UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

NATÁLIA ALMEIDA BASTOS

Is it still necessary to roughen the Y-TZP surface?

Ainda é necessário criar rugosidade na superfície da Y-TZP?

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Orientador: Profª. Dra. Ana Flávia Sanches Borges

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DEDICATÓRIA

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“Todas as coisas contribuem juntamente
para o bem daqueles que amam a Deus, daqueles
que são chamados segundo o seu propósito.”

Romanos 8:28

ABSTRACT

ABSTRACT

Is it still necessary to roughen the Y-TZP surface?

Statement of the problem. The yttria-stabilized tetragonal zirconia (Y-TZP) ceramics are widely used manufacturing of dental prostheses. Since it is most used as framework, the ceramics veneers still show fractures, resulting in reduction of longevity of the prosthesis.

Objective. Evaluate and test the effectiveness of roughening treatments between Y-TZP and veneering ceramic before liner application.

Material and methods. Forty Y-TZP discs (12.5mm Ø and 3.5mm thickness) were divided in four groups (n = 10): LC: application of the proprietary liner only; S40: pre-sintered sonication with 40 % nominal power for 15 minutes; S70: pre-sintered sonication with 70 % nominal power for 15 minutes; JOX: sandblasting with 50 µm aluminum particles. After roughening treatments, the liner was applied to the surface of the samples in all groups. After sintering, the specimens were subjected to confocal laser scanning microscopy (CLSM) and atomic force microscopy (AFM) analyses to evaluate the topography and surface roughness. The phases of the crystalline structures were identified through micro-Raman spectroscopy (MRS). After that, a veneering ceramic was applied to all Y-TZP surfaces and subjected to shear bond strength (SBS) testing at a speed of 0,5mm/min until fracture. Failure modes were classified as adhesive, cohesive, or mixed. SBS results were subjected to one-way ANOVA (α = 0.05) followed by Tukey's test (α = 0.05).

Results. No differences were shown between groups regarding surface roughness (p=0,255), although Confocal and AFM images showed topographical differences among groups. The LC group showed the highest SBS median values, which were significantly different from S70 and JOX (p=0,008), which were not different. S40 showed the lowest SBS median values (15,43 MPa). All specimens exhibited typical Y-TZP tetragonal bands and monoclinic phases. Most specimens of all groups exhibited mixed failures (67,5%) and the remaining specimens exhibited adhesive failures.

Conclusion. Additional surface treatments for Y-TZP, sandblasting, and 70 % power sonochemical treatment showed similar results but did not increase SBS to a feldspathic ceramic.

Key words: Y-TZP ceramic. Air abrasion, Dental. Ceramics. Microscopy. Spectrum Analysis, Raman.

Resumo

RESUMO

Ainda é necessário criar rugosidade na superfície da Y-TZP?

Problematização. As Zircônias tetragonais estabilizadas com ítria (Y-TZP) são amplamente utilizadas na confecção de próteses dentárias. Uma vez que é mais utilizada como infraestrutura, as cerâmicas de cobertura ainda mostram fraturas, resultando em redução da longevidade da prótese.
Objetivo. Avaliar e testar a eficácia de tratamentos de superfície entre Y-TZP e cerâmica de cobertura antes da aplicação do liner.
Material e métodos. Quarenta discos Y-TZP (12,5mm de diâmetro e 3,5mm de espessura) foram divididos em quatro grupos (n = 10): LC: aplicação exclusiva do liner; S40: sonicação pré-sinterizada com 40 % de potência nominal durante 15 minutos; S70: sonicação pré-sinterizada com 70 % de potência nominal durante 15 minutos; JOX: jateamento com partículas de alumínio de 50μm. Após os tratamentos rugosos de superfície, o liner foi aplicado na superfície das amostras todos os grupos. Após a sinterização, os espécimes foram submetidos a Microscopia confocal de varredura a laser (CLSM) e Microscopia de força atômica (AFM) para avaliar a topografia e a rugosidade da superfície. As fases das estruturas cristalinas foram identificadas através de espectroscopia micro-Raman (MRS). Após esta etapa, a cerâmica de cobertura foi aplicada em todas as superfícies da Y-TZP e os espécimes foram submetidos ao teste de Resistência de união ao cisalhamento (SBS) a uma velocidade de 0,5mm/min até à fratura. Os modos de falha foram classificados como adesiva, coesiva ou mista. Os resultados de SBS foram submetidos a uma ANOVA a um critério ($\alpha = 0,05$) seguido pelo teste de Tukey ($\alpha = 0,05$).
Resultados. Não foram observadas diferenças entre os grupos quanto à rugosidade superficial (0,255), embora as imagens da Microscopia Confocal e AFM apresentassem diferenças topográficas entre os grupos. O grupo LC apresentou maiores valores medianos de SBS, significativamente diferentes de S70 e JOX ($p=0,008$), os quais não foram diferentes. S40 apresentou os valores medianos de SBS mais baixos (15,43 Mpa). Todos os espécimes Y-TZP exibiram bandas típicas tetragonais e fases monoclínicas. A maioria dos espécimes de todos os grupos exibiram falhas mistas (67,5%) e os espécimes restantes exibiram falhas adesivas.
Conclusão. Tratamentos de superfície adicionais para Y-TZP, jateamento e tratamento sonoquímico com potência de 70 % mostraram resultados semelhantes, mas não aumentaram a SBS para uma cerâmica de cobertura.

Palavras chave: Cerâmica Y-TZP. Abrasão Dental por Ar. Cerâmica. Microscopia. Espectroscopia Raman.

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LIST OF ABBREVIATIONS AND ACRONYMS

eV	Electron volt
µm	Micrometer
min	Minute
LC	Liner control
JOX	Sandblasting with 50µm aluminum particles
S40	Sonication with 40 % nominal power
S70	Sonication with 70 % nominal power
Ra	Average surface roughness
AFM	Atomic Force Microscopy
CLSM	Confocal Laser Scanning Microscopy
PVC	Cylinder of Polyvinyl Chloride
SBS	Shear bond strength
CTE	Thermal expansion coefficient
nm	Nanometer
^{o}C	Degrees Celsius
mm	Millimeter
mW	Megawatt
Y-TZP	Yttria-stabilized tetragonal zirconia
cm^{-}	Centimeter
h	Hour
kgf	Kilogram-force
MPa	Megapascal

t	Tetragonal
m	Monoclinic
EDX	Energy dispersive X-ray spectrometry
α	Alpha
ZrO_2	Zirconium dioxide
Y_2O_3	Yttrium oxide
HfO_2	Hafnium dioxide
SiO_2	Silicon dioxide
Al_2O_3	Aluminium oxide
Na_2O	Sodium oxide
K_2O	Potassium oxide
CaO	Calcium oxide
P_2O_5	Phosphorus pentoxide
F	Fluor
Rpm	Revolutions per minute
Ø	Diameter

