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Surface roughness and hardness of yttria stabilized zirconia (Y-TZP) after 10 years of simulated brushing

Avaliação da rugosidade e dureza da zircônia Y-TZP após simulação de 10 anos de escovação

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Resumo

Introdução: A zircônia estabilizada por ítria *(Y-TZP*) utilizada para infraestruturas protéticas pode, em algumas situações clínicas, ficar exposta ao meio bucal, e nessas situações, uma superfície sem alterações e polida é extremamente importante. **Objetivo**: Assim, este estudo avaliou a rugosidade média (Ra) e a dureza Vickers da zircônia Lava™ após simulação de dez anos de escovação. **Material e método**: Foram confeccionados 36 espécimes (20mm X 4mm X 1,2mm), divididos em três grupos: armazenamento em água destilada (AD, n=12, controle), escovação com água destilada (EAD, n=12) e escovação com água destilada e dentifrício fluoretado (EDF, n=12). A escovação foi realizada em máquina de escovação simulando 10 anos (878.400 ciclos, 100gf) com escova dental macia. A rugosidade média (Ra, em µm) e a dureza Vickers (VHN) de todos os corpos-de-prova foram mensuradas em dois momentos: antes e após o tratamento experimental, em rugosímetro e microdurômetro (500gf, 30 segundos), respectivamente. Os dados foram analisados por meio do teste *two-way* ANOVA (α=0,05). **Resultado**: A interação entre os grupos não foi significativa tanto para a rugosidade (p=0,701) quanto para a dureza (p=0,928), sendo as médias finais de Ra (µm) iguais a: AD - 0,63; EAD - 0,64 e EDF - 0,68 e as de dureza Vickers (VHN) iguais a: AD - 1301,16; EAD - 1316,60 e EDF - 1299,58. **Conclusão**: Concluiu-se que o procedimento de escovação com água destilada ou com dentifrício fluoretado não foi capaz de alterar a rugosidade e a dureza da zircônia *Y-TZP* utilizada neste estudo.

Descritores: Dureza; escovação dentária; cerâmica.

Abstract

Introduction: The Y-TZP zirconia used for prosthetic infrastructure, in some clinical situations, can be exposed to the oral environment. In these situations, a polished surface without changes is extremely important. **Objective**: The aim of this study was to evaluate the mean roughness (Ra) and Vickers hardness of Y-TZP zirconia (Lava™) after simulating ten years of brushing. **Material and method**: Thirty-six Y-TZP bar-shaped specimens (20mm X 4mm X 1.2mm) were divided into three groups: storage in distilled water (DW, n=12, control); brushing with distilled water (BDW, n=12) and brushing with distilled water and fluoride toothpaste (BFT, n=12). Brushing was performed using a brushing machine with a soft-bristled toothbrush, simulating 10 years of brushing (878.400 cycles, 100gf). The mean roughness (Ra in µm) and Vickers hardness (VHN) of all specimens were measured twice: before and after the experimental treatment, in profilometer and microhardness tester (500gf, 30 seconds), respectively. Data were analyzed using the two-way ANOVA test (α = 0.05). **Result**: The interaction between groups was not significant for roughness (p = 0.701) nor for hardness (p = 0.928). The final averages for Ra (µm) were equal to: DW – 0.63; BDW – 0.64; and, BFT – 0.68. The final averages for Vickers hardness (VHN) were: DW – 1301.16; BDW – 1316.60; and, BFT – 1299.58. **Conclusion:** It was concluded that the brushing with distilled or fluoridated toothpaste was not able to change the roughness and hardness of Y-TZP zirconia used in this study.

Descriptors: Hardness; toothbrushing; ceramics.

