

when  $H_3PO_4$  is applied for 60 s, thus creating the possibility of a faster degradation of the hybrid layer over time. Longer observation times are needed to validate these *in vitro* results and better clarify the role of extended etching time on radicular dentin.

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### Mechanical properties of Y:TZP/TiO<sub>2</sub> coated with hydroxyapatite for dental implants



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**Purpose/aim:** (1) To investigate the effect of titania content in mol% (0, 10 and 30) in the specific surface area (SBET), the size of the agglomerates and the intensity of the peaks corresponding to crystalline phase present in the ceramic powder of Y:TZP/TiO<sub>2</sub>; (2) to investigate the effect of titania addition (0, 10 and 30 mol%) on the microstructure, relative density (RD), Young's modulus (*E*), Poisson's ratio (PR), flexural strength ( $\sigma_f$ ) and Weibull modulus (*m*) of a Y:TZP/TiO<sub>2</sub> composite; and (3) to investigate the effect of the presence of a biomimetic coating on the microstructure and mechanical properties ( $\sigma_f$  and *m*) of Y:TZP/TiO<sub>2</sub> composite.

**Materials and methods:** Y:TZP (3 mol% of yttria) and Y:TZP/TiO<sub>2</sub> composite (10 or 30 mol% of titania) were synthesized using a co-precipitation route. The powders were pressed and sintered at 1400 °C/2 h. Half of the discs were subjected to biomimetic coating. The powders were characterized by X-ray diffraction (XRD), laser scattering, gas adsorption and scanning electron microscopy (SEM). The surfaces, with and without biomimetic coating, were characterized by SEM and XRD. The RD was measured by the Archimedes' principle. *E* and PR were measured by ultrasonic pulse-echo method. For the mechanical properties the specimens (*n* = 30 for each group) were tested in a universal testing machine. Weibull modulus was estimated by the maximum likelihood method and ANOVA with Tukey test ( $\alpha$  = 5%) was used to evaluate  $\sigma_f$ , *E* and RD.

**Results:** All powders had a SBET greater than 42 m<sup>2</sup>/g and the titania addition favored the formation of larger agglomerates. Titania addition increased the grain size of the composite and caused a significant decrease in the  $\sigma_f$  in MPa (control:

815.4a; T10: 455.7b and T30: 336.0c), *E* in GPa (control: 213.4a; T10: 155.8b and T30: 134.0c) and relative density in % (control: 99.0a; T10: 94.4c and T30: 96.3b) of the Y:TZP/TiO<sub>2</sub> composite. The presence of 30% titania caused substantial increase in *m* and PR. Biomimetic coating resulted in the formation of apatite globules heterogeneously distributed on the surface of the material and this treatment did not significantly alter the  $\sigma_f$  and *m* of the composite (Table 1).

**Conclusions:** The Y:TZP/TiO<sub>2</sub> composite coated with a layer of CaP has great potential to be used as implant material. Properties of the powder were affected by titania addition. Addition of titania to Y:TZP caused an increase in grain size, a significant decrease in  $\sigma_f$ , *E* and RD. The presence of biomimetic coating did not jeopardize the reliability of Y:TZP/TiO<sub>2</sub> composite.

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### Antibacterial activity and flexural strength for experimental composites with graphene



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**Purpose/aim:** The aim of this study was to develop composite materials with antibacterial activity using nanopowders of graphene oxide (GO-SiO<sub>2</sub> and GO-ZrO<sub>2</sub> synthesized in INCD-TIM laboratory) in difference concentration. In this study was to evaluate the antibacterial activity, elastic modulus (*E*) and flexural strength (FS).

**Materials and methods:** Five experimental composites were made with organic matrix of BisGMA (synthesized in our laboratory)/TEGDMA (Aldrich) and inorganic phase: (HA-SiO<sub>2</sub>, barium glass, GO-SiO<sub>2</sub>: 1%, 0.8% – for GS1, GS2; and HA-Zr, barium glass, GO-ZrO<sub>2</sub>: 1%, 0.8% for GZ1, GZ2) and GC (HA-Zr, HA-SiO<sub>2</sub>, colloidal silica, barium glass) composite without graphene, in 20/80 proportions of weight. Specimens for flexural properties (25 mm × 2 mm × 2 mm) were half immediate tested (24 h) and half storage (1 month). Elastic modulus (*E*) and flexural strength (FS) was measured using the three point bending test (*n* = 10), in accordance with ISO 4029/2000 (Lloyd Tools- LR5k Plus). The antimicrobial activity materials were evaluated using the agar diffusion method. The bacterial

**Table 1 – Mean ± standard deviation (coefficient of variation) of flexural strength ( $\sigma_f$ ) and Weibull modulus (95% confidence interval) (*m*). Values followed by the same letter are statistically similar (*p* > 0.05).**

Titania content (mol%)	Flexural strength ( $\sigma_f$ ) (MPa)		Weibull modulus ( <i>m</i> )	
	Biomimetic coating		Biomimetic coating	
	No	Yes	No	Yes
0	815.4 ± 145.1 <sup>a</sup> (18%)	763.6 ± 144.2 <sup>a</sup> (19%)	6.4 <sup>b,c</sup> (4.7–8.6)	5.4 <sup>c</sup> (3.9–7.4)
10	455.7 ± 48.4 <sup>b</sup> (11%)	439.4 ± 65.4 <sup>b</sup> (15%)	10.5 <sup>a,b</sup> (7.7–14.1)	8.7 <sup>a,b,c</sup> (6.3–11.7)
30	336.0 ± 38.7 <sup>c</sup> (11%)	334.2 ± 43.6 <sup>c</sup> (13%)	11.7 <sup>a</sup> (8.6–15.8)	9.9 <sup>a,b,c</sup> (7.3–13.4)