Mechanical strength characterization and modeling of hydroxyapatite/tricalcium phosphate biocomposite using the diametral-compression test

M. Es-saddik¹, S. Laasri², A. Laghzizil³, J.M. Nunzi⁴, M. Taha¹, A. Guidara⁵, A. Hajjaji², J. Bouaziz⁵

¹Laboratory of Thermodynamics and Energetics, University Ibn-Zohr, Faculty of Sciences, BP 8106 Agadir Morocco
²Laboratory of Engineering Sciences for Energy, National School of Applied Sciences of El Jadida, BP 1166, EL Jadida Plateau 24002, Morocco
³Laboratory of Applied Chemistry of Materials, Faculty of Sciences, University of Mohammed V in Rabat, BP.1014 Rabat Morocco.
⁴Department of Chemistry; Department of Physics, Engineering Physics and Astronomy, Queen's University, Kingston, ON K7L 3N6, Canada
⁵Laboratory of Advanced Materials, National School of Engineering, Sfax, Tunisia

Abstract

This study reports the enhanced mechanical resistance of the composite bioceramics of hydroxyapatite (HAP) and tricalcium phosphate (β-TCP) used as bone substitute. HAP/β-TCP mixture was prepared by wet mixing of powders and characterized. Effects of powder manufacturing and sintering temperature on the densification, microstructure and mechanical properties of the composite were studied. The rupture strength (σr) was calculated using the Brazilian test. At 1250°C, the relative density and mechanical strength of the HAP/β-TCP ceramics reached the maximum value of 89% and 43 MPa, respectively. Experimental results were modeled by the finite element method to determine the stress distribution in the compacted disc.

Keywords: Hydroxyapatite; β-tricalcium phosphate; Mechanical strength; Brazilian test; Finite element method.
1. Introduction

Calcium phosphate bioceramics were used as a bone substitute for many decades. Their applications were demonstrated in various medical fields such as dentistry, orthopedics and facial surgery [1, 2]. Different calcium phosphates were tested and found interest in biotechnology, but their use depends mainly on their resorbability and/or bioactivity characteristics [3]. Bioceramics of hydroxyapatite (Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\), called HAP), and tricalcium phosphate (β-Ca\(_3\)(PO\(_4\))\(_2\), called β-TCP) are perfectly biocompatible, and can restore weakened bone by promoting bone colonization in a wide variety of clinical processes [4-6]. Unfortunately, despite their attractive characteristics, Thomas and Doremus [7] and Landi et al. [8] reported that their mechanical characteristics were poor, limiting their medical applications to small implants with reduced mechanical load. Mechanical strength of the specimens was measured by several methods, like the compression test [9], the three and four-point bending tests [10, 11], the flattened Brazilian test [12-14] and the diametral compression method (DCM) [15-18]. The diametral compression method was renamed Brazilian test after Carneiro and Barcellos [19] who used it as an indirect procedure to determine the tensile strength of quasi-fragile materials. More recently, Azam et al. [20] praised the method for three reasons. (i) there was a strong similarity between the stresses induced during deformation in the diametral test and those experienced in dental and other biomedical implants. (ii) the tensile strength obtained for a brittle material was more consistent than with other methods because all the bulk material was exposed to the tensile stress in the DCM method, whereas only the specimen surface was subjected to the maximum tensile stress in the four-point bending test. (iii) this method was particularly appropriate for biomaterial testing. For these reasons, this study attempts to elaborate HAP/β-TCP composites sintered at various temperatures and assess their structural, textural and mechanical properties using the DCM for a more accurate assessment of their most important mechanical properties. This study focuses in particular on the determination of the rupture strength of the bioceramics as a function of their preparation procedures. The tensile strength is modeled using mathematical expressions for the determination of the stress-strain characteristic of the biomaterial.
2. Materials and method

