

Simulation of a solid chemical structure of dental material using nanomaterials

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Abstract

Powders of alloys of hydroxyapatite (Ag-HAP), (Cu-HAP), and (Cu/Ag-HAP) were created by employing hydroxyapatite as a starting material and co-substituting it with silver and/or copper. X-ray diffraction (XRD) was used to characterize the alloys after they were produced using the ultrasonic irradiation method. In this study, the ultrasonic irradiation approach was used to effectively synthesis HAP, Ag - HAP, and Cu/Ag-HAP powders. This process is straightforward and inexpensive. XRD testing of silver and/or copper doped HAP samples confirmed the presence of nano-sized particles with a consistent chemical makeup. The X-ray diffraction (XRD) examination showed that the hydroxyapatite (HAP) annealed at 400 °C formed a hexagonal structure, with a little shift in the primary peak location toward lower angles due to the addition of silver. Depending on the Ca/P ratio, this research shows that silver and copper ions may be integrated into the hydroxyapatite crystal during mineralization, providing us with functional groups of calcium-phosphate apatite. Crystallinity, lattice properties, and crystal size all varied as a result of the silver and copper content swap, as evidenced by the XRD technique.

Keywords: nanomaterials, hydroxyapatite, silver, copper, dental.

1. Introduction

1.1 Hydroxyapatite

Materials science and biology work together to create biomaterials, with the goal of facilitating a healthy interaction between the human body and the novel material. Apatite is one of the several calcium orthophosphate crystal structures. The Ca/P mole ratio in pure HA is 1.67, since the mineral comprises 39.68 percent calcium and 18 percent phosphorus by weight (Ramesh et al., 2008). Meanwhile, hydroxyapatite, with a formula of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, is being utilized in medical implants to restore sick or injured bone and teeth [Best et al;2008]. Nanohydroxyapatite in natural bone tissue: physicochemical properties Humans have used biological materials for a very long period. Research into nano apatite for use as a bone healing material is a hot topic in the field of biological materials at the moment [Dou et al.;2018]. Infection after surgery or immune system rejection of the implant is a major concern for anybody getting an implant. Modern implants are coated in extremely biologically

compatible materials with amazing biological qualities to prevent rejection by the human body [Motskin et al;2009, Asuka et al;2012, Binyamin et al;2006, Costescu et al;2010 Ciobanu et al;2010]. Coating different prostheses with HAp has been demonstrated to increase the speed of healing and the degree to which the body accepts the device. Because of its ability to promote bone development and facilitate early attachment between bone and implant surfaces, HAp coatings have found widespread use as bioactive layers on implants. HAp has several chemical similarities with bone, such as an overall similarity in composition and the presence of ionic substitutions (Nathaniel. et al., 2011, Porter et al., 2003).

1.2 Ion doped hydroxyapatite.

The wide range of cationic and anionic substitutions allowed in apatite is a result of the structure's exceptional stability and flexibility. Ca ions in the structure of HA have been partly replaced by several other ions, which have been rearranged. The crystal lattice is often stabilized by ions with a lower ionic radius than Ca^{2+} [Boanini et al; 2010]. Microhardness in HA is improved by the addition of Lithium ions to commercial HA samples. Tricalcium phosphate, which contains zinc, is both biocompatible and bioactive, and it served as an efficient zinc transporter. Zn TCP and HA P formed a composite ceramic that promoted the growth and development of osteoblastic cells in vitro. In addition, this biomaterial shows great promise as a pharmacological agent for enhancing bone production [Atsuo et al; 2002]. Researchers have used XRD, FTIR, chemical analysis, and laser diffraction to learn more about chromium-loaded calcium-hydroxyapatite nanoparticles created by using the exchange capabilities of apatite. A reduction in specific surface area was observed along with an increase in Cr^{3+} loading, suggesting that the synthesized apatite is calcium deficient. Calcium ions are replaced by chromium ions in the CaHAp crystal as the Cr^{3+} atomic ratio rises and the Ca^{2+} atomic ratio falls linearly as more Cr^{3+} is added to the solution. There is no crystalline chromium peak in the X-ray diffraction (XRD) patterns of $\text{Cr}(x)/\text{CaHAp}$; instead, they resemble those of pure calcium-hydroxyapatite. Using carboxy methylated poly (2-hydroxyethyl methacrylate) (pHEMA) as a biomaterial for calcification, we studied the effects of Co^{2+} , Cr^{3+} , and Ni^{2+} on hydroxyapatite (HA) development in vitro. We showed that Cr^{2+} , Cr^{3+} , and Ni^{2+} all lowered the Ca/P ratio by 1.12, 1.05, and 1.08-fold, respectively, while also decreasing the amount of mineral produced on the polymer's surface.