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1 Introduction

1 INTRODUCTION

Yttria-stabilized tetragonal zirconia (Y-TZP) ceramics are widely used in oral rehabilitation. Besides providing excellent esthetic results, Y-TZP ceramics have advantages such as color stability, biocompatibility, staining, high toughness and abrasion resistance, making them the material of choice for dental prostheses.[39] Since it is mostly used as framework, the veneering ceramic is still a concern, presenting fractures[37,42] and resulting in reduction of material longevity. Between 2 and 5 years, clinical failure rates of zirconia and veneering ceramic extent from 13 % to 15 %.[42,48,54] Failure types can be chipping (cohesive fractures inside the veneer layer) or veneer layer delamination at the zirconia-feldspathic interface.[1,10] According to Komine et al.[26] progression of fracture usually occurs from the area subjected to higher tensile stress to the interface. When this occurs, the stresses in the veneer layer may attain the interface with zirconia substructure.[50]

Several fractures may follow different ways, but radial cracks (R) are more prevalent initiating from the lower surface of framework (where flexural stress is concentrated), and inner and outer cone cracks that are developed on ceramic surface near the loaded area.[8] Within the factors that can influence the beginning and development of cracks are: mechanical properties of material (elastic modulus, fracture resistance, fracture toughness), residual stress and thickness of the veneering ceramic.

Studies suggest that a smaller amount of empty spaces is created in the veneer layer when the pressed technique is employed[7], increasing the structural reliability of the zirconia-based restorations. Although, it is important to consider that chip fractures on Y-TZP restorations with hand-layer technique sometimes present higher prevalence[38,42] and sometimes lower.[33,52] Fracture strength and elastic modulus incompatibility between the ceramic layers influences failure of multilayered structures, which results in a significant concentration of tensile stresses.[51] Also, factors such as thermal expansion coefficient (CTE), cooling rate, porcelain thickness and structure design are more important for structural integrity of the veneering ceramic than the manufacturing technique.[6]

Veneering ceramic is applied on zirconia because there is stability between these materials, not only for esthetic reasons[22,42,54] and when used alone feldspathic ceramic becomes friable and limited in tensile strength. However, clinical failures such as veneering ceramic delamination, have been reported as one of the most common concerns of this restorative combination.[40] In this way, the demand for micromechanical retention between Y-TZP and veneering ceramic is continuous and it involves different methods, such as sandblasting,[13,14] grinding,[36] liner application,[24,49] acid etching,[26] polishing,[13,23] silica coating,[13] and laser etching.[30] Some manufacturers recommend airborne-particle abrasion, which is a popular mean used to accomplish this micromechanical retention, increasing the surface roughness.[2,15,32] On the other hand, sandblasting can cause surface damage, affecting mechanical strength and probably the material bonding capacity.[29,58] It is important to consider that surface treatment may promote a phase transformation at the surface, changing the crystal structure from tetragonal to monoclinic.[20,28] Both structures show different coefficients of thermal expansion (CTE),[20] affecting the mechanical strength and probably, the material bonding capacity.[13,15] Other surface treatment alternative is sonochemical therapy, in which sound waves result in acoustic cavitation produced by implosive collapse of bubbles,[46] potentially modifying the treated ceramic surface, promoting greater bond strength between both materials.

It is a well-known concern for long-term clinical success of zirconia restorations that the bond strength between veneering ceramic and infrastructure is sufficient to stand the masticatory load. Evidences on different surface treatments and their influence on strength and bonding mechanisms are limited.[2,32] Considering these informations, the aim of this study was to evaluate if roughening treatments would modify Y-TZP surface improving its bond strength to veneering ceramic before liner application.

2 ARTICLE

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The article presented in this Dissertation was written according to The Journal of Prosthetic Dentistry instructions and guidelines for article submission (Annex A).

IS IT STILL NECESSARY TO ROUGHEN THE Y-TZP SURFACE?

ABSTRACT

Statement of the problem. The yttria-stabilized tetragonal zirconia (Y-TZP) ceramics are widely used manufacturing of dental prostheses. Since it is most used as framework, the ceramics veneers still show fractures, resulting in reduction of longevity of the prosthesis.
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Key words: Y-TZP ceramic. Air abrasion, Dental. Ceramics. Microscopy. Spectrum Analysis, Raman.

INTRODUCTION

The study and use of ceramic materials for dental applications has presented a remarkable improvement in the last 10–15 years due to the increasing demand for long-term, aesthetic, and high- performance implants and restorations. [27,7] In this context, non-silicate ceramics in special zirconia-based materials have been used in dentistry with increasing success. Ytria-stabilized tetragonal zirconia ceramic (Y-TZP) provides high fracture strength and long-term performance if compared to other ceramic materials.[41] However, the vulnerability of Y-TZP/veneering ceramic bilayers is chipping and/or delamination of the weaker material.[35,32] Between 2 and 5 years, clinical failure rates of zirconia and ceramic veneer extend from 13 % to 15 %.[46,35,39] When failures occur, they can be cohesive inside the veneer layer (chipping) or adhesive at the Y-TZP/veneering ceramic interface (delamination), and mixed failure when occour the combination of these two failure types.[1,8]

Surface treatments in Y-TZP dental materials can be performed by different methods: sandblasting,[11,14] grinding,[29] liner application,[21,40] acid etching,[22] polishing,[11,20] silica coating,[11] and laser etching.[25] Among these, sandblasting is the most used and recommended by Y-TZP manufacturers because it provides an effective increase in surface roughness, leading to a better micromechanical retention with a ceramic veneer.[2,15,26] However, the higher roughness obtained by sandblasting has not shown a direct relation with higher Y-TZP/veneering ceramic bond strength.[23] It is known that the topographic quality of a surface can also modify the elastic modulus of the material and, consequently, influence load distributions.[42] Furthermore, the rougher the surface, the higher the stress concentration, which may weaken the bonding interface[9] and may promote an earlier phase transformation, from tetragonal to monoclinic, at the Y-TZP surface.[24,18]

In restorative dentistry, liners are materials used as a thin coating (usually 0.5mm) on the surface of a cavity preparation. In terms of composition, they are composed of SiO_2, Al_2O_3, Na_2O, K_2O, CaO, P_2O_5, and F. Liners can be applied as a layer between the zirconia-veneering ceramic to mask the structure and increase the wetting on the zirconia.[21] One study showed that the use of liner increased Y-TZP/veneering ceramic bond strength when compared to other surface treatments[3], while other studies have shown that the liner decreased its bond strength.[13,44]

The aim of this study was to test and evaluate the effectiveness of roughening treatments between Y-TZP and a veneering ceramic before liner application. The null hypothesis tested was that there would not be a difference in Y-TZP/veneering ceramic bond strength, regardless of roughening treatments, sonochemical and sandblasting.

