UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

ANA PAULA RODRIGUES DE MAGALHÃES CHAVES

A study of the modification of Y-TZP and resin cement with titanium dioxide nanotubes

Estudo da modificação de Y-TZP e cimento resinoso com nanotubos de dióxido de titânio

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Tese apresentada a Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Doutor em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Dentística.

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“We’re gonna get it, get it together

I know we’re gonna get it, get it together somehow

We’re gonna get it, get it together and flower

We’re gonna get it, get it together

I know, we’re gonna get it, get it together and float

We’re gonna get it, get it together and go

Up, and up, and up.”

Coldplay

RESUMO

Estudo da modificação de Y-TZP e cimento resinoso com nanotubos de dióxido de titânio

Nanotubos de dióxido de titânio (TiO_2) tem sido utilizados para melhorar as propriedades mecânicas de materiais odontológicos. Zircônica tetragonal policristalina estabilizada por ítria (Y-TZP) tem sido amplamente utilizada na Odontologia. Apesar de seus excelentes resultados clínicos, a Y-TZP é suscetível a falhas relacionadas à espessura insuficiente do conector da prótese fixa e a soltura da restauração devido à adesão deficiente. O objetivo desse trabalho é avaliar o efeito da adição de diferentes concentrações de nanotubos de TiO_2 à Y-TZP nas suas propriedades mecânicas e microestrutura, além de avaliar a influência na resistência de união da adição desses nanotubos na cerâmica ou no cimento resinoso. Para isso, os objetivos descritos foram divididos em dois artigos diferentes. O artigo 1 descreve os testes de resistência flexural biaxial, análise fractográfica qualitativa em microscopia eletrônica de varredura (MEV), avaliação de microestrutura em MEV de emissão de campo e difração de raios-X. Os grupos avaliados foram: Y-TZP comercial (Ivoclar Vivadent) (ZC) e Y-TZP experimental com diferentes concentrações de nanotubos [0 (Z0), 1 (Z1), 2 (Z2), e 5% (Z5), em volume]. No artigo 2 está descrito o teste de resistência ao cisalhamento que foi realizado com os seguintes grupos: Y-TZP comercial (ZC) e Y-TZP experimental com diferentes concentrações de nanotubos [0 (Z0), 1 (Z1), 2 (Z2), e 5% (Z5), em volume] aderidas ao cimento Panavia F2.0; e Y-TZP comercial aderida ao cimento resinoso RelyX U200 com adição de diferentes concentrações de nanotubos em dois métodos de polimerização: dual [0 (DC), 0,3 (D03), 0,6 (D06) e 0,9% (D09) de nanotubos em peso] ou auto [0 (SC), 0,3 (S03), 0,6 (S06) e 0,9% (S09) de nanotubos em peso]. Os valores de resistência flexural e resistência ao cisalhamento foram submetidos aos testes de ANOVA e Tukey (α=0,05). A resistência flexural também passou por análise de Weibull. Os valores de tamanho de grãos foram submetidos a testes de Kruskal-Wallis e Dunn (α=0,05). Os resultados de resistência flexural encontrados foram: ZC – 896,73±122,70; Z0 – 577,67±62,26; Z1 – 477,32±75,65; Z2 – 492,25±63,19; Z5 – 437,18±53,55. Os resultados de módulo de Weibull encontrados foram: ZC - 7,9; Z0 - 11,2; Z1 - 8,7; Z2

- 8,1; Z5 - 9,3. Os resultados mostraram que a Y-TZP experimental apresentou menores valores de resistência flexural do que a cerâmica comercial, mas a primeira apresentou maior módulo de Weibull (*m*). A Y-TZP experimental apresentou boa microestrutura, comparável à Y-TZP comercial, com tamanhos de grão muito semelhantes. A adição de nanotubos à Y-TZP levou a menor resistência flexural, porém maior *m* que a cerâmica comercial. Poros contendo Ti foram observados na Y-TZP conforme a concentração de nanotubos aumentou. Os resultados de resistência ao cisalhamento foram, do maior para o menor: Z5 - 6,46±3,36; DC - 6,17±0,87; D03 - 5,74±1,70; S03 - 5,73±1,71; Z1 - 5,16±2,62; D06 - 4,82±1,06; D09 - 4,75±1,43; SC - 4,73±1,43; S09 - 4,61±0,85; S06 - 4,51±1,87; ZC - 3,70±1,82; Z0 - 3,33±2,05; Z2 - 2,94±1,38. A resistência ao cisalhamento também foi influenciada pela adição de nanotubos, tanto na cerâmica quanto no cimento, porém não linearmente. A Y-TZP adicionada de 5% de nanotubos de TiO_2 apresentou maior resistência de união, porém sem diferença estatística da maioria dos grupos. O grupo Z1 foi provavelmente o grupo que apresentou a melhor combinação de resistência flexural, *m*, microestrutura e resistência de união. Mais estudos de outras propriedades podem ser realizados com o mesmo.