The physical, chemical, and biological characteristics of HAp have been demonstrated to change when Eu, Ag, Cu, Zn, Ce, and Y ions are added into the structure. The replacement of Ca^{2+} with other metal ions, including as Ag^+ , Cu^{2+} , Ce^{3+} , and Zn^{2+} , has been identified as one of the most successful approaches to enhance the characteristics of Hap in recent research. The present research lends credence to the idea that silver ions or metallic silver, as well as silver nanoparticles, may be used in medicine for uses such as burn therapy, dental materials, coating stainless steel materials, textile fabrics, water treatment, sunscreen lotions, etc. Due to the restrictions placed on the use of antibiotics, physicians have gone back to silver wound dressings containing variable quantities of silver [Kumar and Munstedt;2005, Damm and Munstedt,2008] because of its low toxicity to human cells, high thermal stability, and low volatility. Catheters, infusion sets, and medical fabrics are just some of the medical devices that may benefit from the antibacterial properties of materials based on Ag doped nanocrystalline HAp powder. Because the Ca^{2+} ions may be readily swapped by Ag^+ ions in the matrices, providing a material with good biocompatibility and antibacterial characteristics [Chen et al; 2006, Rai et al;2009], W. Chen et al. demonstrated in 2006 that the HAp is the appropriate biomaterial

to embed silver ions. Recent research by M. Daz et al., 2009 found that silver hydroxyapatite nanocomposites generated by a colloidal chemical approach and a subsequent reduction procedure in H₂/Ar environment at 350 °C had a significant antibacterial activity against *S. aureus*, *Pneumococcus*, and *E. coli*. It has been shown in recent research [Ciobanu et al;2011, Ciobanu et al;2012] that Ag:HAp nanoparticles produced by the co-precipitation technique at 100 °C have considerable potential as antibacterial agents against gram-positive and gram-negative bacteria. By combining these substrates, we are able to create a versatile material with exceptional properties that has wide-ranging potential uses. The Hap/iron oxide composite has been shown to be an effective adsorbent for the elimination of several metal ions (Cd, Cu, Ni, Fe, Mn, Co, and U) in many investigations.

Hap has been shown to be an efficient sorbent in the adsorption processes of metal ions, particularly heavy metals and radioactive ions, in a variety of investigations. In addition, both inorganic and organic substances may be adsorbed [Biedrzycka et al., 2021; Ibrahim et al., 2020; Pai et al., 2020]. Research into the ways in which hydroxyapatite may be altered is growing in popularity in recent years. The particles with magnetic characteristics are iron oxides, and they are deserving of particular study because of their unique qualities. There are several varieties of these oxides, and they are used in many different industries. However, the more frequent magnetite (Fe₃O₄) is the focus of most research rather than the less common maghemite (Fe₂O₃). Magnetite has a metallic luster since it is a mineral. It contains Fe (II) and Fe (III) in a ratio of 1:2 throughout its structure. The magnetic properties and electrical conductivity of this mixed oxide are exceptional. Fe₃O₄ may be found in rocks and can be altered into different structures. Better stability, correct diffusion, and protection against aggregation are all benefits of an extra layer on top of the iron oxide particles. Furthermore, the external magnet allows for simple separation of the magnetic composite from the solution. Drugs, DNA, antibiotics, vitamins, hormones, proteins, and enzymes are only some of the items that might be delivered, according to studies. The hyperthermia method, which kills cancer cells, could also benefit from using the composite. The use of a Hap/iron oxide composite as an implant or rebuilding agent during bone repair processes is a potential prospect. The material's catalytic potential was also studied [Biedrzycka et al;2022].