MATERIALS AND METHODS

Discs were obtained from pre-sintered Y-TZP blocks (15.5mm width x 19mm length x 39mm height) (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein). They were milled from a cylindrical shape of 12.5mm in diameter and 39mm in height. After that, each cylinder was cut using an Isomet 1000 cutter (Buehler, Lake Bluff, IL, USA) and diamond disc (series 15LC Diamond no. 11-4254, Buehler, Lake Bluff, IL, USA) at 275-rpm low speed under cooling water.

The specimens were subjected to a polishing machine (EXACT, Nordestedt, Schleswing-Holstein, Germany) with #1000 and #1200 sandpapers (Polishing paper K2000, EXACT, Nordestedt, Schleswing- Holstein, Germany), followed by a sequence of treatments with felt wheels of medium, fine, and extra-fine granulations and diamond paste (Polishing paper K2000, EXACT, Nordestedt, Schleswing- Holstein, Germany). The specimens were cleaned by double cycle soaking in 100 % ethanol and distilled water in an ultrasound machine (USC 700—Unique Industry and Trade of Electronic Products Ltda, Sao Paulo, SP, Brazil) for 10min. Relevant information on the tested materials is presented in Table 1.

The 40 pre-sintering Y-TZP discs (12. 5mm ø and 3.5mm thickness, before sintering) were randomly divided into 4 groups (n = 10) according to the surface treatment, as presented in Table 2. In the control group (LC) only a liner application was performed after final sintering. Another group (JOX) suffered a sandblasting treatment with particles of aluminum oxide, and the two remaining groups were sonochemically treated with low-power sound waves (S40) and high-power sound waves (S70), both producing acoustic cavitation on the surface of the specimens.[36]

For the LC and JOX groups, the treatments were done after final sintering, as stated.[17,21] For LC, no roughening treatment was performed.

For JOX specimens, sandblasting treatment was done with 50μm alumina particles under a pressure of 0.4MPa for 10s, perpendicular to the surface and at a distance of 10mm, using an airborne-particle abrasion device (Refenrt, Ribeirão Preto, Sao Paulo, SP, Brazil).[30,34,47] For S40 and S70, sonochemical treatment was performed for 15min or both powers, and the Y-TZP discs were fixed in a device to standardize their centered position at the bottom of a beaker filled with deionized water (Fig. 1).

After each of the four different purposed surface treatments, the specimens were sintered according to the manufacturer’s instructions, as shown in Table 3. The final dimensions obtained were 10.5mm ø and 2.8mm thickness.

Morphological Characterization

The discs (n = 10) of each group were analyzed at one point by confocal microscopy (DCM 3D Model, Leica Microsystems, Wetzlar, Germany) in order to evaluate surface roughness (Ra) and surface topography.

An atomic force microscope (AFM) (XE-70, Park Systems, Tokyo, Japan) was used to obtain 3-dimensional images (n = 1). The measurements were performed without contact with an adjusted cantilever at a distance around 5.8nm (set point), and the scan size was 4mm on both the X and Y axes of the center of the specimens.

Phase Analysis

Y-TZP discs (n = 10) were evaluated by means of a Jobin Yvon micro-Raman system, model T64000 (Groupe Horiba—Longjumeau, France). The excitation of an argon ion laser (Spectra Physic, Inc., California, USA) was used with a radiation of 514.5nm (2.41eV), and the beam was focused by microscope magnification of 500x. The laser power was maintained at 10mW with the aim of avoiding thermal damage. The Raman spectra analyses were performed using a double subtractive monochromator with a focal distance of 0.64M and equipped with a diffraction grating with 1,800 grooves/mm, and a spectral resolution of 2cm was provided with a slit width of 200µm. The recording of spectra was performed using a CCD (Spectra One—Groupe Horiba, Longjumeau, France) camera. All specimens were evaluated using the average of 3 measurements of each disc, and finally a general average of this value was generated.

Veneering ceramic application

To build the veneering ceramic layer, the IPS e.max ceramic and Ceram Dentin and Build-Up system were used (Ivoclar Vivadent AG, Schaan, Liechtenstein), applying a ZirLiner layer

(ZirLiner, Ivoclar Vivadent AG, Schaan, Liechtenstein) on the surface of all Y-TZP discs. Thus thereafter, the specimens were taken to be fired, following the temperature control.

The limited heat conductivity of zirconium oxide requires a firing wash, which was a mixture of the required IPS Ceram Dentin or Deep Dentin with IPS e.max Ceram Build-Up liquids. The firing wash was conducted to reach a homogeneous connection with the ZirLiner layer. After that, the firing parameters of the material were followed.

A custom-designed metallic device (5mm in diameter and 5mm in height) was used for the application of the IPS e.max veneering ceramic (Ivoclar Vivadent AG, Schaan, Liechtenstein). The veneering ceramic was mixed with the modeling liquid (Ivoclar Vivadent AG, Schaan, Liechtenstein) and inserted into the device by manual condensation. The device was removed, and the specimens were sintered. Recommended firing procedures were respected. After that, the specimens were ready for shear bond strength testing.

After sintering, each tooth was embedded in a cylinder of polyvinyl chloride (PVC) (21mm in diameter and 25mm in height) using acrylic resin (JET; Classic, Sao Paulo, SP, Brazil).

Shear Bond Strength Test

For this test, the specimens were stored for 24h in water at 37 °C, following ISO TR 11405, Type I, for material bond strength. After this step, all specimens were subjected to shear mechanical testing by a Kratos 5002 universal biomechanical test machine (Kratos Dynamômetros, Sao Paulo, Brazil), with a load cell of 500kgf, in a special device with a metal strip at a speed of 0.5mm/min until fracture. This device [33] was adapted from Sinhoreti et al.[38] in order to minimize bending forces during the test.[37]

The analysis of the ceramic interface and veneering zirconia was performed for all the specimens with a digital microscope DinoLite AM313T (AnMoElectronics Corporation, Dung-Da Road, Taiwan) at 32x magnification. Each specimen was classified according to the type of failure: (a) adhesive failure at the Y-TZP/veneering ceramic interface; (b) cohesive failure of the ceramic veneer; or (c) mixed, meaning both types of failure were present. The types of failure were classified by digital microscopy, and their percentage was calculated.

Statistical analysis

Data on shear bond strength and roughness were calculated and statistically analyzed with Statistica software (Statsoft®, Tulsa, OK, USA). The assumptions of normal distribution and of equality of variances were checked for all the variables using the Kolmogorov-Smirnov and Levene tests, respectively. As the assumptions were satisfied, data were subjected to one-way ANOVA ($\alpha = 0.05$), which required them to be directed to the Kruskal-Wallis test ($\alpha = 0.05$), followed by Tukey's test ($\alpha = 0.05$) for individual comparisons.