Palavras-chave: Nanotubos. Cerâmica. Zircônio. Titânio.

ABSTRACT

A study of the modification of Y-TZP and resin cement with titanium dioxide nanotubes

Titanium dioxide nanotubes (TiO_2) have been applied to enhance the mechanical properties of dental materials. Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) has been increasingly used in dentistry. Aside from its optimal clinical results, Y-TZP is prone to failures related to insufficient thickness of the fixed prostheses connector and debonding due to its difficult adhesion. The purpose of this study is to evaluate the effect of the addition of different concentrations of TiO_2 to Y-TZP in its mechanical properties and microstructure, and also to evaluate the influence of these nanotubes on the bond strength when added to the ceramic or to the resin cement. To evaluate that, the described purposes were divided in two different papers. Paper 1 describes the tests of biaxial flexural strength, fractography qualitative analysis in scanning electron microscopy (SEM), microstructure evaluation in field emission-SEM and X-ray diffraction. Groups evaluated were commercial Y-TZP (Ivoclar Vivadent) (ZC) and an experimental Y-TZP with different blends of nanotubes [0 (Z0), 1 (Z1), 2 (Z2), and 5% (Z5), in volume]. In paper 2 shear bond strength test is described. It was carried out with the following groups: commercial Y-TZP (Ivoclar Vivadent) (ZC) and an experimental Y-TZP with different blends of nanotubes [0 (Z0), 1 (Z1), 2 (Z2), and 5% (Z5), in volume] bonded to the resin cement Panavia F2.0; and commercial Y-TZP bonded to resin cement RelyX U200 added with different blends of nanotubes in two curing methods dual-cured [0 (DC), 0.3 (D03), 0.6 (D06) and 0.9% (D09) of nanotubes in weigth] or self-cured [0 (SC), 0.3 (S03), 0.6 (S06) and 0.9% (S09) of nanotubes in weigth]. Values of flexural strength and shear bond strength were subjected to ANOVA and Tukey (α=0.05). Flexural strength values were also subjected to Weibull statistics. Grain sizes values were subjected to Kruskal-Wallis and Dunn tests (α=0.05). The flexural strength results were: ZC – 896.73±122.70; Z0 – 577.67±62.26; Z1 – 477.32±75.65; Z2 – 492.25±63.19; Z5 – 437.18±53.55. The Weibull modulus results found were: ZC – 7.9; Z0 – 11.2, Z1 – 8.7; Z2 – 8.1; Z5 – 9.3. Results showed that experimental Y-TZP presented lower flexural strength values than commercial one, but the first presented better Weibull modulus (m). Experimental Y-TZP also presented good microstructure, comparable to commercial Y-TZP, with very similar grain sizes. Nanotubes addition to Y-TZP led to lower flexural strength, although higher *m* than commercial ceramic. Pores containing Ti were observed in Y-TZP as the nanotubes concentration raised. Shear bond strength results found were, from higher to lower values: Z5 – 6.46±3.36; DC – 6.17±0.87; D03 – 5.74±1.70; S03 – 5.73±1.71; Z1 – 5.16±2.62; D06 – 4.82±1.06; D09 – 4.75±1.43; SC – 4.73±1.43; S09 – 4.61±0.85; S06 – 4.51±1.87; ZC – 3.70±1.82; Z0 -3.33±2.05; Z2 – 2.94±1.38. Shear bond strength was also influenced by nanotubes addition, either in the ceramic or in the cement, although not linearly. Y-TZP added of 5% of TiO_2 nanotubes presented the highest bond strength, although with no significant difference from most groups. Group Z1 was probably the group that presented the best combination of flexural strength, *m*, microstructure and bond strength. More studies of other properties could be carried out with this group.