2.1. Experimental

Hydroxyapatite was prepared following Laasri et al. [11]. Therefore, the Ca(OH)$_2$ (Merck, Purity>99%) and H$_3$PO$_4$ (Merck, Purity>99%) were neutralized as starting materials in order to reduce the generation of by-products. β-TCP was purchased from Fluka (Purity>98%). The 60%wt.HAP-40%wt.β-TCP composite (called 60HAP/β-TCP) was developed by mixing the two previous powders in 50mL of absolute ethanol and 100 mL of deionized water. The mixture was stirred for 20 hours at room temperature and then dispersed in an ultrasonic bath three times for 15 min. The filtrate precipitates were dried in an oven at 80°C overnight and then calcinated at 800°C for 3h (10 °C min$^{-1}$). The resulting powders were molded in a hardened steel cylinder having a 10 mm-diameter and pressed under 150 MPa using 5 mm/min-pressing rate. Finally, the pellets sintered at different temperatures (1200°C, 1250°C, 1300°C) in an air atmosphere for 3 hours at 10°C min$^{-1}$ were characterized by several techniques.

2.2. Techniques

The particle size distribution was determined using a Micromeritics Sedigraph 5000 (DSL). The crystalline phases were identified by X-ray diffraction (XRD) using a Bruker D8 Advanced diffractometer using CuK$_\alpha$ radiation ($\lambda = 1.54056$ Å). Fourier Transform-Infrared Spectroscopy (FTIR) was used to identify the vibration bands attributed to the functional groups present in the 60HAP/β-TCP bioceramic. Infrared spectra of the powders were recorded on a Fourier Transform Spectrometer (SHIMADZU FTIR-8400S) in the 4000-400 cm$^{-1}$-range. Microstructure of the sintered pellets was investigated by scanning electron microscopy on the fractured sample surfaces. Scanning electron microscope SEM images were taken with a VEGA 3 TESCAN. Samples were metalized with gold for imaging. Mechanical experiments were realized using a LLOYD EZ50 device on 2 mm-thickness and 8 mm-diameter cylindrical samples. The geometrical dimensions were determined with a 0-150 mm / 0-6”-digital caliper. Rupture strength of the ceramics was measured by the DMC test. This method consists in applying a diametral compression onto a cylindrical specimen by adjusting the load uniformly along the line formed by two diametrically opposed generatrices (Fig. 1).
Relative density $d_r$ of the ceramics was determined according to the following formula:

$$d_r = \frac{\rho_{\text{apparent}}}{\rho_{\text{absolute}}} \quad \text{(Eq. 1)}$$

where $\rho_{\text{apparent}}$ is the density of the sintered pellet, which was determined by geometrical measurement, and $\rho_{\text{absolute}}$ is the density of the unsintered powders. This relative density is calculated from density measurements and the theoretical density $d_{th}$ following [21]:

$$d_{th} = w_{\text{HAP}} \cdot d_{\text{HAP}} + w_{\beta-\text{TCP}} \cdot d_{\beta-\text{TCP}} \quad \text{(Eq. 2)}$$

where $d_{\beta-\text{TCP}} = 5.68$ [22-23] and $w_{\beta-\text{TCP}} = 0.4$ are the theoretical density and the content of zirconia, respectively, and $d_{\text{HAP}} = 3.156$ [24] and $w_{\text{HAP}} = 0.6$ are the theoretical density and the HAP content in the composite, respectively.

The elastic modulus, Young’s E and shear modulus G, of HAP/β-TCP composite were calculated by the ultrasonic technique, in which the elastic properties were determined from the longitudinal ($V_L$) and transversal ($V_T$) ultrasonic velocities of the waves. The testing instrument includes a generator of short intense pulses (Sofranel 5052 PR), which excite a broadband ultrasound emitter (Panametrics V 309) with a central frequency of 5 MHz. The sound velocity was calculated by determining the time delay with which the wave propagates in the specimens of thickness $t$. According to the elastic theory, velocities of the wave are expressed with Young’s modulus (E), the shear modulus (G), density ($\rho$) and Poisson’s ratio ($\theta$) [25, 26]:

$$V_L = \sqrt{\frac{C}{\rho}} = \left(\frac{E (1 - \theta)}{\rho (1 - \theta)(1 - 2\theta)}\right)^{1/2} \quad \text{(Eq. 3)}$$
\[ V_t = \sqrt{\frac{G}{\rho}} = \left( \frac{E}{2\rho(1+\vartheta)} \right)^{1/2} \]  

(Eq. 4)

where \( C \) is known as the longitudinal modulus.