Silver (Ag⁺), copper (Cu²⁺), zinc (Zn²⁺), and titanium (Ti⁴⁺) are only some of the inorganic antibacterial agents that have been included into biomaterials in recent years to prevent microbial infection [Chen et al;2006, Heidenau et al;2005, Chung et al;2006]. When compared to the other ions, silver is thought to have better chemical and physical qualities such biocompatibility, high thermal stability, and non-toxicity to human cells at low concentration. Higher levels of Ag in the material have been shown to have a superior antibacterial effect, but at the cost of increased cytotoxicity [Gopi et al;2014]. To mitigate these side effects while yet benefiting from Ag's antibacterial characteristics, an additional substance must be included. Accelerating bone formation include minerals such as strontium (Sr), magnesium (Mg), zinc (Zn), and trace metal elements [Gopi et al.;2014]. Magnesium (Mg) is well-known as a crucial trace element for healthy teeth and bones.

2. Materials and methods

2.1. Materials

All of the Ca, Ag, Cu, and P employed in this analysis came from analytical grade calcium nitrate tetrahydrate (Ca (NO₃)₂·4H₂O), silver nitrate (AgNO₃), copper nitrate (Cu(NO₃)₂), and diammonium

hydrogen phosphate ((NH₄)₂HPO₄, Merck). All of the chemicals were of an analytical grade and were put to use straight from the bottle.

2.2. Synthesis of HAP, Ag-HAP, Cu-HAP and Ag/Cu-HAP

Ultrasonic irradiation was used to produce HAP, Ca(NO₃)₂·4H₂O, (NH₄)₂HPO₄, AgNO₃, and Cu(NO₃)₂, each at varying concentrations. Phosphorus was used to create four different powders.

series 1

(HAP): using Ca (NO₃)₂·4H₂O and (NH₄)₂HPO₄,

series 2

(Ag-HAP): using Ca (NO₃)₂·4H₂O, AgNO₃ and (NH₄)₂HPO₄

series 3

(Cu-HAP): using Ca (NO₃)₂·4H₂O, Cu (NO₃)₂ and (NH₄)₂HPO₄

and series 4

Ag/Cu-HAP): using Ca (NO₃)₂·4H₂O, AgNO₃, Cu (NO₃)₂ and (NH₄)₂HPO₄.

Series 1

HAP is made by dissolving Ca (NO₃)₂·4H₂O to a concentration of 0.5 M in double-distilled water (solution 1), then adjusting the pH to 10 using sodium hydroxide solution, and then dissolving (NH₄)₂HPO₄ to a concentration of 0.3 M in double-distilled water (solution 2). Sodium hydroxide solution was used to keep the pH at 10 while solution 2 was added to solution 1 drop by drop over the course of two hours while the mixture was agitated. The obtained precipitate was held in an ultrasonicator (Microplus EN60 ultrasonicator) for 1 hour at a frequency of 28 kHz to guarantee a homogenous mixture before being dried in a hot air oven at 110 C and afterwards calcined for 1 hour at 400oC.

Series 2

Ag-HAP is made by combining Ca (NO₃)₂·4H₂O and AgNO₃ to a concentration of 0.5 M in double-distilled water, mixing well, and then adjusting the pH to 10 using a sodium hydroxide solution. Series 2 Ag-HAP is then made by dissolving 0.3 M (NH₄)₂HPO₄ in double-distilled water. Sodium hydroxide solution was used to keep the pH at 10 while solution 2 was added to solution 1 drop by drop over the course of two hours while the mixture was agitated. To establish a uniform mixture, we dried the obtained precipitate in a hot air oven at 110 degrees Celsius, followed by one hour of calcination at 400 degrees Celsius (Microplus EN60 ultrasonicator).

Series 3

After adjusting the pH of 0.5 M Ca (NO₃)₂·4H₂O and 0.5 M Cu(NO₃)₂ dissolved in double-distilled water (solution 1) to 10 using a sodium hydroxide solution, 0.3 M (NH₄)₂HPO₄ dissolved in double-distilled water (solution 2) was obtained. Sodium hydroxide solution was used to keep the pH at 10 while solution 2 was added to solution 1 drop by drop over the course of two hours while the mixture

was agitated. The obtained precipitate was held in an ultrasonicator (Microplus EN60 ultrasonicator) for 1 hour at a frequency of 28 kHz to guarantee a homogenous mixture before being dried in a hot air oven at 110 C and afterwards calcined for 1 hour at 400oC.