Key-words: Nanotubes. Ceramic. Zirconium. Titanium.

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LIST OF ABBREVIATIONS AND ACRONYMS

10-MDP	10-Methacryloyloxydecyl dihydrogen phosphate
Å	Ångström
ANOVA	Variance Analysis
Bis-GMA	Bisphenol A-glycidyl methacrylate
$C_{18}H_{34}O_2$	Oleic oil
CAD-CAM	Computer Aided Design – Computer Aided Manufacturing
CaO	Calcium oxide
cc	Compression curls
CeO_2	Cerium oxide
cm	Centimeters
DC	Dual-cured RelyX U200 without nanotubes
D0.3	Dual-cured RelyX U200 with 0.3% of nanotubes
D0.6	Dual-cured RelyX U200 with 0.6% of nanotubes
D0.9	Dual-cured RelyX U200 with 0.9% of nanotubes
DC	Dual-cured RelyX U200 without nanotubes
EDX	Energy Dispersive X-Ray Spectroscopy
FAPESP	Fundação de Amparo à Pesquisa do Estado de São Paulo
FE-SEM	Field Emission Scanning Electron Microscopy
g	Grams
h	Hours
HCl	Chloridric acid
hl	Hackle lines
ISO	International Organization for Standardization
kN	Kilonewtons
kV	Kilovolts
LED	Light-Emitting Diode
m	Meters
m	Weibull modulus
M	Molar
m	Mirror region
mA	Miliampere
MEV	Microscopia Eletrônica de Varredura

MgO	Magnesium oxide
min	Minutes
ml	Mililiters
mm	Milimeters
MPa	Megapascal
NaOH	Sodium hydroxide
nm	Nanometers
^{o}C	Celsius degrees
p	Probability value
p	Pores
PABA	4-amino benzoic acid
PMMA	Polymethyl methacrylate
Psi	Pounds per square inch
PVB	Polyvinyl butyral
PVC	Polyvinyl chloride
s	Seconds
SC	Self-cured RelyX U200 without nanotubes
S0.3	Self-cured RelyX U200 with 0.3% of nanotubes
S0.6	Self-cured RelyX U200 with 0.6% of nanotubes
S0.9	Self-cured RelyX U200 with 0.9% of nanotubes
SC	Self-cured RelyX U200 without nanotubes
SEM	Scanning Electron Microscopy
Ti	Titanium
TiO_2	Titanium dioxide
wh	Wake hackle lines
XDR	X-ray diffraction
Y-TZP	Yttria-stabilized tetragonal zirconia polycrystals
Y_2O_3	Yttrium oxide
Z0	Experimental zirconia without nanotubes group
Z1	Experimental zirconia with 1% of nanotubes group
Z2	Experimental zirconia with 2% of nanotubes group
Z5	Experimental zirconia with 5% of nanotubes group
ZC	Commercial zirconia group
Zr	Zirconia

ZrO_2	Zirconium dioxide
α	Significance level
μm	Micrometers
σ	Biaxial flexural strength
σ_0	Characteristic strength

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1 Introduction

1 INTRODUCTION

Ceramics have been used in dentistry more in the recent years and their types and applications are constantly growing. Ceramics generally are inorganic, nonmetallic solids synthesized by heat treatment and subsequent cooling (CALLISTER JR; RETHWISCH, 2007; GAUTAM et al., 2016). Nowadays the word “ceramic” refers to materials such as glass, advanced ceramics and cement systems as well (GAUTAM et al., 2016), leading to a much broader meaning. High-strength oxide ceramic materials, such as zirconium dioxide (ZrO_2), have many different applications such as extrusion dyes, valves and port liners for combustion engines, low corrosion and thermal shock resistant refractory liners, valve parts in foundries, blades to cut Kevlar, etc (PICONI; MACCAURO, 1999).

Zirconia, as the zirconium dioxide (ZrO_2) is known, was identified in 1789 by the German chemist Martin Heinrich Klaproth in the reaction product obtained after heating some gems, and was used for a long time blended with rare earth oxides as pigment for ceramics (PICONI; MACCAURO, 1999). At ambient pressure, zirconia can assume three different crystallographic forms depending on the temperature: cubic, monoclinic and tetragonal forms. When zirconia is mixed with various stabilizing oxides such as CaO, MgO, Y_2O_3 or CeO_2 the tetragonal structure is maintained at room temperature (GAUTAM et al., 2016). When a crack starts in the material, the tetragonal grains are able to transform into monoclinic ones and the resultant volume increase inhibits the crack from further growth and propagation (LARSON et al., 2007).