INTRODUCTION

Zirconia is a polymorph material that occurs in three phases: monoclinic (M) (from room temperature up to 1170°C), tetragonal (T) (1170°C to 2370°C) and cubic (C) (above 2370°C)[1,2]. It exhibits better performance in the tetragonal form[3]. However, as its stable phase at room temperature is monocyclic, the use of oxides to stabilize zirconia in the tetragonal form at this temperature becomes necessary[1,2]. The addition of stabilizing oxides like CaO, MgO, CeO_2 and Y_2O_3 to pure zirconia allows the creation of metastable materials known as Tetragonal Polycrystalline Zirconia (TZP)[1,2], that are used in different systems[2].

In dentistry, Y-TZP zirconia has been used as the framework of all-ceramic crowns and fixed partial dentures, implants, abutments and brackets[2-4]. Studart et al.[4] demonstrated that Y-TZP zirconia is considered the most suitable material to resist high stress on posterior all-ceramic bridges, being suitable for the manufacture of frameworks having up to five elements.

Among its properties, zirconia exhibits high Vickers hardness around 1300VHN[3,5], and must comply with criterion F1873 of the American Society for Testing and Materials (ASTM), that suggests values above 1200HV[3]. Mean roughness (Ra) values between 0.2 and 0.98μm can be found[6-8]. However, in prolonged contact with a moist environment (water, saliva, blood, synovial fluid), zirconia undergoes a degradation process called aging[3,9-11], that can change its properties[12].

In some clinical situations, such as orthodontic brackets and abutments, zirconia may be exposed in the oral environment. Thus, the material will be in contact with chemical and mechanical agents that may alter its surface and create irregularities[3,9,10,11,13]. These irregularities can serve as shelter for microorganisms, protecting them from forces of salivary flow, chewing, swallowing and oral hygiene[14,15]; favoring microbial colonization; and, possibly leading to failure of the material.

Furthermore, mechanical wear such as abrasion can also contribute to changes in the material surface, directly reflecting on the roughness and accelerating the process of degradation[5,16]. Moreover, zirconia is susceptible to chemical degradation due to its dependence on pH and the concentration of fluoride[17], since alkaline and acid pH values may cause a type of corrosion on the surface of the material[18]; free fluoride in an acid medium can dissolve Y^{3+} ions, forming YF_3 and destabilizing the material[17].

So, the aim of this study was to evaluate the mean roughness (Ra) and Vickers hardness of Y-TZP zirconia (Lava™) after simulating 10 years of brushing with fluoride toothpaste. The null hypothesis was that there would be no change in the surface roughness and/or hardness after 10 years of simulated brushing.

MATERIAL AND METHOD

After a pilot study and considering sample calculation for obtaining power of statistical test equal to 0.80, thirty-six bar-shaped specimens (25 mm X 5mm X 1.5 mm) of tetragonal zirconia polycrystalline, stabilized with yttria 3% mol (Y-TZP) (Lava™ Frame Zirconia, 3M ESPE, Sumaré, São Paulo, Brazil), were cut using a high precision sectioning saw (ISOMET 1000, Buehler, Lake Bluff, Illinois, USA) with a diamond disc (Series 15LC Diamond, Buehler, Lake Bluff, Illinois, USA) under water coolant. Zirconia bars were finished in a polishing machine (Metaserv® 2000 Grinding/Polishing, Buehler, Illinois, USA) at 30 rpm under water coolant using sandpapers (Ultra-Prep™ 45 μm and 15 μm, Buehler, Lake Bluff, Illinois, USA) and polishing clothes (TexMet C, Buehler, Lake Bluff, Illinois, EUA) impregnated with diamond suspension (15μm, MetaDi® Supreme Polycrystalline Diamond Suspensions, Buehler, Lake Bluff, Illinois, USA)[19].

A sintering procedure was conducted following the manufacturer's instructions at 1500°C for 8 hours in the Lava furnace (Lava™ Therm, 3M ESPE, Sumaré, São Paulo, Brazil). The final dimensions of the specimens after shrinkage (≈ 25%) were checked with a digital caliper (500-144B, Mitutoyo Sul Americana, Suzano, São Paulo, Brazil) and were equal to 20mm X 4mm X 1.2mm.