The elastic modulus (E and G) and the Poisson's ratio \( \nu \) can be calculated using the formula:

\[ G = \rho V_t^2 \]  

(Eq. 5)

\[ E = G \frac{3C-4G}{C-6G} \]  

(Eq. 6)

\[ \vartheta = \frac{C - 2G}{2(C - G)} \]  

(Eq. 7)

The applied force induces tensile and compressive stresses in the plane containing the cylinder axis and the generatrix (Fig.2a). The diametral plane connecting the sample and the two plates is uniformly in tension, assuming an elastic behavior of the sample. If the thickness of the sample is smaller than its diameter, an increased load creates a stress distribution, which gradually increases until failure (Fig. 2b).

Maximal rupture strength \( (\sigma_r) \) values were obtained from the following equation [27]:

\[ \sigma_r = \frac{2F}{\pi Dt} \]  

(Eq. 8)

where \( F \) is the maximum applied load, \( t \) is the thickness and \( D \) the diameter of the specimen.
2.3. Theoretical stress distribution analysis within a circular disk

Computation was achieved by supposing a point loading \( P \) (Fig. 3a) uniformly distributed on the contact surface defined by the solid angle \( 2\varphi \) (Fig. 3b). As reported by Timoshenko and Goodier [28] and Frocht [29], the elastic analysis of the stress was achieved within a circular disc subjected to two concentrated diametrically opposed forces, assuming a plane stress in the case of a point loading. The stress fields on the disc surface are computed in a polar coordinates system by the following expressions:

\[
\sigma_r = \frac{2F}{\pi R} \left[ \frac{1}{2} - \frac{1 - (r/R)^2 - (r/R)^2 \cos^2 \theta}{(1 + (r/R)^2 - 2r/R \cos \theta)^2} \right] \quad \text{(Eq. 9)}
\]

\[
\sigma_\theta = \frac{2F}{\pi R} \left[ \frac{1}{2} - \frac{1 - (r/R)^2 \sin^2 \theta}{(1 + (r/R)^2 - 2r/R \cos \theta)^2} \right] \quad \text{(Eq. 10)}
\]

\[
\sigma_{r,\theta} = \frac{2F}{\pi R} \left[ \frac{(1 - (r/R)^2)(\cos \theta - r/R)^2 \sin \theta}{(1 + (r/R)^2 - 2r/R \cos \theta)^2} - \frac{(1 - (r/R)^2)(\cos \theta + r/R)^2 \sin \theta}{(1 + (r/R)^2 + 2r/R \cos \theta)^2} \right] \quad \text{(Eq. 11)}
\]

where, \( \sigma_{r,\theta} \) is the shear stress, \( \sigma_r \) and \( \sigma_\theta \) are the normal stresses in the directions perpendicular and parallel to the loaded diameter, respectively. \( F \) is the applied load and \( R \) the radius.

Note that a point like loading is not possible since the local pressure would become infinite, which is why the load is distributed (Fig. 3b), as was considered by Hondros [16]. Expressions of the stresses applied to the loading plane are then given by the following equations:

\[
\sigma_{yy} = -\frac{2P}{\pi} \left[ \frac{(1 - (y/R)^2)^2 \sin 2\varphi}{1 - 2(y/R)^2 \cos 2\varphi + (y/R)^2} \right] \quad \text{(Eq. 12)}
\]

\[
\sigma_{xx} = \frac{2P}{\pi} \left[ \frac{(1 - (y/R)^2)^2 \sin 2\varphi}{1 - 2(y/R)^2 \cos 2\varphi + (y/R)^2} \right] \quad \text{(Eq. 13)}
\]

Where \( P \) is the applied pressure (MPa) and the force \( F = P \cdot \varphi \cdot D \cdot t \).