RESULTS

Surface roughness

The performed sandblasting and sonochemical surface treatments did not show a significant quantitative difference in increasing the surface roughness (p=0,255). The median roughness (Ra) for each group was 121.98 (LC), 119.95 (S40), 125.22 (S70), 118.89 (JOX) and data dispersion, as shown in Figure 2.

In order to deeper explore the surface morphologies due to the proposed surface treatments, confocal microscopy (CLSM) and atomic force microscopy (AFM) analyses were conducted. Representative images of CF (Fig. 3) and AFM (Fig. 4) of the zirconia surfaces pointed to micron and nanoscale morphological differences. The red color represents a vertical (z-axis) size of around 400nm and the dark blue color represents -400nm.

In Figure 3 it is possible to compare the surface morphologies and observe that sandblasted surfaces resulted in micro-sized, rougher and more irregular surfaces due to the high impact of alumina particles (Fig. 3D). Otherwise, the sonochemically treated surfaces (Fig. 3B and 3C) showed more regular morphologies compared to sandblasted specimens and presented morphological characteristics more similar to the liner control group (Fig. 3A).
Atomic force microscopy (AFM) was used to investigate surface morphology at the nanoscale level. In Figure 4, besides differences in roughness, it is possible to observe the grain size, topological changes in the nanostructure, and grain damage due to surface treatments. In Figure 4C and 4D it is possible to note that high-energy sonochemical and sandblasting treatments produced damages in the grain of the Y-TZP surfaces.

Phase analysis

Figure 5 shows the Raman spectra of each group, reporting the bands of monoclinic and tetragonal phases. Previous literature reported that each crystalline phase of Y-TZP is represented by the peaks of the major bands. The tetragonal phase is characterized by strong peaks (~256, 320, 466, and 637cm^{-1}), while the monoclinic phase presents at ~177, 185, and 382cm^{-1}.[49] The peaks regarding the bands were found for the groups as follow: LC ~266, 324, 468, 182, and 304 cm^{-1}; JOX ~264, 326, 468, 182, and 306 cm^{-1}; S40 ~180, 264, 324, 468, and 526 cm^{-1}; and S70 ~180, 266, 322, 466, and 528cm^{-1}. All groups showed characteristic bands of the tetragonal (T) and monoclinic (M) phases, suggesting that there was phase transformation (t → m).

Shear Bond Test

The lowest initial median shear bond strength (MPa) was obtained in the S40 group (15.43MPa), which was lower than the LC group (29.9MPa), the JOX group (25.0MPa), and

S70 group (18.49MPa) (Fig. 6). The Tukey test results showed a significant difference (p=0,008) in the shear bond strengths between the LC, S40, and JOX groups, as the LC and JOX groups exhibited a higher median strength than S40. This can be explored in Figure 7, where if an interval does not contain 0, the corresponding means are significantly different.

Failure mode analysis

Figure 8 shows the failure mode and the distribution for each group. Most specimens in all groups exhibited a mixed mode of failures (67,5%), and the remaining specimens showed adhesive failures. Few cohesive failures were observed, and none of the specimens fractured within the zirconia.

DISCUSSION

The null hypothesis tested was rejected . There was no difference in Y-TZP/veneering ceramic bond strength associated with roughening treatments. The adhesion mechanism of zirconia and veneer ceramic has not been totally explained, but the literature has reported the existence of a fusion between the zirconia and veneering ceramic, resulting in diffusion of the elements of each material at the bonded interface.[4,10]

The current study did not show statistical quantitative differences among groups, which could be confirmed by confocal and atomic force microscopies. The average surface roughness was statistically similar in all groups on a microscale, but it was possible to detect some effects of high-energy treatment only at the nanoscale.

The effect of sandblasting on increasing the shear strength of veneer-zirconia was not confirmed in the current study. This could be because sandblasting affects the Y-TZP mechanical strength and probably the bonding capacity of the material,[11,15] due to the CTE difference of tetragonal (10.8ppm/K) and monoclinic (7.5ppm/K) zirconia, which is

considerably lower.[12] A nano-modified surface can be obtained by sonochemical therapy, whereby sound waves result in acoustic cavitation produced by the implosive collapse of bubbles,[36] potentially modifying the treated ceramic surface. Although sonochemical therapy is innovative, it did not increase the zirconia/veneer shear bond strength compared to other treatments. There were different surface characteristics for each treatment surface. For JOX, the surface appeared rough and irregular, with well-defined, micro-sized elevations and depressions, possibly by the high impact of alumina particles, while there was more regularity and homogeneity on the surfaces of S40, S70, and LC (Figs. 5–6). This could be attributed to the surface of the sandblasted Y-TZP not having a significant influence on the veneering ceramic bond strength when compared to non-sandblasted groups (LC, S40, and S70). These results could be confirmed in a related study wherein there was no influence of sandblasting on the shear strength for all zirconia and corresponding veneering ceramics investigated.[16]
The application of a liner increased the zirconia/veneer shear bond strength, which might be due to the sufficient wetting of the liner over the zirconia surface[47] suggested that there is no evidence of chemical bonding between zirconia/veneer and zirconia/liner because the ceramic components cannot be found on the surfaces of all specimens upon EDX analysis. [47] However, because the wettability depends the composition of the veneer and liner, the morphology of the zirconia surface, and the surface energy of the core material,[31] provides free atoms available for chemical bonding.[5] The Y-TZP grains' borders are higher surface area interfaces ,[43] which also have Y_2O_3, HfO_2, and Al_2O_3. Al_2O_3 is also present in the liner, which could lead to the hypothesis that the Al_2O_3 from both materials could flow and fuse with each other.
In order to investigate the types of fractures, the microscope is the most important device for understanding the location of crack onset, size, and spread.[28] Regarding the type of failure, most specimens in all groups exhibited a mixed mode of failures, while the remaining specimens showed adhesive failures. Few cohesive failures were observed, and none of the

specimens fractured within the zirconia. Most mixed-type fractures showed began as an adhesive type at the side of the load application and terminated as a cohesive type on the opposite side,[45] and Yoon et al.[50] observed, with the same parameters used for sandblasting, a higher percentage of mixed failures, data similar to this study.

Raman analysis showed that the alternative sonochemical treatment caused a crystallographic transition, as did sandblasting. The specimens' preparations seem to have caused this event, as the liner group without roughening treatment also had identifiable monoclinic peaks.