The bars were randomly divided into three groups: storage in distilled water (DW, n=12, control), brushing with distilled water (BDW, n=12) and brushing with distilled water and fluoride toothpaste (BFT, n=12). The suspension was made with a 1:2 ratio, respectively, of toothpaste (Oral-B® 1.2.3, Procter & Gamble Brazil S/A, Queimados, Rio de Janeiro, Brazil) measured in grams and distilled water measured in mililiters[14].

Specimens were brushed using a mechanical device (Mavtec, Comércio e Serviços - Desenvolvimento para Laboratório, Ribeirão Preto, São Paulo, Brazil) equipped with 6 soft bristle toothbrush heads (Oral-B Indicator®, Procter & Gamble Brazil S/A, Queimados, Rio de Janeiro, Brazil) at a rate of 60 reciprocal strokes per minute, and to provide a vertical load of 100g on the specimens. Toothbrushes and vehicles were changed every 22,080 strokes[14]. The specimens of the DW group remained statically submersed in distilled water for the same amount of time as those of the BDW and BFT groups.

Vickers hardness and mean roughness (Ra, μm) values were determined before and after the experimental treatments. The Vickers hardness was measured using a microhardness tester (MMT-3, 1600-6300, Buehler, Lake Bluff, Illinois, USA), with 500 gf for 30s. Measurements were made on four points, obtaining an average for each bar. The mean roughness was measured using a profilometer (Mitutoyo SJ 400, Mitutoyo Corporation, Yokohama, Kanagawa, Japan) at three different locations with reading accuracy of 0.01μm, length of 2.5mm, active tip radius of 5μm and speed of 0.5mm/s. The data were submitted to normality test (Kolmogorov-Smirnov test) and were subsequently analyzed using the two-way ANOVA test with significance level of 5%.

RESULT

The averages of the Vickers hardness and mean roughness (Ra) values, according to the experimental treatments, are shown in Table 1. There were no significant differences among the experimental groups (Vickers hardness, $p = 0.928$; Ra, p =

Table 1. Averages and standard deviations of Vickers hardness (VHN) and mean roughness (Ra, in μm), according to the experimental groups. Araraquara, 2014

Property	Group	Initial	Final
Mean roughness (μm)	DW	0.62 ± 0.17	0.63 ± 0.14
	BDW	0.60 ± 0.08	0.64 ± 0.19
	BFT	0.60 ± 0.14	0.68 ± 0.13
Vickers hardness (VHN)	DW	1305.33 ± 105.240	1301.16 ± 89.89
	BDW	1310.25 ± 76.52	1316.60 ± 84.47
	BFT	1313.44 ± 55.11	1299.58 ± 100.29

0.22); there were also no significant changes between initial and final values for both properties (Vickers hardness, $p = 0.856$; Ra, $p = 0.793$).

DISCUSSION

Various authors have designed studies of the surface roughness and hardness of Y-TZP zirconia[5,7-12,20]. These studies have attracted attention because of the degradation process to which zirconia is subject when exposed to the oral environment and, in particular, when it is without veneering porcelain. An increase in roughness may provide greater plaque accumulation, favoring the development of periodontal disease[14,21]; while a decrease in hardness can cause cracks and fractures[5,9-13,15]. However, studies evaluating the effect of brushing on Y-TZP zirconia are scarce.

The oral cavity is an inhospitable environment due to the presence of moisture, temperature changes, pH fluctuations, chewing forces and other factors that create situations for zirconia degradation, inducing a t→m phase transformation[3,9-11,13,22,23]. Thus, the aim of this study was to evaluate if the simulation of 10 years of brushing on the Y-TZP zirconia surfaces could change the roughness and hardness of this material due to the friction of the brush bristles associated with the abrasive particles and chemical components of the dentifrice. Based on the results, the null hypothesis was accepted because there were no changes in the Ra and Vickers hardness values after simulating 10 years of brushing, regardless of the toothpaste use.