Zirconia stabilized with an addition of yttrium oxide (Y-TZP) becomes a high strength ceramic with enhanced mechanical properties and higher biological stability (DERAND et al., 2005; GAUTAM et al., 2016). These characteristics have made it suitable to use in orthopedic prostheses, and also made it interesting as a restorative material in the dental clinic. The advantages of Y-TZP in combination with the development of new processing techniques have made zirconia an interesting material in the production of metal-free crowns and bridges since 1998, and recently in the nineties of more esthetic endosseous implants (DERAND et al., 2005; DENRY; KELLY, 2008).

Some properties have made Y-TZP such an atractive material for dentistry like flexural strength in the 800-1000MPa range and fracture toughness in the 6-

8MPa $m^{0.5}$ range. The Weibull modulus can vary more as it is strongly dependent on the type of surface finish and the processing conditions (DENRY; KELLY, 2008). All-ceramic fixed partial dentures use has increased significantly in the last decade, specially due to the CAD-CAM system introduction, facilitating and speeding the proccess (LARSON et al., 2007). The development of new ceramic-based implants is based on their enhanced capacity of osseointegration, less plaque accumulation leading to an improvement of the soft tissue management, and aesthetic consideration as an alternative to monolithic titanium implants (HISBERGUES et al., 2009; CHEN et al., 2014).

Although, even if we consider the optimal clinical results obtained with Y-TZP (SAILER et al., 2007; SORRENTINO et al., 2012) a few characteristics still limit its use in some applications. A literature review showed that most fractures of Y-TZP dental restorations involve connectors of multi-unit prostheses (≥4) or second molar abutments (DENRY; KELLY, 2008). Limiting the use of Y-TZP to certain situations, where certain thickness ratios or framework designs can be obtained in order to control the porcelain cracking development (DENRY; KELLY, 2008). Even though, Y-TZP is a very hard material, it is also brittle, and that makes its strength dependent on the presence of surface irregularities or porosities and internal voids formed during its manufacture proccess (MCCABE; WALLS, 2008; VOLPATO et al., 2011).

Also Y-TZP's adhesion to resin luting cements is one of its drawbacks. Researches present the need of specific surface treatments to obtain enhanced bond strength, although no agreement exists yet in the literature (AMARAL et al., 2014; ERDEM et al., 2014; LIU et al., 2015). Different treatment for the polycrystalline ceramics involving the most different procedures are suggested: particle air abrasion (ERDEM et al., 2014; AHN et al., 2015), zirconia primers (CHEN et al., 2014), laser treatment (ERDEM et al., 2014; KASRAEI et al., 2014), plasma treatment (DERAND et al., 2005), and tribochemical treatment, that consists of silica coating of the surface followed by silanization (CHEN et al., 2014; ERDEM et al., 2014).

Cements and adhesives containing 10-MDP (10-methacryloyloxi-decyl-dihydrogen-phosphate) have also been indicated to enhance Y-TZP's adhesion (AHN et al., 2015; ÖZCAN; BERNASCONI, 2015; ALVES et al., 2016; XIE et al., 2016). 10-MDP has bifunctional ends in its structural formula; one is a hydrophilic phosphate ester group that bonds strongly to Y-TZP. Other is a vinyl group that copolymerize with other monomers in the adhesive, leading to bifunctional direct adhesion between metal oxides and bis-GMA matrices (KOIZUMI et al., 2012; AHN et al., 2015). It is of utmost importance to select the best combination of surface treatment and resin cement in order to assure the clinical survival of Y-TZP restorations.

Nanotechnology is developing in an increasing speed with potential to become an essential element in every field. TiO_2 nanostructures have been the subject of recent researches, since their synthesis until their practical applications, like photocatalysis, solarcells, hydrogen storage and nanobiomaterials. This variety of applications is due to their chemical stability, antimicrobial properties, mechanical properties, non-toxicity and high refractive index (AI et al., 2011; ARRUDA et al., 2014). Nanotubes present big length, high area-volume ratio with a hollow structure that enables aditional interlocking with the material, increasing its physical and mechanical properties (CUI et al., 2012; KHALED et al., 2012).