At the center of the disc where \( x = y = 0 \), the principal stresses become:

\[
\sigma_{yy}(0; 0) = -\frac{2P}{\pi} \left[ \sin 2\varphi + \varphi \right] \quad \text{(Eq. 14)}
\]

\[
\sigma_{xx}(0; 0) = \frac{2P}{\pi} \left[ \sin 2\varphi - \varphi \right] \quad \text{(Eq. 15)}
\]

When \( \varphi \) is small, \( \sin 2\varphi \approx \varphi \) and equations 7 and 8 become:

\[
\sigma_{yy}(0; 0) = -\frac{6P \varphi}{\pi} = -\frac{6F}{\pi D t} \quad \text{(Eq. 9)}
\]

\[
\sigma_{xx}(0; 0) = \frac{6P \varphi}{\pi} \quad \text{(Eq. 16)}
\]
$$\sigma_{xx}(0;0) = \frac{2P\varphi}{\pi} = \frac{2F}{\pi D \epsilon} \quad (Eq. 10)$$

Along the loaded diameter \((x = 0)\), the normal stress \(\sigma_{xx}\) is tensile and constant, and \(\sigma_{yy}\) parallel to the loaded diameter is a compressive stress.

Fig. 3. The diametral disk test: (a) under a point load, (b) under a distributed pressure.

2.4. Finite element analysis
The Cast 3m calculation code based on the finite element method (FEM) was used in this work to determine the stress distribution in a circular disk subjected to two concentrated diametral forces. Boundary conditions are defined as follows (Fig. 4a): (i) Line L1 is blocked in the X direction, (ii) Line L4 is blocked in the Y direction and (iii) the center of the disc is blocked in the x and y directions. The meshes were created by quadrilateral elements with four nodes in two dimensions (Fig. 4b) and the deformation of the disc after diametral compression is shown in figure 4c. It shows the effect of two concentrated diametral forces on the circular disc, confirming that the boundary conditions were respected.
Fig.4. (a) Boundary conditions, (b) Meshes and (c) Deformation.

The elastic behavior of the 60HAP/β-TCP specimen was considered isotropic and its physical properties as well as geometrical dimensions are given in Table 1. The Young modulus and Poisson ration of 60HAP/β-TCP sintered at 1250°C were determined experimentally using the ultrasonic technique. From the symmetry of the system, it was possible to limit the FEM-analysis to a quadrant of the circular disc and a maximum experimentally force load $F = 1117$ N was applied on the arc defined by the angle $2\varphi$ of the specimen.

Table1. Characteristics of 60HAP/β-TCP ceramic sintered at 1250°C.

<table>
<thead>
<tr>
<th>Angle 2\varphi (°)</th>
<th>Thickness (mm)</th>
<th>Diameter (mm)</th>
<th>Young modulus (GPa)</th>
<th>Poisson ratio</th>
<th>Density (Kg/m³)</th>
<th>Applied pressure (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1547.84</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>773.92</td>
</tr>
<tr>
<td>15</td>
<td>1.85</td>
<td>8.94</td>
<td>77.27</td>
<td>0.1895</td>
<td>2797</td>
<td>515.94</td>
</tr>
<tr>
<td>16</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>483.70</td>
</tr>
<tr>
<td>17</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>455.24</td>
</tr>
<tr>
<td>20</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>386.96</td>
</tr>
</tbody>
</table>

3. Results and discussion
3.1. Characterization

Figure 5 shows the DSL analysis of the particle size distribution in the studied powders. The main particle population has an average diameter $D_{50}$ of 67.3 µm, 6.08 µm and 4.42 µm for HAP, β-TCP and 60HAP/β-TCP, respectively. As in the primitive powders, the fine granulation of 0.4 µm in the composite is expected to improve its compactness and subsequently, its rupture strength.
Fig. 5. Particle size distribution of HAP, β-TCP, and 60HAP/β-TCP powders calcined at 800°C for 3 h.

Figure 6 displays the XRD patterns of 60HAP/β-TCP800 compared to those of HAP800 and β-TCP 800 as references. There are only HAP peaks (ICDD data file no. 00-054-0022) without any other phase. Similarly, all the peaks of tricalcium phosphate are those of the β-TCP phase (ICDD data file no. 00-009-0169), while the peaks detected in the 60HAP/β-TCP800 correspond to the simultaneous coexistence of HAP and β-TCP phases, without any other phases. There is only a slight change in the intensity of a few peaks observed for 60HAP/β-TCP800.