In dentistry, Y-TZP zirconia becomes clinically usable after sintering, which transforms the monoclinic phase to the tetragonal phase, enhancing its hardness and resistance[1-3]. On the other hand, the reverse transformation (t→m) of the surface layers of zirconia can be harmful, simultaneously increasing surface roughness and decreasing hardness.

Long-term degradation studies have shown increases in the Ra values of zirconia due to grain loss and t→m transformation[5,9,20]. The mechanism by which this occurs was described by Chevalier et al.[9]; it refers to grain nucleation on the surface that generates stress to neighboring grains and microcracking of the material. Hence, there is a growth of the transformed zone, leading to the extension of microcracks and increased surface roughness; moreover, detachment of these grains still can occur. This same mechanism was used to explain the decrease in hardness values found by Catledge et al.[12], establishing an inverse relationship between the surface roughness and hardness of zirconia[5,10].

Some authors consider mechanical abrasion and friction forces to be factors that can act directly on the roughness, and contribute to the degradation process of zirconia[5,9,20]. In general, the mean roughness of zirconia ranges from 0.2 to 0.98μm[6-8], with differences attributed to the various compositions and methods for obtaining samples (type of cut, type of polishing) prior to the reading of this parameter. In this study, the initial roughness of the samples was in the range of 0.6μm, with no statistically significant difference among the groups after the experimental treatments. This shows that simulating 10 years of brushing (878, 400 cycles, 100gf) is not sufficient to change the roughness of the specimens, as reported by Pereira*, who assessed 400,000 brushing cycles. Regarding the Vickers hardness, data in the literature establish values close to 1300 VHN[3,5], which values are consistent with those found in this study. Hence, the null hypothesis, that simulating 10 years of brushing would not affect the hardness of zirconia, was accepted. Statistically significant differences in Vickers hardness between brushed and control specimens were not found.

Unobserved changes in the values of roughness and hardness in this in vitro study may be attributed to the absence of variation in temperature and pH of the solutions that occur in the oral environment, that some authors have been trying to simulate in aging tests of the zirconia. Several papers reported the occurrence of t→m phase transformation due to heating of the material in procedures of grinding and/or sandblasting[8,16], mechanical stress of chewing[10,22], pH fluctuations[17,18] and humidity[9,10,20]. However, in this study, the mechanical stress caused by the bristles of the toothbrushes and by the abrasiveness of the dentifrice (silica with 3.3μm and RDA equal to 105, calcium carbonate with 2.13μm and RDA equal to 33)[24] were not sufficient to induce a t→m phase transformation or to change the roughness and hardness values of the zirconia. Also, in vitro simulation conditions of 10 years of brushing do not accurately correspond to what occurs in the oral environment, where there are physical and

*Pereira PC. Efeito da escovação na formação in situ de biofilme dentário inicial e na rugosidade superficial em cerâmica de *Y-TZP* após vitrificação e polimento [dissertação mestrado]. São José dos Campos: Faculdade de Odontologia da UNESP; 2010.

chemical interferences such as temperature and pH changes, which were not implemented in this study. On the other hand, the use of toothpastes with different abrasiveness, like bleaching toothpastes[25], could exert a greater influence on the zirconia properties, which is an issue for further investigations.

The brushing time chosen (10 years) may also not have been enough to cause interference in the evaluated properties; however, it is not known if such interference would become visible with more years of brushing. In addition, the 10-year period corresponds to the mechanical stress caused by in vitro brushing (about 244 h in solution) but it does not correspond to an immersion of zirconia for 10 years.

CONCLUSION

It was concluded that the procedure of brushing with distilled or fluoridated toothpaste was not able to change the roughness and hardness of Y-TZP zirconia used in this study.

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CONFLICT OF INTEREST

The authors declare no conflicts of interest.

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