Series 4

Cu/Ag-HAP is made in a manner similar to that of HAP precipitation. Ca(NO3)2.4H2O and AgNO3 are both dissolved in water to form a 0.5 M solution, but the sole difference is the addition of Cu (NO3)2. The second method is similar to the first.

2.3. Sample characterization

The crystalline phase composition of the as-synthesized materials was determined using X-ray Diffraction with Cu Ka radiation produced at 35 kV and 25 mA. In this investigation, we used the Debye-Scherrer formula ,

$$D = 0.9 \lambda / \beta \cos \theta$$

to calculate an estimate for the average particle size based on the peak at degrees.

Where, 'λ' is X-ray wavelength (0.1541nm), FWHM (full width at half maximum), 'θ' is diffraction angle and (D) particle diameter size are all variables.

3. Results and discussion

3.1. The XRD patterns of HAP

In Fig. 1 we see HAP's XRD patterns. It was determined that HAP was the primary phase during this whole occurrence. Sharp and strong peaks at 2 values of 26.2, 32.3, and 33.2 deg suggest the presence of the (002), (112), and (300) reflections of metallic calcium, confirming the peaks in Fig. 1 and Table 1 as HAP according to JCPDS no. 09-0432. The powders as synthesized included no additional undesirable peaks [EL Boujaady et al;2016]. Standard charts provide a close approximation of XRD diffraction patterns. The creation of hydroxyapatite nanoparticles is supported by the fact that their average size is less than 3 nm, as seen in table 1.

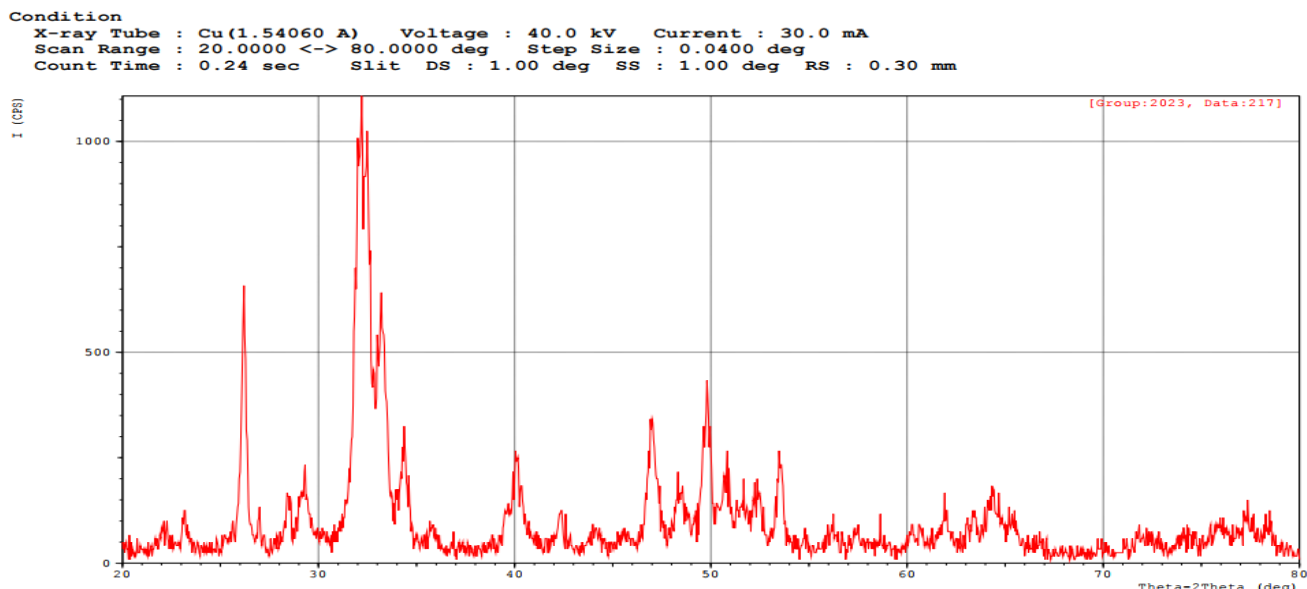


Fig. 1: X-ray diffraction patterns of nano hydroxyapatite (HAP).