Fig.6. XRD patterns of 60HAP/β-TCP biocomposite dried at 100 °C (60HAP/β-TCP100) and calcined at 800°C (60HAP/β-TCP800) compared to those of HAP and β-TCP calcined at 800°C, as references.
The infrared spectra of the powders are shown in Figure 7. Two absorption domains located between 1100-900 cm\(^{-1}\), and between 500-600 cm\(^{-1}\) characterize the PO\(_4\) vibrations. The first one corresponds to symmetrical and antisymmetric P-O (\(\nu_1+\nu_3\)) vibrations and the second is related to the O-P-O (\(\nu_2+\nu_4\)) deformation. As figure 7 shows, the absorption bands of PO\(_4\) in HAP and \(\beta\)-TCP overlap because the absorption ranges of the different oscillations are close. For HAP and 60HAP/\(\beta\)-TCP samples, two characteristic bands of the OH hydroxyl are detected at 3570 and 630 cm\(^{-1}\) [30, 31].

![Fig. 7. FT-IR spectra of calcium phosphate powders:](image)

(a) HAP; (b) \(\beta\)-TCP; (c) 60HAP/\(\beta\)-TCP dried at 80°C; (d) 60HAP/\(\beta\)-TCP800.

### 3.1.2. Sintering and mechanical properties of the elaborated materials

Figure 8 shows histograms representing density and diametral strength of \(\beta\)-TCP, HAP and 60HAP/\(\beta\)-TCP as function of the sintering temperature. As seen in Figure 8a, the relative densities of HAP, \(\beta\)-TCP, and 60HAP/\(\beta\)-TCP reach their maximum values of 98\%, 79\% and 89\% at 1250°C, 1250°C and 1300°C, respectively. This correlates well with published data [4]. Figure 8b shows the histograms representing the diametral strength of \(\beta\)-TCP, HAP and 60HAP/\(\beta\)-TCP sintered at 1200, 1250 and 1300°C. The maximum rupture strength of HAP is 24 MPa at 1250°C. It then drops to 6.91 MPa at 1300°C. This can be explained by its density increase at this temperature. On the other hand, the 60HAP/\(\beta\)-TCP composite exhibits a diametral strength of 43 MPa at 1250°C. However, increase of the sintering temperature to 1300 °C does not lead to a remarkable drop in its diametral strength. This would imply that the sintering temperature has fewer impact on the mechanical strength increase of the 60HAP/\(\beta\)-TCP ceramic than on the HAP one.
Fig. 8. Histograms representing, (a) Relative densities and diametral strength of β-TCP, HAP and 60HAP/β-TCP materials as a function of the sintering temperature.

3.1.3. Characterization of the samples after sintering

Figure 9 presents the XRD analysis of the 60HAP/TCP composite sintered at various temperatures. There is a clear presence of the HAP and β-TCP phases as well as traces of α-TCP phases. The latter may be due to the transformation of β-TCP into α-TCP. Nevertheless, the HAP peaks decreased with the increase of the sintering temperature. At 1300°C, intensity of the peaks related to α-TCP phase increases. This would explain the slight decrease in density and diametral strength of the 60HAP/β-TCP at 1300°C.

Figure 10 shows two different magnifications of an SEM image of the fracture surface of the 60HAP/β-TCP bioceramic sintered at 1250°C. As seen in Fig. 10a, there are dense structures with the existence of some closed pores. This would corroborate with the finding on the density and mechanical strength. Thus, at 1250°C, 60HAP/β-TCP composite reach the optimum density and mechanical strength because at the microstructure level, their density is at its maximum and the pores are closed. Hence, their mechanical strength should be at the best.
Figure 9. XRD patterns of 60HAP/TCP biocomposite sintered at various temperatures (h: HAP, β: β-TCP, α: α-TCP).

Table 2 summarizes the findings related to the Brazilian test tensile strength measurement as a function of temperature. The mixture of HAP with β-TCP results in the 60HAP/β-TCP bioceramic having and improved mechanical resistance. Indeed, when sintered at 1250 °C, this composite reaches a diametral strength of 43.0 MPa. Furthermore, its mere 11% porosity is an advantage to accelerate the growth of bone tissues without negatively affecting the mechanical properties. The presence of
absorbable β-TCP or α-TCP phases may also contribute to subsequent implant-bone interfacial adhesion and bone intergrowth. Therefore, the 60HAP/β-TCP biocomposite combines several desired properties and is likely to be efficient as a bone implant into the human body.

