr^{2+}, Fe^{3+},Ni^{2+} and Ce^{3+} (Heshmatpour et al. 2018).

Satpathy (2019) et al. studied to incorporate HA nanoparticles in trace doping amount in PVA-chitosan nanofiber matrix to fabricate PVA-chitosan composite nanofibers with improved performance for application as a bone tissue regeneration material.

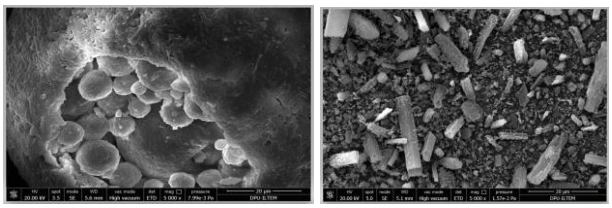
Sreekanth (2012) et.al. studied the development and characterization of MgO/hydroxyapatite composite containing on AZ31 magnesium alloy by plasma electrolytic oxidation coupled with electro phoretic deposition. The results showed that the MgO/HA composite coating significantly improved the corrosion resistance of AZ31 magnesium alloy.

Mg controls biological functions of human body and reduces risks of cardiovascular diseases (Demirkol et al. 2012).

The aim of this study is produce nano biocomposite containing nano hydroxyapatite-magnesium oxide with high mechanical properties at low sintering temperature.

2. Materials and Methods

The nano hydroxyapatite (nHA) and nano magnesium oxide (nMgO) powders used in this study were obtained from Nanografi Company in Ankara, Turkey.



(a) **(b)**

Figure 1. SEM micrograph of (a) nano hydroxyapatite (b) nano magnesium oxide powders (5000X)

Figure 1 exhibits the SEM micrograph of nano HA (Fig.1a) and nano MgO (Fig.1b). Mean particle sizes of used nHA and nMgO were 50 nm and 20 nm, respectively. 80 wt.% nHA -20 wt.% MgO (nMH1) and 70 wt.% nHA -30 wt.% MgO (nMH2) nano powder mixtures including nontoxic binder (20 wt.% PVA) were prepared by mechanical alloying. Polyvinyl alcohol (PVA) is a non-toxic synthetic polymer to humans with good biological affinity (Ni et al. 2019). Firstly, composites prepared without using PVA. They showed no density gain after sintering and dispersed. For this reason, 20 wt.% PVA was used for both composites. Pressed green samples were sintered at 1000 and 1100°C for 2 hrs. Density, compression strength and Vickers Microhardness values were measured. SEM and EDS analysis were done by using Scanning Electron Microscopy (Nova NanoSEM 650). The crystalline structure of the samples was determined with X-ray diffraction analysis on a PANalytical EMPYREAN X-ray diffractomer. The compression tests were done with an universal testing machine (Shimadzu) at the crosshead speed of 3 mm/min.

Microhardness values were determined under 200 g. Load for 15 s.

3. Results

Table 1 shows the density and mechanical properties of both composites sintered at 1000 and 1100°C.

Table 1. Density Compression Strength and Vickers Microhardness results of nMH1 and nMH2 samples sintered at 1000 and 1100°C.

Temperature (°C)	Composite Code	Density (g/cm³)	Compression Strength (MPa)	Vickers Microhardness (HV)
1000	nMH1	2,94	105	210
	nMH2	3,05	121	287
1100	nMH1	3,09	124	347
	nMH2	3,16	135	438

All properties increased with increasing sintering temperature for both composites. When the amount of nano MgO increased from 20% to 30%, the density and mechanical properties increased. Demirkol (2016) studied the mechanical and bioactivity properties of commercial synthetic hydroxyapatite-MgO composites. In her study, used particle sizes of MgO and HA were 65 µm and 6.5 µm, respectively. According to her results, the best density, compression strength and Vickers Microhardness results were obtained with commercial synthetic hydroxyapatite (CSHA)-5 wt.% MgO composite sintered at 1300°C as 2,90 g/cm^3, 85 MPa and 370 HV, respectively.

In this study, higher density and mechanical properties were obtained by using nano HA and nano MgO at lower sintering temperature (1100 °C).

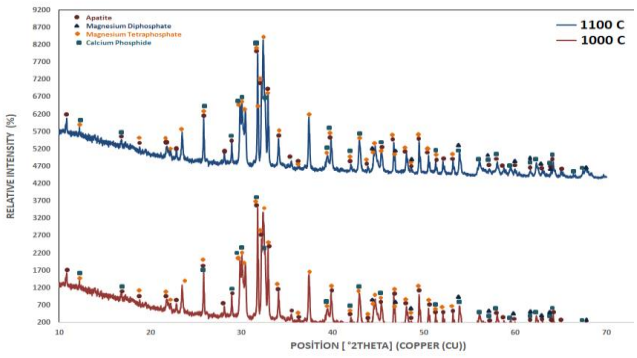


Figure 2. X-ray diffraction diagrams of 70 wt.% nHA-30 wt.% nMgO (nMH2) nano composites sintered at 1000 and 1100 °C.

