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تحضير وتوصيف مسحوق الهيدروكسيباتيت وفحص مركبات الهيدروكسيباتيت والزركونيا والألومينا

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المستخلص

تم تحضير مسحوق الهيدروكسيباتيت $Ca_{10}(PO_4)_6(OH)_2$ محلياً من عظام البقر واجري لها توصيف. كما تم تحضير المركبات من الهيدروكسيباتيت–الألومينا (0,5,10,15,20,25%wt) وأعطي لها الرمز (HA)، وهيدروكسيباتيت–زركونيا (0,5,10,15,20,25%wt) وتم اختيار اسم لها (HZ)، وهيدروكسيباتيت–(الألومينا – زركونيا) (0,5,10,15,20,25%wt) وتدعى بالرمز (HAZ) وللحالة النهائية تضمنت الألومينا بنسبة (80%) والزركونيا بنسبة (20%). استخدم البولي إيثيلين جلايكول كمادة رابطة. بعد ذلك تم المعالجة الحرارية بدرجة حرارية 1350°C لمدة ثلاث ساعات. اجريت فحوص حيود الأشعة السينية (XRD)، وفحوصات تشمل الخصائص الميكانيكية والفيزيائية منها التقلص القطري ، صلادة فيكرز ، متانة الانضغاط ، المسامية ونسبة امتصاص الماء. أظهرت اختبارات حيود الأشعة السينية أن طور الهيدروكسيباتيت قد تم تثبيتها للعينات المحضرة قبل وبعد المعالجة الحرارية باستخدام الصيغة الكيميائية $Ca_{10}(PO_4)_6(OH)_2$، أي دون تجزئتها إلى مكونات منفصلة.

الكلمات المفتاحية: الهيدروكسي ابتايت، الومينا، زركونيا ، مركب، تطبيقات طبية

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Preparation and Characterization of Hydroxyapatite Powder and Examination of the Composites of Hydroxyapatite, Zirconia, and Alumina

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Abstract

Hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ powder has been locally extracted and prepared from bovine bones. Compounds were also prepared from hydroxyapatite-alumina (0,5,10,15,20,25%wt), labeled (HA), and hydroxyapatite-zirconia (0,5,10,15,20,25%wt) and were chosen. Its name is (HZ), and hydroxyapatite-(alumina-zirconia) (0,5,10,15,20,25%wt) is called by the symbol (HAZ) and the final state includes alumina (80%) and zirconia (20%). Use polyethylene glycol as a binder. After that, heat treatment was carried out at 1350ºC for three hours. X-ray diffraction (XRD) tests, and tests that include mechanical and physical properties, including radial shrinkage, Vickers hardness, compressive strength, apparent density, porosity, and water absorption rate were conducted. X-ray diffraction tests showed that the hydroxyapatite phase was stabilized for the samples prepared before and after heat treatment with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, i.e., without partitioning into separate components.

Keywords: zirconia, medical applications, composite, alumina, and hydroxyapatite

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1: Introduction

Hydroxyapatite is important because it has many uses, particularly in medicine. Calcium apatite occurs naturally as hydroxyapatite, having the molecular formula Ca5(PO4)3(OH). To indicate that the crystal unit cell is made up of two entities, it is commonly written as $Ca_{10}(PO_4)_6(OH)_2$. Its hexagonal crystalline structure has dimensions a= 9.41Å and c= 6.88Å, and its molecular weight is 502.31 g/mol. It may be milky, yellow, white, gray, or yellowish green, among other colors. Human bones are primarily composed of inorganic substances called hydroxyapatite, which makes up 65–70% of the weight of natural bone, and organic substances called collagen fibers. The metal component, hydroxyapatite, is what gives bones their stiffness; collagen fibers are what give bones their flexibility. In bone tissue, hydroxyapatite has a molecular ratio of roughly 1.67 for calcium to phosphorus (Ca/P) (Kupiec et al., 2012; Uddin et al., 2010; Azmat and Nagtode, 2015).