In dentistry, TiO_2 nanostructures have been applied in some studies for the last years (KHALED et al., 2010; SUN et al., 2011; CUI et al., 2012; GJORGIEVSKA et al., 2015; DAFAR et al., 2016). When incorporated to flowable dental composites, the TiO_2 nanotubes improved the dynamic Young's modulus and fracture toughness with minimum decrease in flowability and radiopacity (DAFAR et al., 2016). Glass-ionomer cements reinforced with TiO_2 nanoparticles presented improved biocompatibility and mechanical properties (GJORGIEVSKA et al., 2015). A resin based cement presented higher mechanical properties with 1% of TiO_2 nanotubes, without altering the handling properties, radiopacity and cytocompatibility of the material (KHALED et al., 2010). TiO_2 nanotube layers with Ti biocomposites as a dental implant material presented very good antibacterial activity to resist *Streptococcus mutans* with wider application prospects (CUI et al., 2012). The addition of a small amount of TiO_2 to a dental resin improved dramatically its degree of conversion and the mechanical properties (SUN et al., 2011).

The interaction of TiO_2 nanotubes and zirconia, to our knowledge, has not yet been studied, so, some hypotheses were raised. The TiO_2 nanostructures were able to enhance mechanical properties when blended to other dental materials, so the possibility to enhance Y-TZP flexural strength and its clinical reliability could be an interesting finding for its application as a restorative material or as endosseous implants. Even though zirconia is a chemically inert material (BASU, 2005), controlling all manufacture steps and adding the nanotubes on the ceramic powder may be an interesting method to control its microstructure and hence, its mechanical behavior. Also the possible interaction between the TiO_2 nanotubes and the 10-MDP on the resin cement could lead to better bond strength between luting cement and Y-TZP

4 Discussion

4 **DISCUSSION**

The microstructural characteristics have been a key issue in the development of high toughness ceramics, like Y-TZPs, due to their important influence on the bulk properties and dependence on the manufacture process. Literature available has already determined that a successful manufacture process needs to be controlled in each step, from the synthesis of the powders, to their processing and sintering, in order to controll the grains size, phase content and sintered density (PALMERO, 2015; LI et al., 2003; BASU, 2005). This highlights the importance of controlling the microstructure characteristics during processing and sintering, in order to maximize the mechanical properties (PALMERO, 2015; LI et al., 2003).

The goal of research over recent decades has been to produce ceramics having an optimum combination of high toughness and high strength. Basu (2005) classifies toughening mechanisms in ceramics in two categories: one involves a process that develops around the crack tip (transformation toughening); the other is associated with reinforcements, like nanotubes. The growing interest in using nanotubes, nanofibers and whiskers is explained by the local toughening effect offered by their elongated form, able to provide crack bridging and deflection mechanisms (BASU, 2005; PALMERO, 2015).

Flexural strength is generally considered an important mechanical property for brittle materials that are much weaker in tension than in compression. It aids in predicting the performance of fragile materials (VOLPATO et al., 2011; BONA; ANUSAVICE; DEHOFF, 2003). Biaxial flexural strength test has several advantages over 3-point and 4-point flexure tests, as production of multi-axial stress states, elimination of edge failures and being more indicated for Weibull statistics (XU et al., 2015).

Flexural strength results showed that the commercial Y-TZP presented higher strength than the other groups. Nanotubes addition made the strength of the experimental ceramic decrease, but not linearly. Considering the pure Y-TZP groups, both materials have very similar compositions with very close amounts of zirconia, yttria and other oxides (manufacturer informations). Probably the different results for flexural strength can be explained by the differences in the manufacture process. ZC was produced industrially and latter was cut to the shape necessary for the biaxial flexural test. Z0 was produced manually and then conformed to the desired shape.

Ceramic strength values typically fall within an asymmetrical distribution when compared to metals that exhibit a Gaussian (symmetrical) strength distribution (BONA; ANUSAVICE; DEHOFF, 2003). The *m* and characteristic strength are commonly used statistics parameters to describe structural reliability of ceramics. The characteristic strength or scale parameter represents the probability of failure of 63.2% of a particular test specimen and loading configuration, it corresponds to the mean value for a material with Gaussian strength distribution. The *m* is the shape parameter and describes the asymmetrical strength distribution, corresponding to the standard deviation (ISO 6872:2015). So, *m* is used to illustrate the variation of the strength or asymmetrical strength distribution due to flaws and micro-cracks size distribution in the microstructure.

