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Original article

Evaluation of surface treatments of monolithic zirconia in different sintering stages

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ABSTRACT

Purpose: This study evaluated the effect of surface treatment of zirconia in pre-sintered and post-sintered stages on its surface roughness, phase transformation, and biaxial flexural strength (BFS).

Methods: Zirconia ceramic discs (n=40) having a final dimensions of 12 mm diameter and 1.2 mm thickness were milled then divided into three main groups according to the type of surface treatment performed (Group 1 (n = 16); air-abrasion using Al₂O₃ particles, Group 2 (n = 16); silica coating using Rocatec soft, and Group 3 (n = 8); a control group receiving no surface treatment). Groups 1 and 2 were divided into two subgroups each according to the stage in which the surface treatment was performed (Subgroup A; surface treatment performed in the pre-sintered stage and subgroup B; surface treatment performed in the post-sintered stage). Surface roughness, phase transformation, and biaxial flexural strength (BFS) were later assessed. Data was then analyzed using two-way ANOVA and Tukey post-hoc tests at a significance level of $p \leq 0.05$.

Results: Subgroups treated in the pre-sintered stage showed higher mean Ra in μm (1.81 ± 0.36) when compared to the subgroups treated in the post-sintered (0.68 ± 0.07) stage and the control group (0.51 ± 0.10) ($p \leq 0.05$). The pre-sintered treated group and the control showed no monoclinic phase while the post-sintered group showed significantly higher portions of monoclinic phase. Regarding BFS the post-sintered treated group had statistically significant higher values in MPa (1228 ± 81) when compared to the pre-sintered treated group (940 ± 101) and the control (1019 ± 82) ($p \leq 0.05$).

Conclusions: Air abrasion in the pre-sintered stage might be a promising surface treatment method to produce promising surface roughness values of zirconia without subjecting it to early degradation.

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1. Introduction

The increased popularity of all-ceramic materials as an alternative to metal-ceramic restorations is mainly due to their excellent esthetics, chemical stability and biocompatibility [1]. The development of advanced dental ceramics has led to the application of partially stabilized zirconia in restorative dentistry which can be produced using a computer aided design/computer-aided manufacturing systems (CAD/CAM). The use of zirconia-based ceramics for dental restorations became more popular due to their superior fracture strength and toughness when compared

to other dental ceramic systems [2–4]. The application of zirconia in the fabrication of all-ceramic restorations has become one of the most focused on topics in dental research. Such increase in the interest is mainly due to its high mechanical strength and exceptional biocompatibility [5,6].

Zirconia can assume three different crystal structures, according to the temperature. At room temperature it is monoclinic, but transforms at 1170 °C into a tetragonal structure and at 2370 °C becomes cubic. The tetragonal structure of zirconia can be retained in a metastable phase at room temperature, by adding oxides like CaO, MgO, Y₂O₃ or CeO₂ [7].

Partially sintered zirconia also known as un-sintered zirconia is milled from porous blocks with incomplete sintered grains and open boundaries to larger dimensions and require further sintering for the ceramic to gain its full density [7,8]. This sintering

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procedure is accompanied by a sintering shrinkage of about 20–30% [6]. In this stage heat is transmitted to the surface of the material and reaches its core by thermal conduction transforming zirconia from its un-sintered condition to its post-sintered or fully sintered condition [9].

For dental applications, tetragonal zirconia polycrystals are commonly stabilized with 3 mol% yttria (3Y-TZP) [7]. The high strength and toughness of these zirconia materials result from a phenomenon called transformation toughening, which occurs due to the transformation from the tetragonal to the monoclinic structure ($t \rightarrow m$ transformation). This transformation is associated with a volume increase of about 4%, thus inducing localized compressive stresses and, eventually, hinders microcracks around the transformed zirconia particles [5]. This transformation tends to take place in regions of concentrated stress, in particular at flaws at the surface or within the lattice, and effectively opposes the opening of cracks, thus increasing the resistance to further crack propagation.

Aside from this favorable effect at crack tips, the $t \rightarrow m$ transformation may also be unintentionally induced by hydrothermal aging, acidification during food intake, cyclic loading during chewing, and abrasion surface treatments protocols. This spontaneous transformation is commonly referred to as low-temperature degradation (LTD) [10,11]. Consequently, the $t \rightarrow m$ transformation spreads gradually along the surface and penetrates into the depth of the material [12]. This spontaneous transformation tends to decrease the mechanical stability of zirconia restorations [13,14].