Table 1: The grain size of hydroxyapatite nanoparticles.

2 θ of the intense peak (deg)	Miller indices (h k l)	FWHM of intense peak (β) radians	Size of the particle (D) nm
26.2	002 Ca	0.518	3.12
32.3	112 Ca	0.937	1.83
33.2	300 Ca	0.800	2.16
Average size of particles			2.37

3.2. The XRD patterns Ag-HAP

Since XRD is often used to determine a material's chemical composition and crystal structure, it may also be used to identify silver nanoparticles in samples. Fig. 2 shows the XRD patterns of the samples after the HAP and Ag-HAP layers were deposited on the anodized surface.

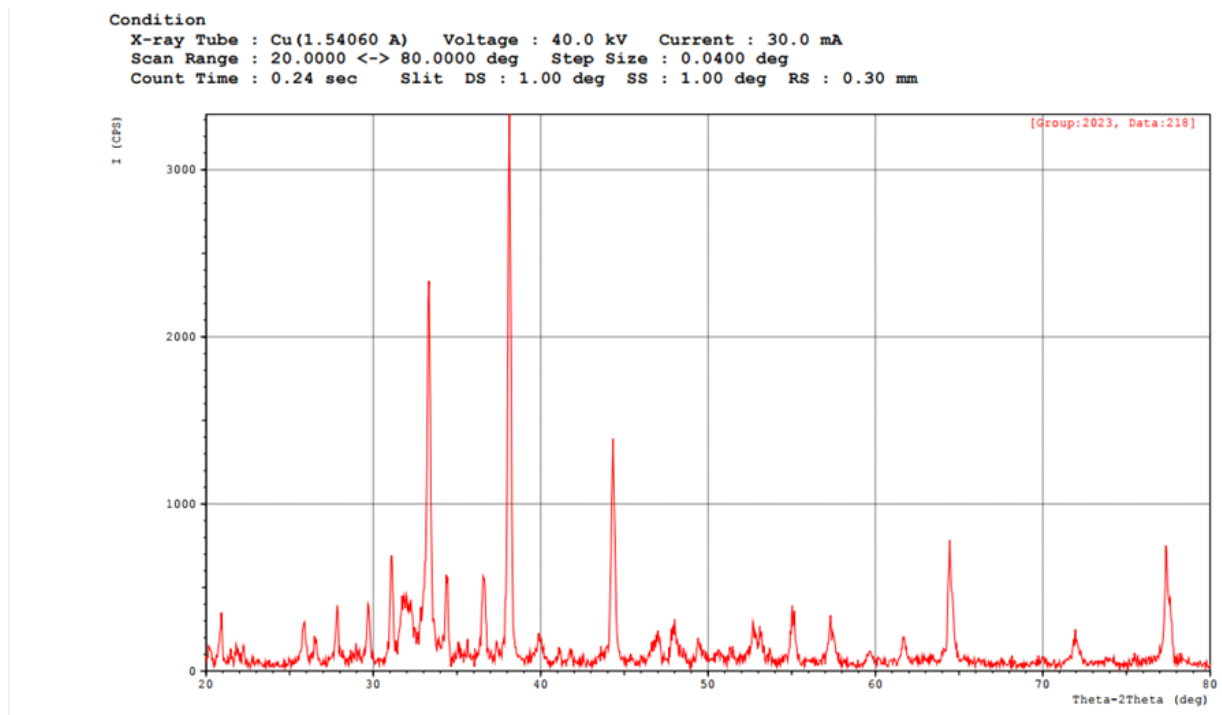


Fig. 2: X-ray diffraction patterns of silver nano hydroxyapatite (HAP)

Using JCPDS 09-0432 and JCPDS 21-1272, the peaks were determined to be HAP and a new phase, respectively [Mirzaee et al., 2014]. The (111) and (200) reflections of silver are highly reflective, having peaks at 2 values of 38.1 and 44.3 deg, respectively. A peak at 2 values of 33.3 deg was also

seen in the XRD patterns of Ag-HAP, making them similar to those of HAP. As can be seen in Table 2, the samples had a particle diameter of less than 9 nm.