**Table 2.** Diametral strength and optimum sintering temperature of the samples.

<table>
<thead>
<tr>
<th></th>
<th>Diametral strength (MPa)</th>
<th>Optimum temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HAP</td>
<td>23.00±0.24</td>
<td>1250</td>
</tr>
<tr>
<td>β-TCP [17]</td>
<td>5.30</td>
<td>1320</td>
</tr>
<tr>
<td>60HAP/β-TCP</td>
<td>43.00±0.45</td>
<td>1250</td>
</tr>
</tbody>
</table>

3.2. **Numerical analysis**

Dorémus et al. studied the influence of the sample diameter \(D\) and thickness \(t\) on the tensile strength from a compacted metal powder [32]. From their work, we conclude that the ratio \(t/D < 0.25\) warrants the validity of the plane stress conditions in our numerical study. Figure 11 shows the distribution pattern of \(\sigma_{xx}\), \(\sigma_{yy}\), and \(\sigma_{xy}\) stresses corresponding to the loaded angle \(2\varphi = 16^\circ\). Most of these are active on the entire volume of the disc.

**Fig.11.** Distribution pattern of (a) \(\sigma_{xx}\), (b) \(\sigma_{yy}\), and (c) \(\sigma_{xy}\) stresses in the specimens after diametral compression at the loaded angle \(2\varphi=16^\circ\).
Figure 12 displays two representations of the compressive stress. As can be seen in Figure 12a, the stress seemed to have been constant from the center of the disc to the surface along the diametral axis, generating a crack perpendicular to the main direction X; this phenomenon is shown schematically in Fig. 12b indicating the stress distribution around the loaded diameter of a disc. As a result, the sample was broken into two pieces along this diameter.

![Figure 12](image.png)

**Fig.12.** Representations of the compressive stress. (a) photo of fractured disc after diametral compression; (b) schematically stress distribution across the loaded diameter of a disc.

Figure 13 displays the variation of \( \sigma_{xx} \) and \( \sigma_{yy} \) stresses in function of the normalized distance. A comparison of the stress distribution model with the experimental results would lead to the expected conclusion that fracture occurred under the action of the maximum tensile stresses. However, fracture occurrence would be possible under the effect of maximum shear stresses. Nevertheless, the numerical results are in good agreement with the analytical ones proposed by Hondros [16]. Indeed, the simulated and analytical stresses are comparable up to \( y/R = 0.9, 0.8, 0.8, 0.8, 0.8, \) and 0.7 for \( 2\varphi = 5^\circ, 10^\circ, 15^\circ, 16^\circ, 17^\circ, \) and \( 20^\circ; \) respectively. The contact angle affects the two solutions close to the point of application of the forces. In all the finite element computations, the stress fields computed at the center of the disc are of the same order of magnitude as those obtained from the analytical solutions. Therefore, it can be concluded that there is a good agreement between the two curves for the component \( \sigma_{yy}. \)
Fig. 13. Variation of $\sigma_{XX}$ and $\sigma_{YY}$ stresses versus the normalized distance $y/R$: 

$2\phi = 5^\circ$, $2\phi = 10^\circ$, $2\phi = 15^\circ$, $2\phi = 16^\circ$, $2\phi = 17^\circ$ and $2\phi = 20^\circ$.

To assess this, the diametral strength determined experimentally using equation (8), and the analytical (Handros) and numerical (FEM) results at the center of the disc were compared. The results are listed in Table 3. According to Griffith’s theory, if the tensile stress is considered positive and $\sigma_{xx} \geq \sigma_{yy} \geq \sigma_{zz}$, the equivalent stress $\sigma_G$ is calculated as follows:

when $3\sigma_{xx} + \sigma_{yy} \geq 0$; $\sigma_G = \sigma_{xx}$ and when $3\sigma_{xx} + \sigma_{yy} < 0$;

$$\sigma_G = \frac{(\sigma_{xx} - \sigma_{yy})^2}{-6(\sigma_{xx} + \sigma_{yy})}$$  \hspace{1cm} (Eq. 18)