The success of zirconia-based all-ceramic restorations is highly dependent on the establishment of a strong adhesion between zirconia and the luting cement [15]. However, without any surface treatment, the resin zirconia integration was found to be susceptible to aging conditions [16]. In addition, acid etching followed by the application of silane coupling agents, could not effectively improve the bond strength between zirconia and resin cement due to the absence of a glass content [7,17]. Air-abrasion with alumina particles followed by an appropriate chemical bonding process using 10-methacryloxydecyl dihydrogen phosphate (MDP) was recommended to achieve long-term resin bonding to zirconia [18]. The incorporation MDP in primers or resin cements was a major factor in producing durable bond between resin and zirconia which has already been confirmed in the related clinical trials [19,20]. Other surface treatments, such as the tribochemical silica coating which air-abrades the ceramic surface with alumina particles that have been coated with silica, embedding/coating the surface with silica have been developed to enhance resin zirconia bonding [21].

However, there are still concerns about the influence of air-abrasion and tribochemical silica coating on the mechanical properties and long-term stability of zirconia ceramic since it has been reported that they induce some flaws and phase transformation on the surface, thus promoting low temperature degradation [22]. The generation of such flaws and transformation might produce some detrimental effects on the liability of zirconia ceramic [23].

The aim of this study was to evaluate the effect of air-abrasion or silica coating zirconia in the pre-sintered or post-sintering stages on its surface roughness, phase transformation, and biaxial flexural strength in an attempt to surface treat zirconia prior to bonding without subjecting it to phase transformation nor affecting its final strength. The null hypothesis of this study was that the type of surface treatment and the stage in which it is performed will not affect the roughness and phase transformation of zirconia ceramic and its biaxial flexural strength.

2. Materials and methods

Forty zirconia discs (Bruxzir Shaded, Glidewell, California, United States) (Fig. 1) (Table 1) having a final dimensions after sintering of 12 mm diameter and 1.2 mm thickness were milled using a dental milling machine, polished using SiC paper till 600 grits then divided into three main groups according to the type of surface treatment performed (group 1; (n = 16) air-abrasion using Al_2O_3 50 μm particles, group 2; (n = 16) silica coating using 30 μm Rocatec soft particles (3M ESPE, St. Paul, United States), and group 3; (n = 8) a control group receiving no polishing or surface treatment). Groups 1 and 2 were divided into two subgroups (n = 8) each according to the stage in which the surface treatment was performed (subgroup A; surface treatment performed in the pre-sintered stage and subgroup B; surface treatment performed in the post-sintered stage). Surface treatments were performed using perpendicular air-abrasion in a crosswise motion of the nozzle from a distance of 10 mm for 15 s. In subgroup A an air pressure of 0.5 bar was used, while in subgroup B the pressure was 2.8 bar. Specimens were later ultrasonically cleaned in 99% isopropanol solution for 3 min and then dried with air. All specimens were then

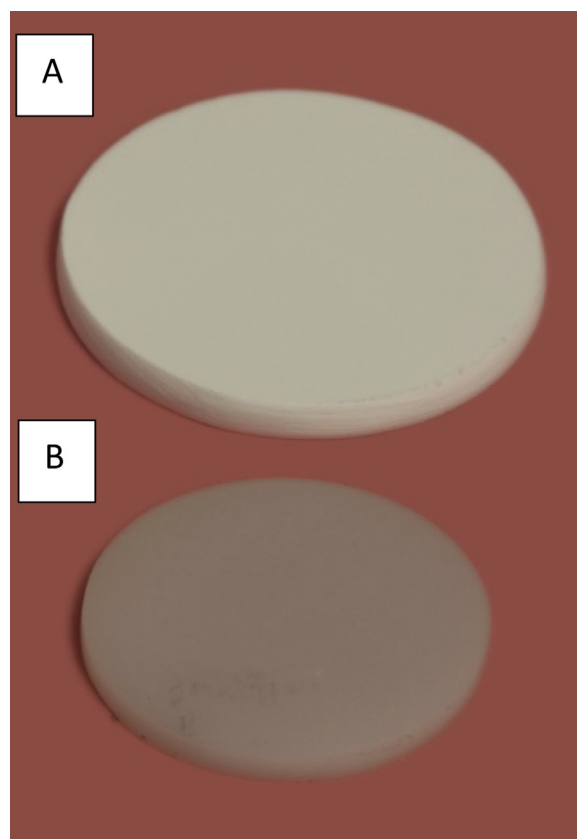


Fig. 1. Milled zirconia discs: (A) before sintering (un-sintered) and (B) after sintering (post-sintered).

Table 1

Composition of Bruxzir shaded zirconia as stated by manufacturer.

Chemical name	% by weight
Zirconium oxide (ZrO_2) + Y_2O_3 + HfO_3 + Al_2O_3	>99.9
Yttrium oxide (Y_2O_3)	<5.15
Hafnium oxide (HfO_3)	<3
Aluminum oxide (Al_2O_3)	<0.5
Silicon oxide (SiO_2)	<0.02
Iron oxide (Fe_2O_3)	<0.01
Sodium oxide (Na_2O)	<0.04

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