Some studies have reported the effect of sandblasting on the mechanical properties and bonding reliability of zirconia.[6,25] It was observed that could lead to a phase transformation (t→m) of the surface grains due to the formation of a compressive layer on the zirconia surface,[19] resulting in surface flaws and voids, which could prejudice the longevity and clinical performance of zirconia-based restorations.[51] The clinical implication of this finding is that the investigated all-ceramic systems, instead of catastrophic failure of the core, have a tendency to lead to chip-off fractures and delamination of the veneering ceramic.[48]

This present study suggests that only the application of a liner on the Y-TZP as a pretreatment to the zirconia substrate increased the bond strength between the zirconia and the veneering ceramic. This treatment could be an alternative to sandblasting and sonochemical treatments, as it is not so aggressive on the surface of zirconia and is therefore less harmful to the longevity of the ceramic restoration. Moreover, using a liner decreases laboratory time and cost, because sonochemical treatment requires buying specific equipment.

The specimen engendered by the zirconia-veneer ceramic did not represent a clinical condition found in dental restoration, although it was a shape necessary to evaluate shear bond strength. This design could be considered a study limitation because of its shape, which could have affected the thermal conductivity of the zirconia during firing.

The gold standard for shear bond strength values was not reached between the core of Y-TZP and its corresponding veneering ceramics.[16] The high rates of veneering ceramics chipping observed in clinical studies may be explained by the interactive factors from both materials, their composition, and processing. Clinical trial studies are still required to evaluate the behavior of the interface of Y-TZP/veneer ceramics over the long term.

CONCLUSIONS

It is suggested that the application of the liner on the surface of the Y-TZP may be an alternative because it is not so aggressive, which contributes to the longevity of the restoration. In addition, its use reduces time and labor cost

There is no statistically direct relationship between modification of surface morphology with bond strength

There is no evidence that there is diffusibility between Zirconia and veneering ceramics

Roughening treatments seem unnecessary to provide enough bond strength at Y-TZP/veneer ceramic interfaces.

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FIGURE CAPTIONS

Fig 1. A. Ultrassonic for sonochemical treatment; B. Device used for specimen centralization; C. Sample in deionized water for treatment with microtip. Centralization of Y-TZP discs at the bottom of a Becker with deionized water.

Fig 2. Effect of surface treatment on roughness of Y-TZP ceramic surfaces. Vertical bars indicate the inner quartile range, vertical lines are the maximum and minimum values, and the horizontal line is represented by the median.

Fig 3. Surface topography represented in 3D images of the selected groups: LC (A), S40 (B), S70 (C) and JOX (D).

Fig 4. AFM images of Y-TZP: LC (A), S40 (B), S70 (C) and JOX (D).

Fig 5. Raman analysis of the four groups studied, for Monoclinic (M) and Tetragonal (T) phases.

Fig 6. Shear bond strength (SBS) of veneering ceramics after surface treatments. Vertical bars indicate the inner quartile range, vertical lines the maximum and minimum values, and the horizontal line is represented by the median. Increased data dispersion in the JOX group.

Fig 7. Results of Tukey's multiple comparisons for shear bond strengths. If an interval does not contain zero, the corresponding means are significantly different (p=0,008).

Fig 8. Percentage of failure types in each group (%) (n = 40).


(a)


(b)


(c)