Most ceramics are reported to have *m* values in the range of 5-15, while metals, present values of *m* in the range of 30-100 (BONA; ANUSAVICE; DEHOFF, 2003). Higher values of *m* correspond to materials with greater structural reliability, indicates a smaller error range, and potentially, greater reliability under clinical conditions (BONA; ANUSAVICE; DEHOFF, 2003; ELSAKA; ELNAGHY, 2016). A lower *m* value may indicate greater variability in defect population size and lower reliability (ELSAKA; ELNAGHY, 2016).

Contrary to the flexural strength, the *m* found for Z0 was higher than that of ZC. When TiO_2 nanotubes were added, *m* for experimental ceramic decreased, although these values were still higher than the one found for ZC. High *m* materials are more predictable and less likely to break at a stress much lower than the mean value found for flexural strength (QUINN; QUINN, 2010).

Fractography is the study of the fracture surfaces of a material and is well-established as a mean of failure analysis in the field of glasses and ceramics, representing an important tool for interpreting the mechanical behavior of ceramic materials (BONA; ANUSAVICE, DEHOFF, 2003; RAMOS et al., 2016). In the Y-TZP specimens evaluated it was not easy to identify the tell-tale marks, making it harder to understand the material behavior. As more nanotubes were added, less tell-tale marks were visible and it was harder to identify a possible fracture origin, due to the presence of pores.

Usually, fracture occurs when pre-existing cracks, induced by mechanical means, by processing, or by intrinsic defects and imperfections, propagate under excessive tensile stresses (BONA; ANUSAVICE; DEHOFF, 2003). According to “weakest link theory”, flexural strength is strongly influenced by variations in specimens flaw types and sizes and fracture happens as soon as the weakest of these flaws starts to grow (XU et al., 2015). Thus, the fracture origin is the site that presented the worst combination of tensile stress and flaw severity (QUINN, 2016). In the specimens analyzed for fractography most flaws observed were pores probably introduced in processing, as no mechanical means as griding and polishing were used in the specimens production. These pores were more evident as the nanotubes concentration was augmented, probably associated with the Ti melting due to the high sintering temperature, leading to lower fracture strength in the groups with nanotubes (QUINN; QUINN, 2010). Althoug, this pore formation induced by manufacture control made the *m* higher, creating a more reliable Y-TZP structure.

Grain sizes in the Y-TZP can be controlled by some manufacture steps like sintering temperature; higher sintering temperatures generates bigger grains (STAWARCZYK et al., 2016). Higher concentration of oxides can also lead to grain growth (BASU, 2005), adding TiO_2 increases oxide concentration in the Y-TZP, leading to bigger grains, what can be observed in this work results. A zirconia with smaller grain sizes is desireble. Ceramics with bigger grains may present more structural deffects, which may affect the flexural strength (STAWARCZYK et al., 2016). Also an increased light transmittance is achieved with grain sizes below the wavelength of visible light, resulting in higher translucency and improved aesthetics (COTIC et al., 2015; ZHANG, 2014). In this study, grains were slightly bigger with TiO_2 addition, althoug there was no statistical significance and they were still smaller than the ISO 13356:2008 standard recommendation (smaller than 400nm).

Zirconia’s three crystal structures are: monoclinic (from room temperature to 1170°C), tetragonal (from 1170-2370°C) and cubic (above 2370°C). They differ from each other in geometry and dimensional parameters. The transformation of tetragonal to monoclinic phases can be employed to improve the mechanical properties of zirconia, especially its tenacity. The addition of stabilizing oxides such as yttrium oxide (Y_2O_3), calcium oxide (CaO) or magnesium oxide (MgO) is important because it allows the maintenance of the tetragonal form at room temperature (VOLPATO et al., 2011; LI et al., 2003). Mechanical properties degradation in zirconia, known as low temperature degradation, occurs due to the progressive spontaneous transformation of the metastable tetragonal phase into the monoclinic phase (PICONI; MACCAURO, 1999).