Table 2: The grain size of silver- hydroxyapatite nanoparticles.

2θ of the intense peak (deg)	Miller indices (h k l)	FWHM of intense peak (β) radians	Size of the particle (D) nm
33.3	300 Ca	0.213	8.15
38.1	111 Ag	0.201	9.18
44.3	200 Ag	0.229	8.84
Average size of particles			8.72

3.3. The XRD patterns Cu-HAP

Because of its potential technical uses in a wide range of sectors including catalysis, lubricants, electronics, etc. [Park et al;2003], copper nanoparticles have previously been investigated extensively. In the study of nanomaterials, X-ray diffraction is both a simple and crucial characterisation technique. In this work, ultrasonic technology was used to effectively create copper nano powder, an important nanomaterial, and X-ray diffraction was used to study its structural characterizations. According to the data, nano powder with a mean particle size of less than 4 nm does exist (Table 3).

The sole notable peak in Figure 3 occurs at a 2θ value of 49.5 deg, which corresponds to the (200) plane of copper. However, when comparing the JCPDS, copper file No. 04-0836 and ASTM 03-1005- [Nasirian 2012], it was found that peaks at various 2θ values correspond to other planes of copper were less significant.

Condition
X-ray Tube : Cu(1.54060 Å) Voltage : 40.0 kV Current : 30.0 mA
Scan Range : 20.0000 <-> 80.0000 deg Step Size : 0.0400 deg
Count Time : 0.24 sec Slit DS : 1.00 deg SS : 1.00 deg RS : 0.30 mm

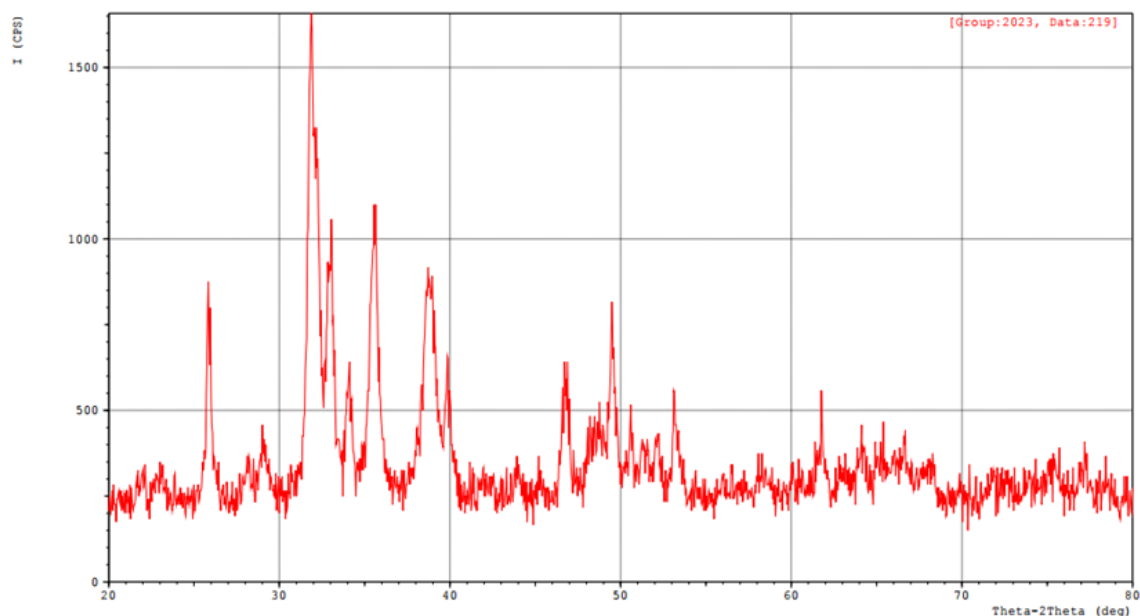


Fig. 3: X-ray diffraction patterns of Copper nano hydroxyapatite (HAP)

Table 3: The grain size of copper- hydroxyapatite nanoparticles.