Fig 1

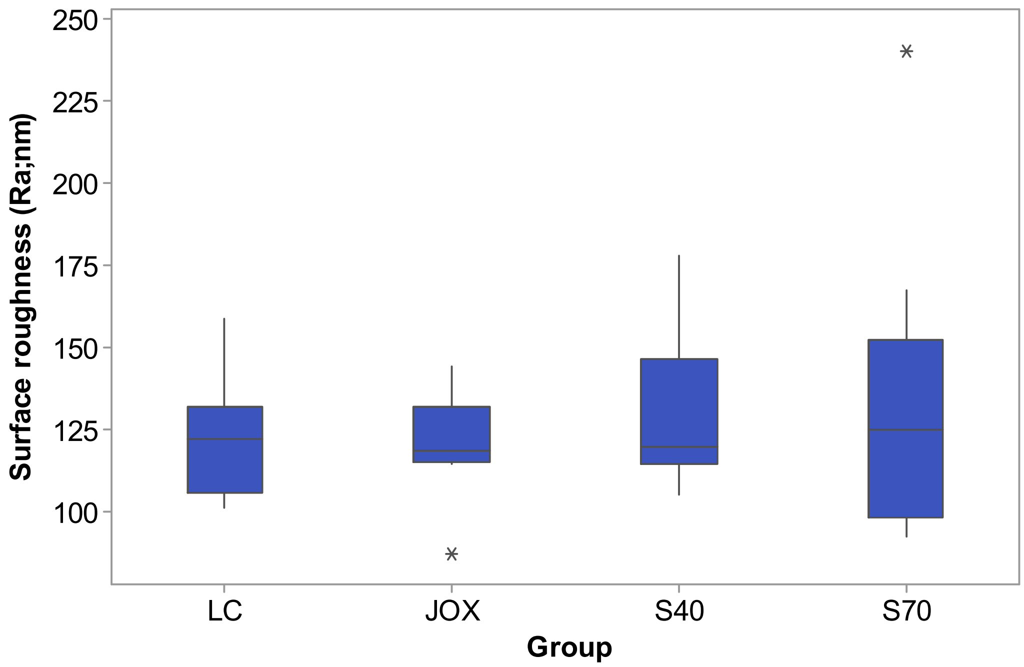

Surface roughness (Ra;nm)
250
225
200
175
150
125
100
LC
JOX
S40
S70
Group

Fig 2

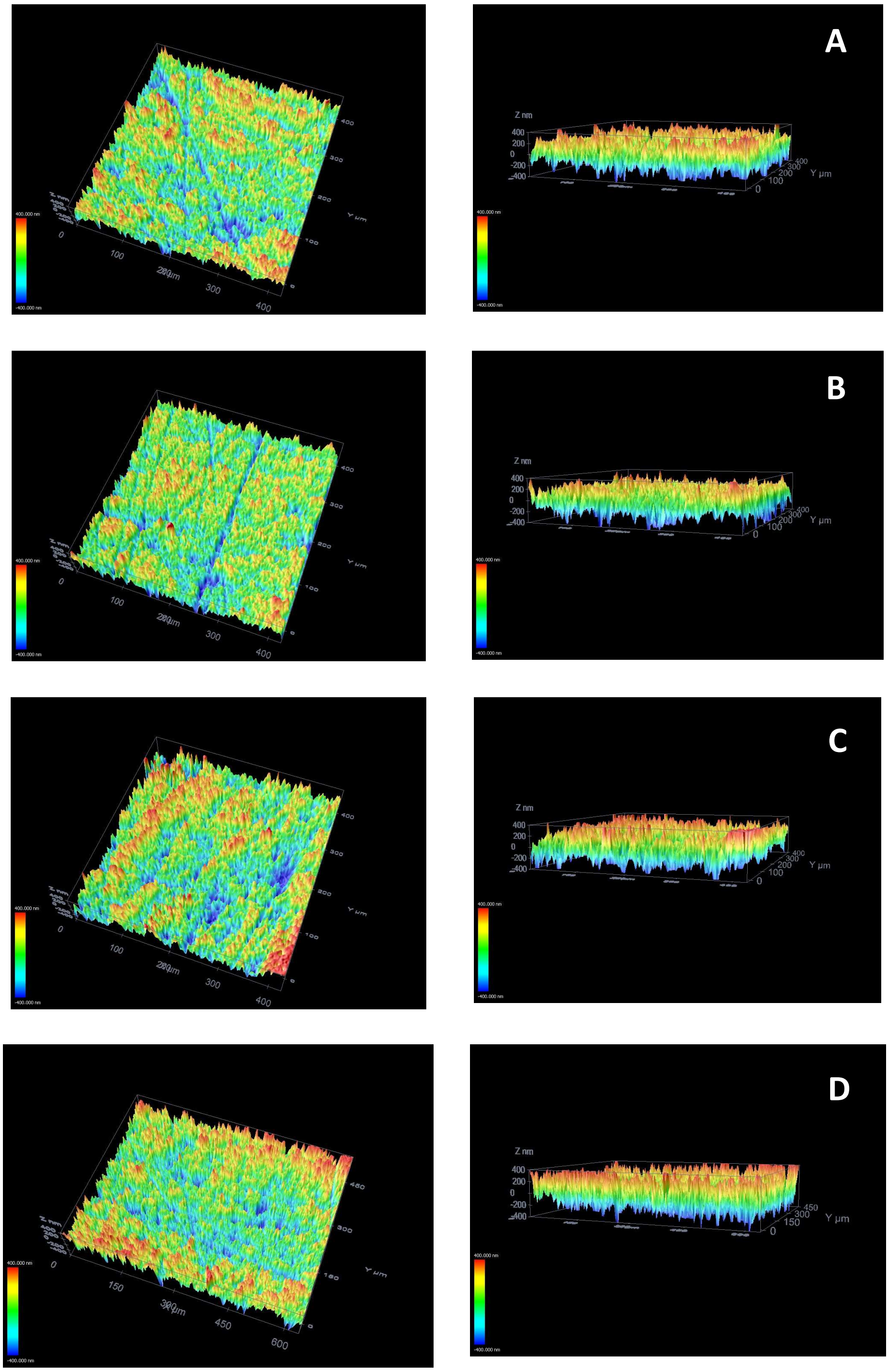
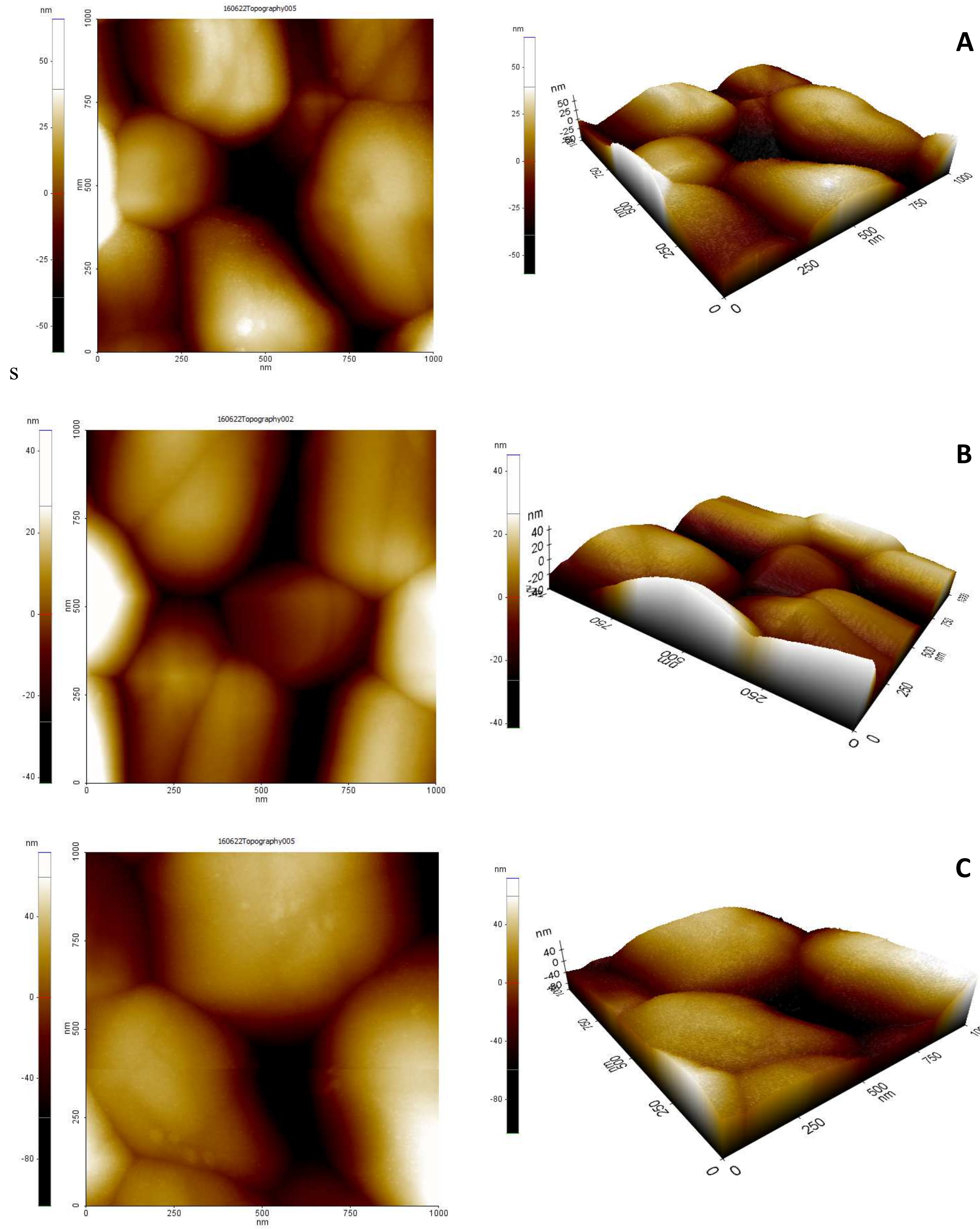