In many medical applications as well as the direct replacement of solid tissues like bone, calcium phosphate (CaP) is a material that is widely used. There are numerous varieties of calcium phosphate (CaP), including calcium phosphate in binary form (BCP), anhydrous dia-calcium phosphate (DCP), hydroxyapatite, tetra-calcium phosphate (TTCP), tri-calcium phosphate (TCP), and amorphous calcium phosphate (ACP). Because of its consistency and biocompatibility, hydroxyapatite represents the most promising and efficient substance for restoring lost bone (Pal and Bag, 2005). Numerous methods, including electrodeposition, precipitation, hydrothermal technique, sol-gel, biomimetic deposition, direct use of animal bones, and other

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methods, were used to prepare hydroxyapatite. (Nayak. 2010). Upon thermal treatment, samples that have undergone any form of formation disintegrate into multiple compounds, including β-Ca3 (PO4) 2, also known as β-TCP, once the temperature reaches over 1100°C (Hannora, 2014; ShekharNath et al., 2010; Bulut et al., 2015; Pujiyanto et al., 2012). It can dissolve based on the reactions listed below (Aminzare et al., 2013; Ebrahimi et al.,2012).

$$Ca_{10}(PO_4)_6(OH)_2 \rightarrow Ca_4P_2O_9 + 2Ca_3(PO_4)_2 + H_2O \dots\dots (1)$$
$$Ca_{10}(PO_4)_6(OH)_2 \leftrightarrow 3Ca_3(PO_4)_2 + CaO + H_2O \dots\dots\dots (2)$$

It is possible to coat every part of the human body with hydroxyapatite. Parts of the bones, jaws, face, teeth, and skull can also be prepared as whole human bodies. It also finds its way into many body parts' cosmetic procedures. can also play a significant role in the process of making animal feed (Mohamaddoost et al., 2014).

2: Experimental Work:

First; to make the hydroxyapatite powder on-site, do the following:

1. Prepare Iraqi beef bones from local markets, which were first cleared of any remaining meat and fat.
2. To get rid of the fat, boil the bones in water for five hours, several times.
3. Use an electric dryer set to 75 °C for a full day to dry the bones.
4. Using an iron hammer to crack bones into tiny pieces.
5. The broken bones should be placed in a ceramic package and heated to 750°C for 4 hours. This procedure will guarantee that every fractured bone has become completely white and has lost any organic material.
6. The bones were ground in a rotary ball mill to get a fine powder.
7. Using sieves to filter the powder with particles smaller than 150 µm.
8. Using the XRD diffraction system to perform tests for substance and phase diagnosis.

Second; sample preparation:

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1. Using a sieve with a particle size of less than 150 microns to screen various powders ALM-41 -41 –SUMITO CHEMICAL CO., UK is the source of the alumina used in the first composition (HA) preparation of hydroxyapatite alumina with alumina weight percents of 0, 5, 10, 15, 20, and 25%. The Zirconia percentages in the second composition (HZ) preparation of Hydroxyapatite-Zirconia are 0, 5, 10, 15, 20, and 25 weight percent. It should be noted that the zirconia (ZrO2-plus 5.4 weight percent Y2O3) utilized comes from ZIROCNIA SALES-GU185SS-U.K. As alumina and zirconia were present in 80% and 20% of the third composite (HAZ) preparation of hydroxyapatite, respectively, the percentages of alumina and zirconia were 0.5, 10, 15, 20, and 25 weight percent, respectively.
2. Polyethylene glycol (SIGMA-ALDRICH-CO.-WG) has a molecular weight of 6000. Polyethylene glycol was dissolved in alcohol ethanol with a volume of 1 g/100 ml at 40°C to serve as a bond material. To produce a uniform solution, use a magnetic stirrer to continuously move the mixture.
3. To prepare mixtures of hydroxyapatite composite 99% wt versus 1% wt bonding material. The polyethylene solution was diluted, and a magnetic mixer was used to create a homogenous solution.
4. Use a dryer to dry the mixture of materials for 24 hours at 75°C.
5. Compressing the samples into a semi-dry form using a 9-mm metal mold and 2.75 tons of pressure.
6. Apply the heat treatment at 5 ^{o}C/min and hold the temperature at 200 ^{o}C for two hours. Then, increase the temperature at the same rate to 1350 ^{o}C and hold it there for three hours. Finally, reduce the temperature at 5 ^{o}C/min to room temperature.