2θ of the intense peak (deg)	Miller indices (h k l)	FWHM of intense peak (β) radians	Size of the particle (D) nm
32.0	112 Ca	0.753	2.27
33.0	300 Ca	0.595	2.91
35.6	301 Ca	0.590	3.02
49.5	200 Cu	0.407	5.49
Average size of particles			3.42

3.4. The XRD patterns Cu/Ag-HAP

The XRD pattern of Cu co-substitution in the Ag-HAP was shown in Fig. 4. Peaks similar to those observed for Ag-HAP are seen in the Cu co-substituted Ag-HAP, as shown by the data. On the other hand, Table 4 shows that the Cu co- substituted Ag-HAP sample exhibits substantial peaks at 2 value of 44.3 and 77.4 deg, corresponding to the (111) and (220) planes of copper. The co-substitution of Cu ions in Ag-HAP may have triggered this peak shift, demonstrating the ionic action of Cu on Ag-HAP. Because of this, we may conclude from the XRD pattern that the Cu has been successfully absorbed

into the nanoparticle-sized Ag-HAP complex. Meanwhile, HAP nanoparticles showed excellent stability and crystallinity. The average particle size was 11.42 nm, as shown in table 4.

Condition

X-ray Tube : Cu(1.54060 Å) Voltage : 40.0 kV Current : 30.0 mA
 Scan Range : 20.0000 <-> 80.0000 deg Step Size : 0.0400 deg
 Count Time : 0.24 sec Slit DS : 1.00 deg SS : 1.00 deg RS : 0.30 mm