One of the primary uses of x-ray diffractometry is for the determination of crystal structure (CALLISTER JR; RETHWISCH, 2014). X-ray diffraction permits a quantitative measurement of the monoclinic fraction, but it is limited to the first hundredth of a micrometer below the surface (RAMOS et al., 2015). The results obtained with the Y-TZPs studied showed that ZC and Z0 presented similar crystal structures, and the nanotubes addition did not lead to important changes in this structure. A small increase in monoclinic composition can be osberved althoug, it is probably not enough to impair the transformation toughening mechanism in the long-term.

Bond strength is a major concern in Y-TZP restorations (CHO et al., 2017; AHN et al., 2015; OZCAN; BERNASCONI, 2015). The difficult adhesion of this material usually narrows its indications to situations where a preparation with mechanical retentions can be obtained (ALVES et al., 2015). Many procedures and materials have been studied for surface treatment of Y-TZP restorations in order to achieve enhanced bonding, but most of them rely on new advanced technologies and some on chair-side more simple solutions (AHN, et al., 2015; ALVES et al., 2015; CHEN et al., 2014; CHO et al., 2017; DERAND; MOLIN; KVAM et al., 2005; ERDEM et al., 2014). Finding the treatment or material with best cost-benefit relation is imperative to make the clinical procedure easier and less sensitive to clinical variations.

The study of the bond strength can be carried out through different methods. According to DeHoff, Anusavice and Wang (1995), due to the known variation in bond strength with specimen preparation and design, data on the same systems may show great variability in mean and large standard deviations. Thus, it is advised that these tests should be used to compare materials or surface treatments, and try to determine the effect of changing some variable for the same system, and not to determine the real bond strength (DEHOFF; ANUSAVICE; WANG, 1995). In this study the variables studied for bond strength were the TiO_2 nanotubes addition in the Y-TZP or in the cement, the different concentrations and the two curing modes.

The TiO_2 nanotubes addition influenced the bond strength in both materials, but it did not present a linear influence. The dual-cured RelyX U200 had its bond strength lower with the nanotubes concentration increase. However, the self-cured cement bond strength was higher with 0.3% of nanotubes. This could mean that 0.3% is the optimal concentration of TiO_2 in the cement, and it led to a better distribution of the nanotubes in the cement mass. Another study showed that the TiO_2 nanotubes reinforced RelyX U200 showed better physicochemical and mechanical properties than the ones not reinforced (RAMOS-TONELLO et al., 2017), but the same relation was not observed for bond strength. Ramos-Tonello et al also showed that the self-cured cement had increased degree of conversion with nanotubes addition. The cure-mode did not influence much on the bond strength, although the dual-cured cements usually performed better in adhesion than the self-cured ones, as described in a previous study (ZORZIN et al., 2014).

For the Y-TZP, the highest bond strength was registered for the 5% group, followed by 1%, 0% and 2%. This not linear variation could also be explained by the nanotubes distribution. Probably the 5% concentration led to a higher amount of nanotubes in the interface, which could react with the 10-MDP present in Panavia F2.0 cement and lead to higher bond strength.

The monomer 10-MDP is part of Panavia F2.0 compostion and it is responsible for a higher bond strength when this cement is used for Y-TZP cementation (CHO et al., 2017; ERDEM et al., 2014). One of its bifunctional ends reacts directly with the oxides present on the zirconia surface, leading to a chemical bond between these two materials (OZCAN; BERNASCONI, 2015; TANIS; AKÇABOY, 2015). RelyX U200 contains other phosphate monomers, not identified as 10-MDP, but that might also have some kind of chemical reaction with Y-TZP, as observed for other cement with similar composition of the same manufacturer, RelyX Ultimate (ZHAO et al., 2016). Both cements studied are known to have good adhesion to zirconia (VECHIATTO-FILHO et al., 2017; ALVES et al., 2015), but Panavia F2.0 has a longer history for this application (ERDEM et al., 2014; SHIN et al., 2014; SUBASI; INAN, 2014).

The results for bond strength were not comparable to the mechanical tests done before with Y-TZP added of TiO_2 nanotubes, where the best results were usually found for the groups with less nanotubes. Probably Z1 was the group that presented the best combination of flexural strength, *m*, microstructure and bond strength, and could be further studied for other characteristics, such as biocompatibility and citotoxicity. A zirconia with higher structural reliability and bond strength, but with lower flexural strength, could be more interesting to the clinical practice than the strongest ceramic, with less adhesion and less reliability.