Third; Examinations:

1. To determine the kind and phase of the substance, use X-ray diffraction tests. Figure (1) illustrates this by comparing the phase of the material to the hexagonal phase of hydroxyapatite with the card number (34-0010).

2. Figures (2–7) represent the radial shrinkage (RS) or diameter contraction (DC), Vickers hardness (H_V), compressive strength (σ), apparent density (ρ), porosity (P), and water absorption ratio (WA), respectively.

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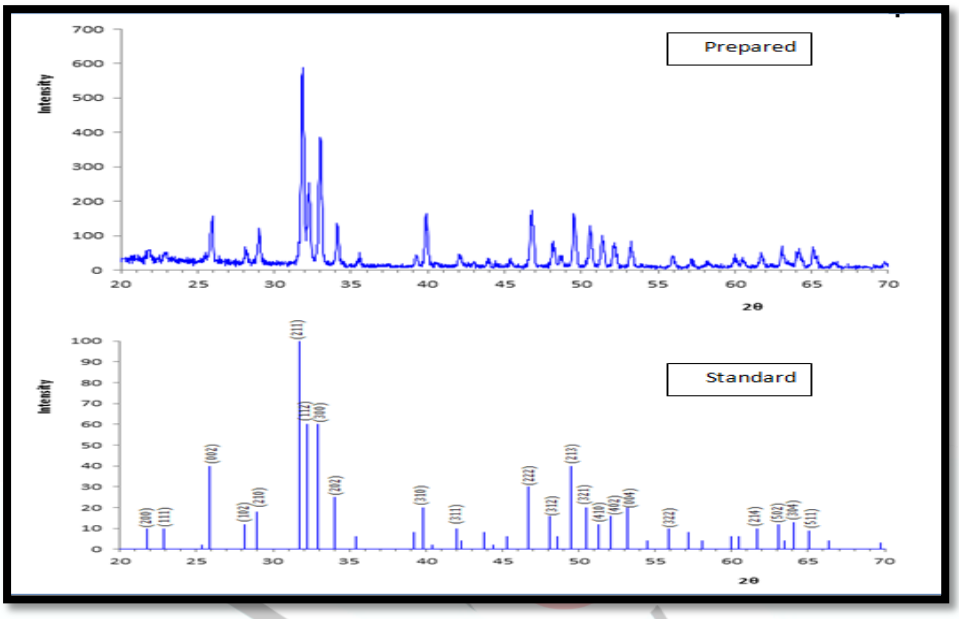

Figure (1) shows the X-ray diffraction spectra of the hydroxyapatite sample by itself after three hours of heat treatment at 1350 °C.