As seen in Figure 2, apatite, magnesium diphosphate, magnesium tetraphosphate calcium phosphide phases were obtained for nMH2 composite at 1000 and 1100°C. Peak intensity of phases increased with increasing sintering temperature. The increase in the intensity of peak was due to the interaction of nHA with the nano MgO (Kumar et al. 2011)

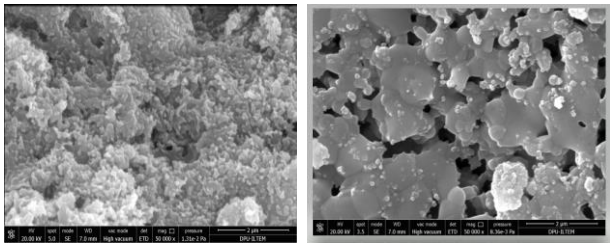


Figure 3. SEM micrograph of 70 wt.% nHA-30 wt.% nMgO (nMH2) nano composite sintered at (a) 1000 °C (b) 1100 °C.

Figure 3 exhibits the SEM micrographs of nMH2 composite sintered at 1100°C. SEM observation was performed at Dumlupinar University. The sample is coated with gold and placed in Nova NanoSEM 650. The grain growth occured with increasing sintering temperature in Figure 3(b). The incorporation of nHA particles with nano MgO caused a slight reduction in the porosity due to the interaction powders with incresing sintering temperature. It has also been demonstrated at Abidi (2014) et al's and Kumar (2011) et al's studies.

As seen in Figure 4 and 5, according to the SEM and EDS results of nano composite material (nMH2) sintered at 1100°C, selected area 1 includes more MgO than the other parts. The other parts of the micrograph contain more CaO than the selected area 1.

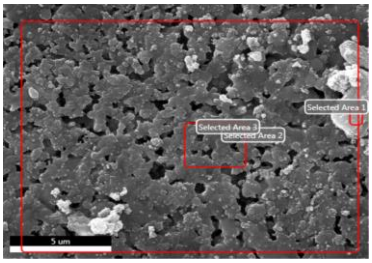
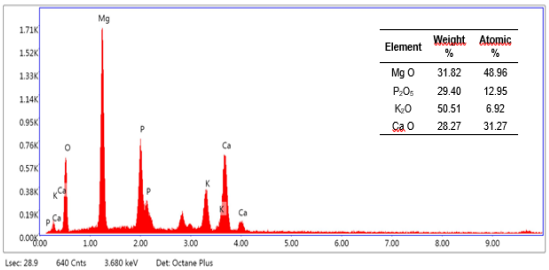
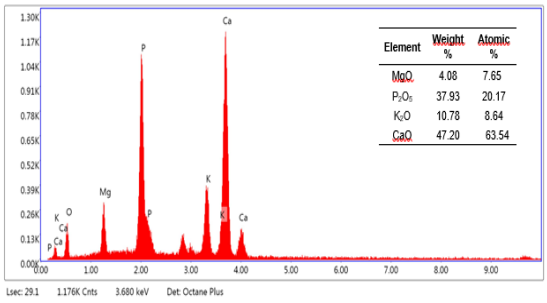


Figure 4. SEM micrograph and EDS marking of 70 wt.% nHA-30 wt.% nMgO (nMH2) nano composite sintered at 1100 °C.

Selected Area 1 - EDS



Selected Area 2 - EDS



Selected Area 3 – EDS

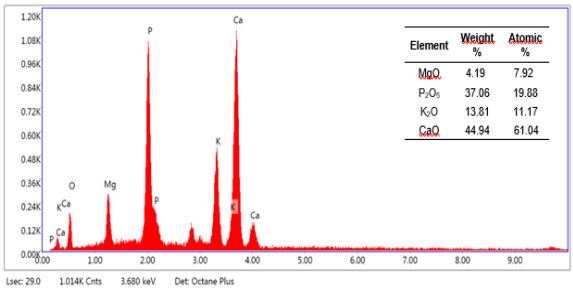


Figure 4. exhibits SEM micrograph and EDS marking of 70 wt. .% nHA-30 wt.% nMgO (nMH2) nano composite sintered at 1100 °C.

4. Discussion and Conclusion

Apatite is the major inorganic component in bone, and the crystals are at nanometer scale and embedded in the collagen matrix (Huang et al. 2007).

In this study, the microstructural and mechanical properties of nano hydroxyapatite-magnesium oxide biocomposites were examined to determine optimum sintering temperature and properties for nano HA-MgO biocomposite.

The following conclusions were obtained.

1- Density, compression strength and Vickers microhardness values increased with increasing sintering temperature for 80 wt.% nHA-20 wt.% MgO (nMH1) and 70 wt.% nHA-30 wt.% MgO (nMH2) nano biocomposites.
2- Density and mechanical properties increased with increasing nano MgO addition to composite due to good compaction.
3- For both composites, 20 wt.% PVA addition is necessary for density gain. PVA is very useful to incorporate to nano HA to nano MgO (Satpathy et al. 2019)
4- The highest density, compression strength and Vickers microhardness values were obtained with nMH2 nano composite sintered at 1100°C as 3,16 g/cm^3, 135 MPa and 438 HV, respectively.
5- The use of nano-sized hydroxyapatite and nano MgO contributes more to the density and mechanical properties than the use of micron-sized materials. When comparing composites prepared with micron sized powders and those prepared with nano sized powders higher temperatures are needed for micron sized powders (Demirkol et al. 2012 and Verma et al. 2019).
6- Bioactivity and cell culture studies are going on.

Acknowledgements

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