Also other studies with TiO_2 application in other nanoforms, as nanoparticles for example, could be carried out in order to see its influence on the dispersion and properties of zirconia. As Y-TZP is an inert material (BASU, 2015), probably functionalization of the particles or tubes with 10-MDP, for example, could be studied as a method to functionalize the nanostructures. This could avoid agglomeration (AI et al., 2011) and create a chemical interaction between the particles and the ceramic oxides, leading to different results.

5 Conclusions

5 CONCLUSIONS

A Y-TZP added or not of TiO_2 nanotubes in different blends was successfully manufactured and some properties were evaluated. Considering the initial hypotheses, the addition of different concentrations of TiO_2 nanotubes influenced Y-TZP's mechanical properties, as the nanotubes addtion led to lower flexural strength than the commercial and experimental ceramics. Although, higher *m* values were found for the experimental ceramics, with or without nanotubes, when compared to the commercial Y-TZP, probably resulting in better clinical reliability. The microstructure was also influenced by the nanotubes as pores containing TiO_2 were more evident as their concentration was raised, even though the grain sizes were very similar among groups.

These results might be explained by agglomeration and fusion of the nanotubes due to the high sintering temperature, and also by the ceramic's inertness, leading to small deffects in the specimen body. As Y-TZP is a brittle material, its flexural strength is affected by the amount and size of the deffects, affecting the mechanical properties.

The other hipothesis was related to the bond strength between Y-TZP and resin cement. The addition of TiO_2 nanotubes was carried out in both materials, leading to different results among groups. Y-TZP with 5% of nanotubes presented the highest bond strength for the groups of modified ceramic; and dual-cured cement with 0.3% of nanotubes the highest for the resin cements modified. This may be explained by the amount of Ti available in the materials' surface that could lead to higher influence of these components on the bond between them.

Concentration of 0.3% of nanotubes on the resin cement appeared to be the optimal concentration for both curing modes, when bond strength was analysed. For the Y-TZP, 1% of nanotubes led to best combination of flexural strength, *m*, microstructure and bond strength. More studies of other properties could be carried out with this group.

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Annexes

ANNEX A – Guidelines for authors used in preparation of Paper 1 – Journal Ceramics International

Introduction

Ceramics International primarily deals with the fundamental aspects of ceramic science and their application to the development of improved ceramic materials. The journal particularly encourages papers that show how ceramic science can be used to improve the quality, reliability and performance of ceramics through the development of advanced materials and manufacturing techniques. Fabrication processes which *Ceramics International* concentrates on include all the advanced techniques employed to produce ceramic components of improved quality, reliability and performance. *Ceramics International* is particularly concerned with powder and material processing. Subjects covered in these areas include:

Powder Processing Solid state, chemical precipitation, hydrothermal, vapour phase, laser, plasma and SHS synthesis, polymer and gel processing.

*Processing Methods*Pressing, extrusion, injection moulding, rolling, calendering, electrophoretic deposition, casting, hot pressing (including isostatic), forging, plasma sintering, high pressure, ion-beam and laser processing.

Ceramics International covers process related topics including advanced coatings and sealants, surface finishing and conditioning and composite technology.

Materials covered by the journal include engineering, electrical, electro-optic, magnetic, nuclear and bioceramics, refractories, whiteware and heavy clay based products.

Ceramics International is particularly keen to attract papers which also deal with the science and design aspects of ceramics that are relevant to service performance and which cover the whole range of ceramic products.

Types of paper

Types of contributions: Original papers; review articles; short communications; letters to the editor. Internal laboratory reports are not usually suitable without drastic revision.

Original material: Submission of an article implies that the work described has not been published previously (except in the form of an abstract or as part of a published lecture or academic thesis), that it is not under consideration for publication elsewhere, that its publication is approved by all authors and that, if accepted, it will not be published elsewhere in the same form, in English or in any other language, without the written consent of the Publisher.

Submission checklist

You can use this list to carry out a final check of your submission before you send it to the journal for review. Please check the relevant section in this Guide for Authors for more details.

Ensure that the following items are present:

One author has been designated as the corresponding author with contact details:
• E-mail address
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All necessary files have been uploaded:
Manuscript:
• Include keywords
• All figures (include relevant captions)
• All tables (including titles, description, footnotes)
• Ensure all figure and table citations in the text match the files provided
• Indicate clearly if color should be used for any figures in print
Graphical Abstracts / Highlights files (where applicable)
Supplemental files (where applicable)

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