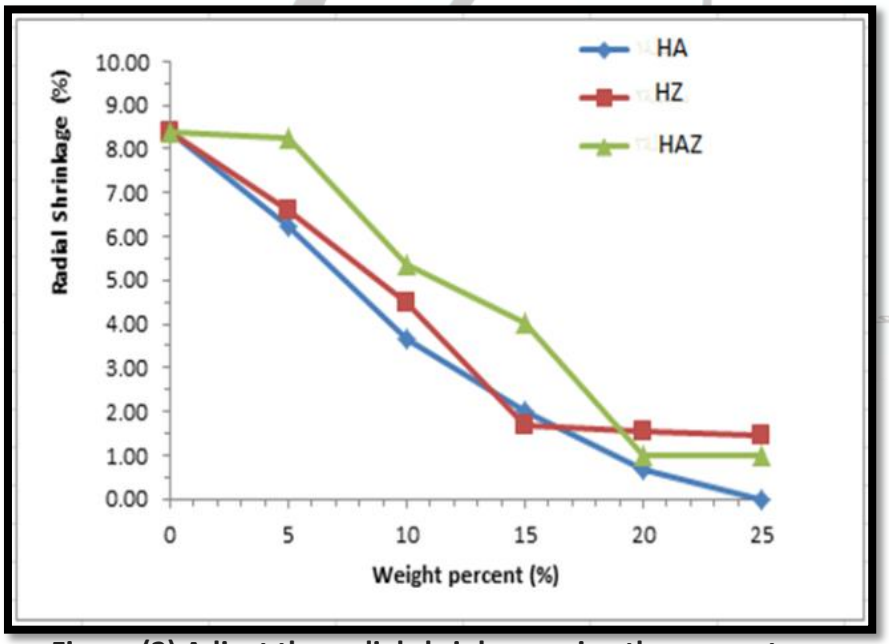

Figure (2) Adjust the radial shrinkage using the percentage

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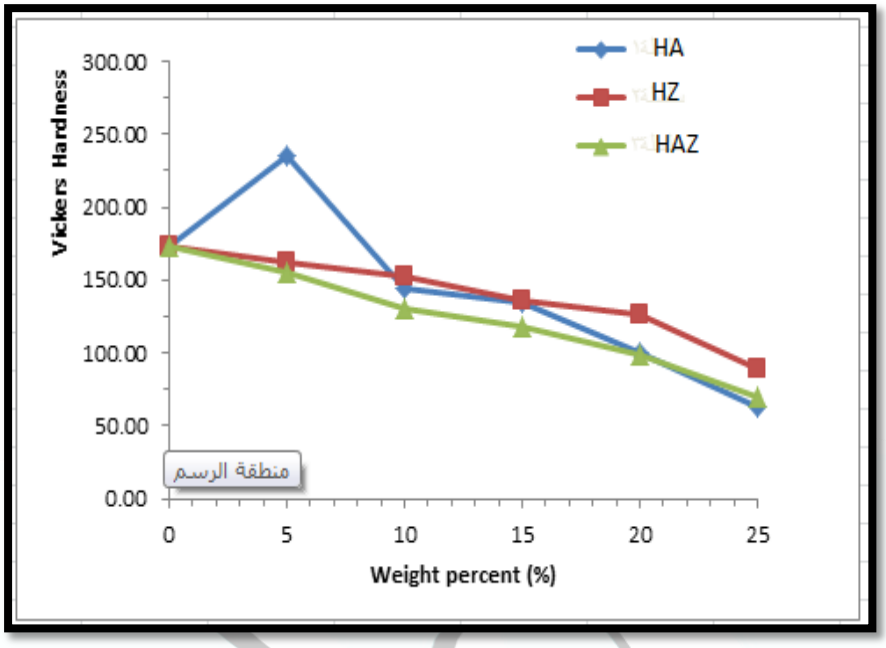

Figure (3) Vary Vickers hardness according to a percentage.

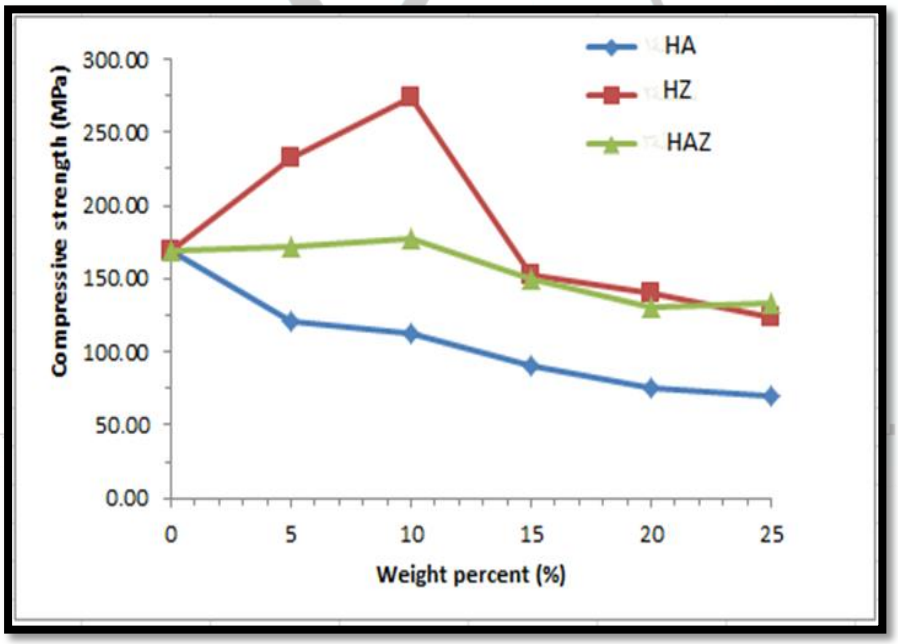

Figure (4) Variation in compressive strength with percentage.