Fig 3


160622Topography005
A
160622Topography002
B
160622Topography005
C

S

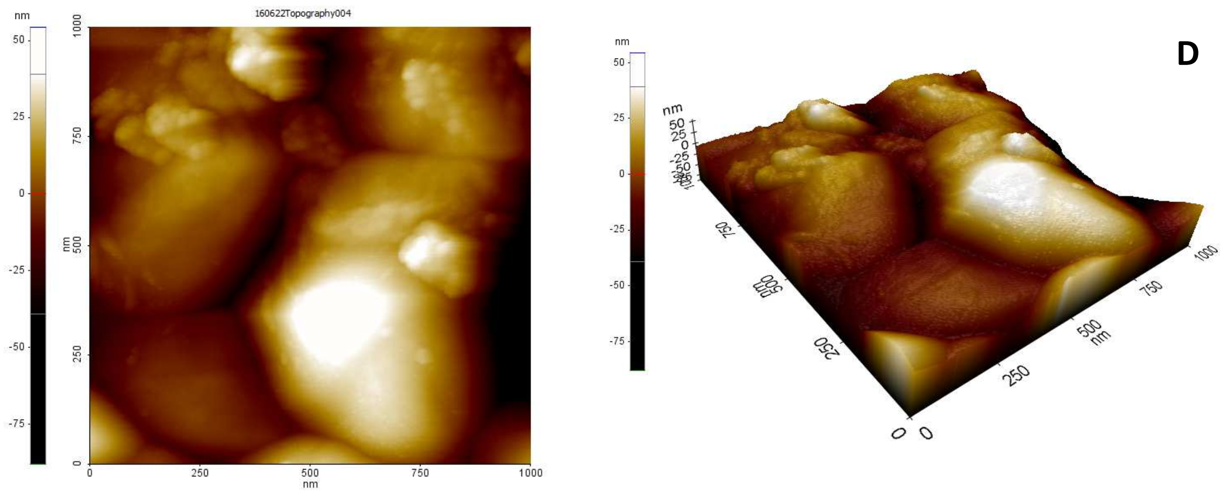
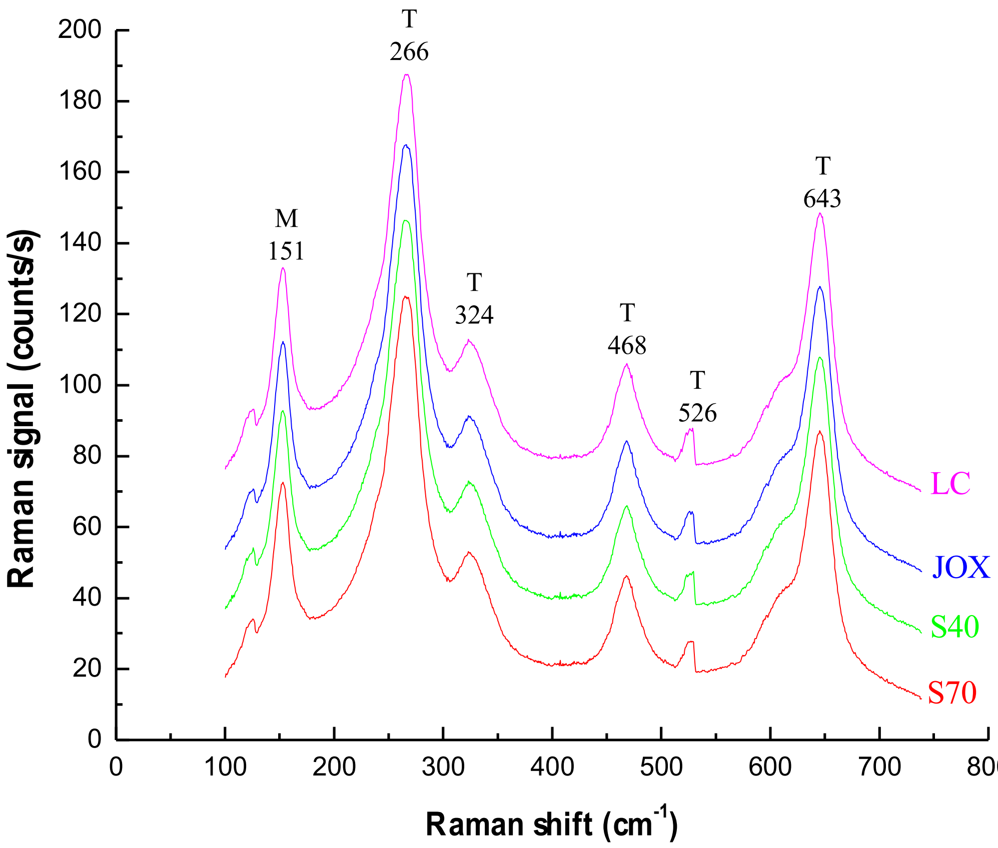


Fig 4



200
180
160
140
120
100
80
60
40
20
0
Raman signal (counts/s)
0
100
200
300
400
500
600
700
800
Raman shift (cm-1)
M
151
T
266
T
324
T
468
T
526
T
643
LC
JOX
S40
S70