At the center of the disk $3\sigma_{xx} + \sigma_{yy} = 0$,

$$\sigma_G = \frac{2F}{\pi d t}$$  \hspace{1cm} (Eq. 19)

which is equivalent to (Eq.8).
In this study, $3\sigma_{xx} + \sigma_{yy} < 0$. The relation given by equation (3) was used to determine the tensile strength in the diametral test. Figure 14 shows the variation of the equivalent stress $\sigma_G$ as a function of the $\frac{y}{r}$ ratio for various $2\varphi$ values. The maximum value of the equivalent stress at the center of the disc was recorded for $2\varphi \geq 16$. This would imply that the crack was initiated from the center of the sample. Table 3 reveals that the equivalent stress was closer to the stress $\sigma_{xx}$ at the center of the sample. The tensile strength is calculated by:

$$\sigma_t = \max(\sigma_G) = \left. \left(\frac{(\sigma_{xx}-\sigma_{yy})^2}{-\delta(\sigma_{xx}+\sigma_{yy})}\right) \right|_{x=0: y=0} \quad (Eq. 20)$$

or $\sigma_t \approx \sigma_{xx}|_{x=0: y=0} = \frac{2F}{\pi dt}$

Therefore, we recorded a strong correlation between the tensile strengths determined numerically and experimentally, although the experimental value of the contact angle $2\varphi$ was not fixed.

**Table 3.** Comparison of experimental, numerical and analytical solutions

<table>
<thead>
<tr>
<th>$2\varphi$ (°)</th>
<th>Experimental from Eq. (8) $\sigma_t$ (MPa)</th>
<th>Hondros $\sigma_{xx}$ at center (MPa)</th>
<th>FEM $\sigma_{xx}$ at center (MPa)</th>
<th>Numerical max.$(\sigma_G)$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>42.86</td>
<td>42.7</td>
<td>46.73 at $y/r = 0.9$</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>42.45</td>
<td>42.19</td>
<td>48.97 at $y/r = 0.8$</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>41.77</td>
<td>41.51</td>
<td>41.96 at $y/r = 0.6$</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>41.60</td>
<td>41.36</td>
<td>41.41 at $y/r = 0$</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>41.42</td>
<td>41.2</td>
<td>41.24 at $y/r = 0$</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>40.82</td>
<td>40.66</td>
<td>40.73 at $y/r = 0$</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 14. The equivalent stress $\sigma_G$ as a function of the normalized distance $y/R$ with different contact angles $2\varphi$.

It is also observed in Fig. 14 that except for regions near the contact zone, the tensile stress was constant along the $y$-axis of the load. However, the compressive and shear stresses were considerably reduced in this zone. To get a constant amplitude of the tensile stress, the contact must guarantee that a maximum length along the diameter of the load is subjected to a constant tensile stress. It is associated with minimum values of the shear and compression, lower than the load, otherwise the disc is fractured into two pieces along its diameter.

Conclusion
The tensile strength of the 60HAP/$\beta$-TCP biocomposite was determined experimentally and modelled using the diametral compression test. Mechanical strength of the composite was obtained from the Brazilian test. Evolution of the diametral strength was investigated as a function of the sintering temperature. It results that the association of HAP with $\beta$-TCP improves the mechanical resistance of the 60HAP/$\beta$-TCP bioceramic. It reaches 43 MPa at 1250°C, which is way larger than obtained for HAP and $\beta$-TCP. Distribution of the principal stress fields in the 60HAP/$\beta$-TCP bioceramic was determined by the finite element method. It reveals that the tensile stress is constant along the diameter of the load and generates a crack perpendicular to the $x$-axis, thus breaking the sample into two pieces. The experimental, numerical and analytical results all agree together. We conclude that the 60HAP/$\beta$-TCP bioceramic can be used in prosthetic applications owing to its similarity in size, structure and chemical composition with hard tissue.
Contribution statement: M. Es-saddik, Conception, Methodology, and Formal analysis; S. Laasri, Software, Writing, Original draft preparation; A. Laghzizil, Visualization, Writing and Supervision. J-M Nunzi, Conception, Validation, Writing and Editing; M. Taha, Conception, Resources; A. Guidara, Characterization, Investigation; A. Hajjaji, Project administration, Data processing; J. Bouaziz, Characterization and Investigation.

References