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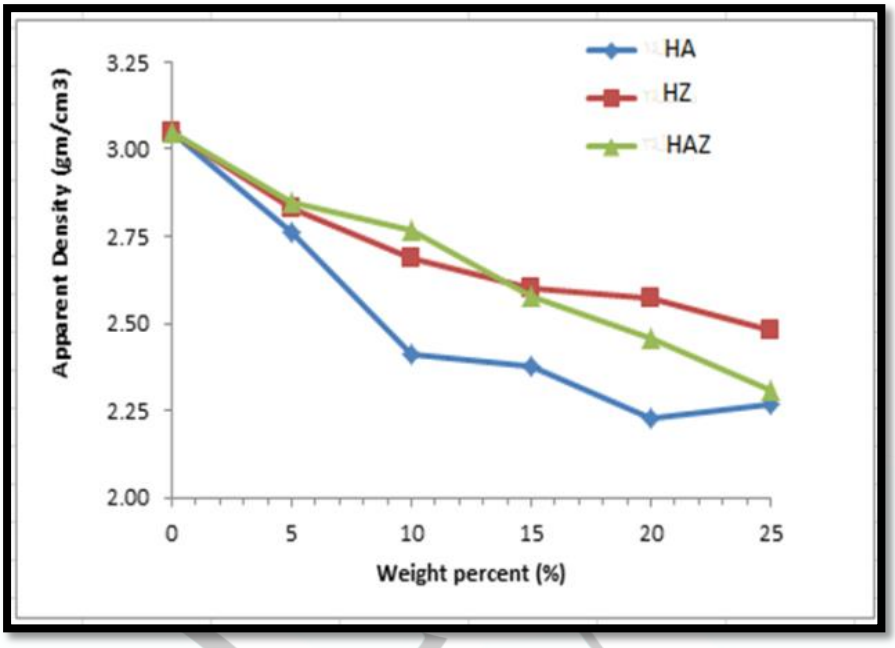

Figure (5) Vary the apparent density using a percentage.

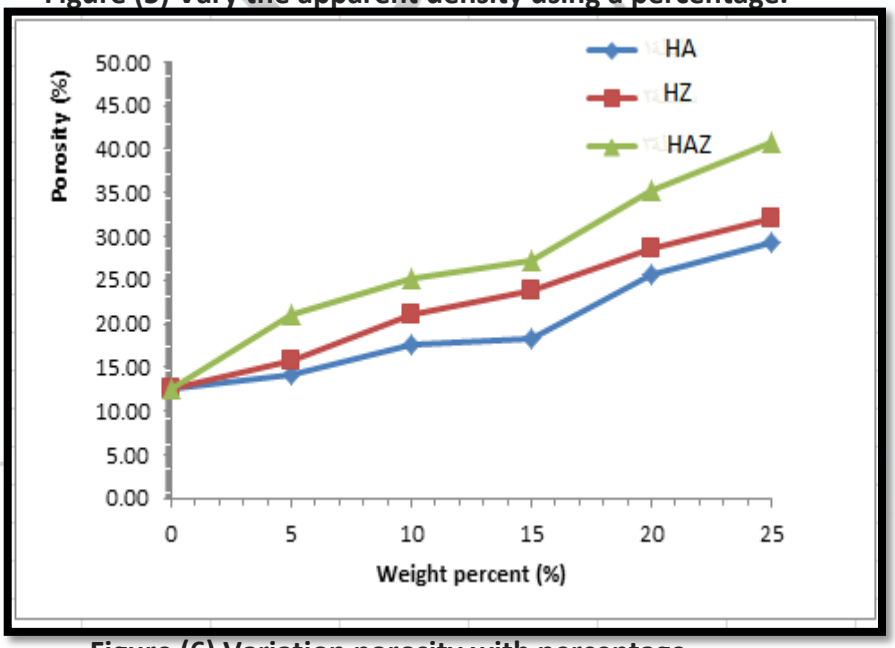

Figure (6) Variation porosity with percentage.