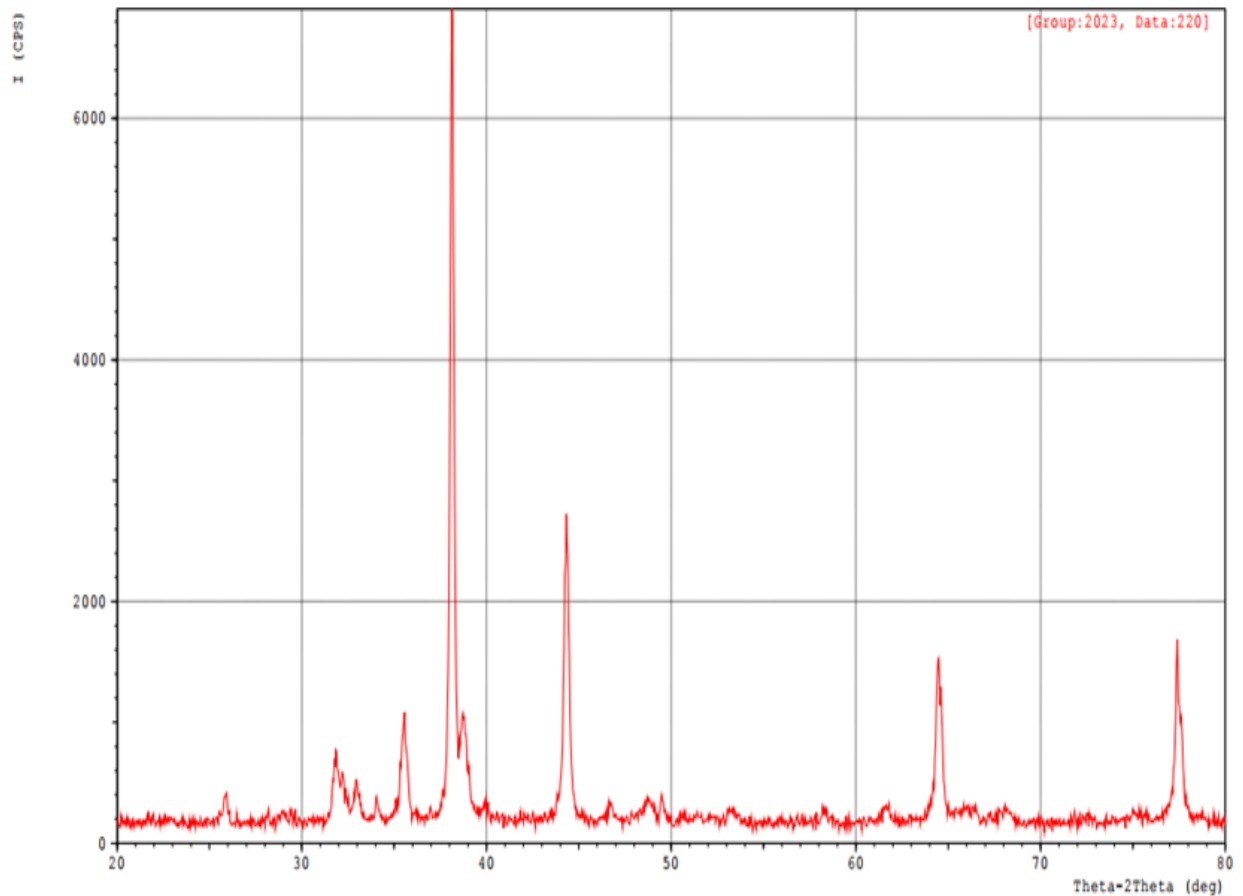


Fig. 4: X-ray diffraction patterns of Cu/Ag- nano hydroxyapatite (HAP)

Table 4: The grain size of Cu/Ag- hydroxyapatite nanoparticles.

2θ of the intense peak (deg)	Miller indices (h k l)	FWHM of intense peak (β) radians	Size of the particle (D) nm
25.9	002 Ca	0.230	7.01
32.2	112 Ca	0.160	10.74
33.0	300 Ca	0.242	7.14
38.1	111 Ag	0.217	8.48
44.3	111 Cu	0.278	7.29

77.4	220 Cu	0.237	27.88
Average size of particles			11.42

4. Conclusion

In conclusion, the ultrasonic irradiation approach described in this research was used to effectively synthesis HAP, Ag -HAP, and Cu/Ag-HAP powders. This process is straightforward and inexpensive. XRD testing of silver and/or copper doped HAp samples confirmed the presence of nano-sized particles with a consistent chemical makeup. Adding silver to hydroxyapatite (HAp) and annealing it at 400 degrees Celsius results in a little change in the primary peak location toward lower angles, as validated by XRD analysis. This research shows that the functional groups of calcium-phosphate apatite and its replacement occurs during production based on the alteration of Ca/P ratio, with silver and copper ions being integrated into the hydroxyapatite crystal during mineralization. Crystallinity, lattice properties, and crystal size all varied as a result of the silver and copper content swap, as shown by the XRD technique. Therefore, the synthesized materials are extremely promising and may aid in the creation of more efficient orthopedic and dental implants by providing a better knowledge of cell/ biomaterial interactions.

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محاكاة التركيب الكيميائي الصلب للمادة السنية باستخدام المواد النانوية

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الملخص

يهدف العمل الحالي إلى تخليق التركيب الكيميائي الصلب لمواد سنية باستخدام هيدروكسيباتيت كمادة أولية ومشاركة بديلة من الفضة و / أو النحاس لإنتاج سبائك هيدروكسيباتيت (Ag-HAP)، (Cu-HAP) ومساحيق (Cu / Ag-HAP). لهذا الغرض، تم تحضير السبائك بتقنية الشعاع بالموجات فوق الصوتية وتميزت بانحراف الأشعة السينية (XRD). تم تصنيع مساحيق HAP و Ag-HAP و Cu / Ag-HAP بنجاح عن طريق طريقة الشعاع بالموجات فوق الصوتية وهي منهجية بسيطة ومنخفضة التكلفة للحصول على مسحوق الجسيمات النانوية الموصوفة في هذه الورقة. أظهر تحليل XRD أن جزيئات الفضة و / أو النحاس مخدر عينات HAP ذات حجم نانو ومتجانسة في التركيب. أكد تحليل XRD تشكيل بنية

سداسية من هيدروكسيباتيت (HAp) صلب عند 400 درجة مئوية مع تحول طفيف في موضع الذروة الرئيسي نحو الزوايا السفلية مع إضافة الفضة. تشير هذه الدراسة إلى أنه يمكن دمج أيونات الفضة والنحاس في بلورة هيدروكسيباتيت أثناء التمدن، مما يعطينا مجموعات وظيفية من أباتيت الكالسيوم والفوسفات ويدخل استبدالها أثناء التحضير اعتمادًا على تباين نسبة الكالسيوم / الفوسفور. توضح طريقة XRD أنه كانت هناك تغييرات في التبلور ومعلومات الشبكة وحجم الكريستال بسبب استبدال محتويات الفضة والنحاس.