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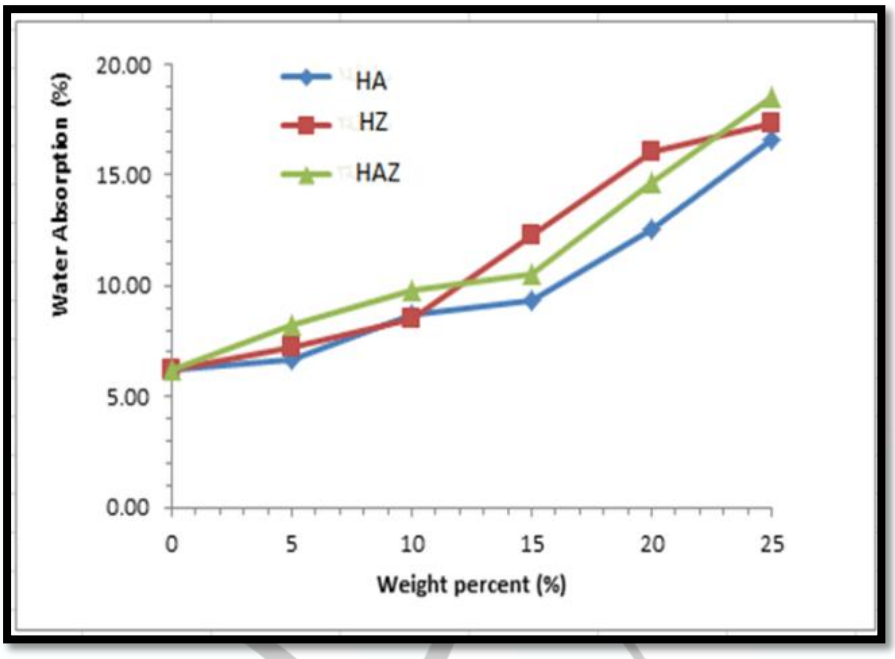

Figure (7) Vary water absorption based on weight percentage.

3- Discussion:

By comparing the prepared material's X-ray diffraction in Figure (1) with the standard spectra of card No. (34-0010), it can be determined that the phase is hexagonal and that the material is white color. Because of the selected thermal treatment program, the XRD spectra of the samples prepared at 1350 °C for 3 hours preserve the hydroxyapatite phase without changing in phases, which is very helpful in the different applications for hydroxyapatite and hydroxyapatite-composites. The majority of the references discuss how hydroxyapatite breaks down during heat transactions that are higher than 1100°C (*Hannora, 2014; ShekharNath et al., 2010; Bulut et al., 2015*).

Figure (2) illustrates the variation in radial shrinking with alumina, zirconia, and alumina-zirconia ratios. It demonstrates a linear decrease with ratios because, in order to complete the sintering process, we require a temperature greater than 1350°C,. Vickers hardness with alumina, zirconia, and alumina-zirconia ratios is represented in Figure (3). At 5% wt of alumina ratio, the maximum hardness value is displayed; for the same reason as shown in Figure (2), the values of hardness decreased, which is insufficient for the heat treatment temperature. The values of hardness match the reference values. (*Bulut et al., 2015*). Figure (4) illustrates the compression strength in relation to alumina, zirconia, and alumina-zirconia ratios. The highest compression strength value is displayed at 10% weight percent zirconia ratio, and the form indicates a decline in values as ratios increase because of the low sintering temperature. The results are consistent with the reference

values (*Hannora, 2014*). A decrease in the density ratios is observed in Figure 5, along with changes in the zirconia, alumina, and alumina-zirconia ratios. The reason for this decrease is the same as previously mentioned, even though the ratios have increased. In order to counteract the apparent density's behavior, Figures (6,7) illustrate the variations in porosity and water absorption rate. The figures show that hydroxyapatite alone has the lowest porosity and water absorption needed to reach the right level of sintering.

4- Conclusion:

The powder has the following characteristics: it is milky white, highly pure, hexagon-shaped hydroxyapatite, easily ground and screening to the appropriate particle size (PS), and has a stable phase throughout time and in various climates. With the exception of the Vickers hardness, which peaked at 5% weight of alumina, and the compression strength, which peaked at 10% weight of zirconia ratio, pure hydroxyapatite exhibited the best qualities. Additionally, by choosing the right heat treatment program, the hydroxyapatite phase was maintained both before and after thermal treatment.

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