Fig 5

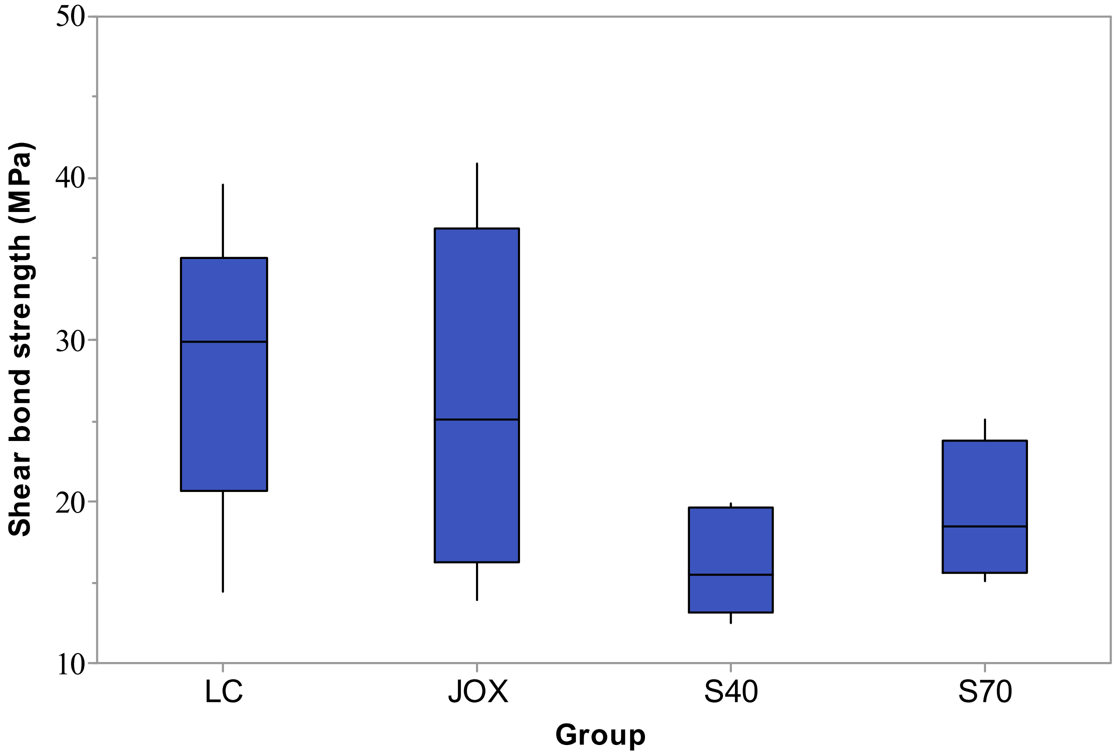


Fig 6

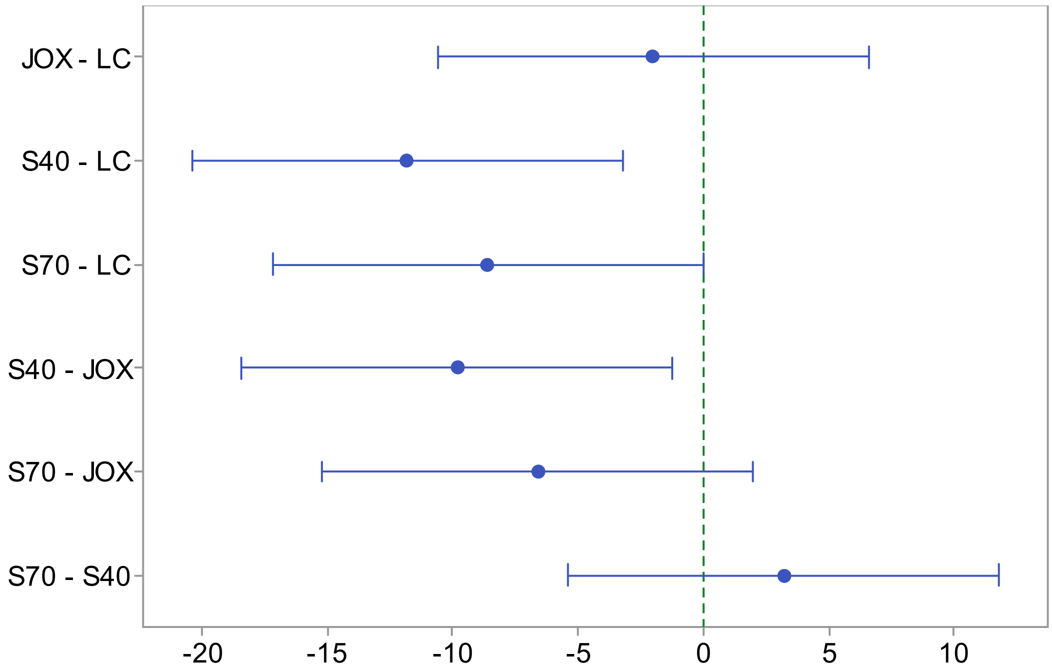


Fig 7

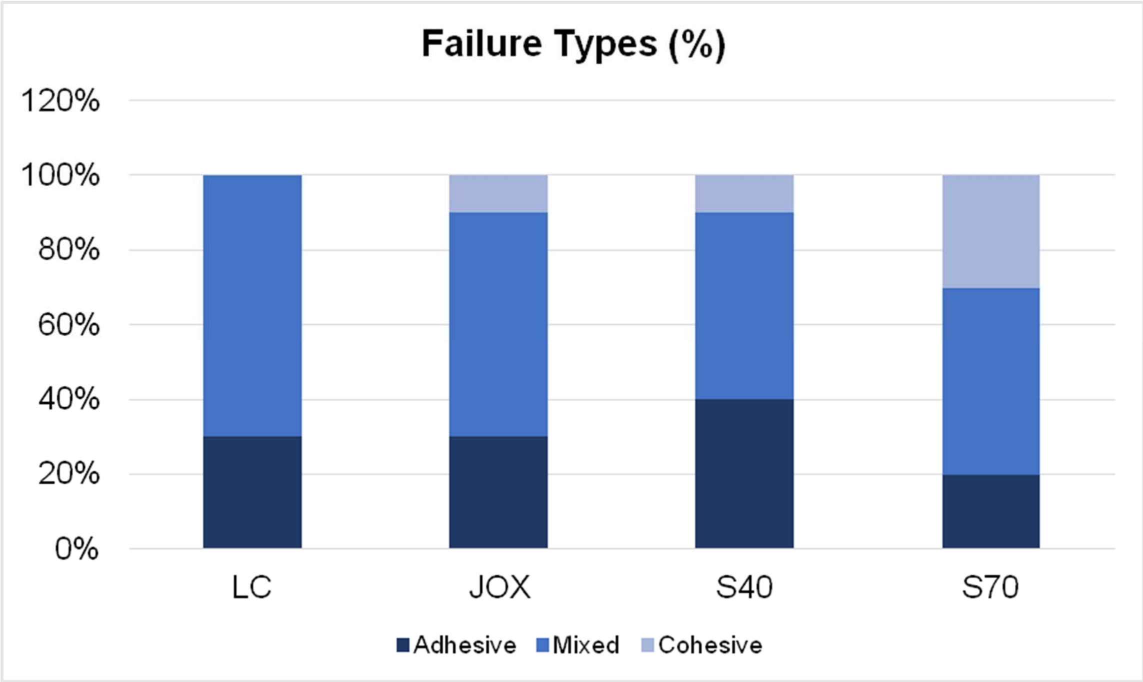

Failure Types (%)
120%
100%
80%
60%
40%
20%
0%
LC
JOX
S40
S70
Adhesive
Mixed
Cohesive

Fig 8

Table 1. Materials, Composition and Coefficient of thermal expansion (CTE) of this study.

Material/Code	Classification	Manufacturer	Composition (% by weight)	CTE
IPS e.max Zircad**/EZ	Y-TZP ceramic	Ivoclar Vivadent, Schaan, Liechtenstein	ZrO_2, 87 %; Y_2O_3, 4 %; HfO_2, 1 %; Al_2O_3, 1 %	10.75
IPS e.max Ceram/EC	Veneer ceramic	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2, 60 %; Al_2O_3, 8 %; Na_2O, 6 %; K_2O, 6 %; ZnO, 8 %; CaO, P_2O_5, F, 2 %	9.5
IPS e.max Ceram ZirLiner	Liner	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2, 50 %; Al_2O_3, 16 %; Na_2O, 6 %; K_2O, 4 %; CaO, P_2O_5, F, 2.5 %	9.8

*Coefficient of thermal expansion (CTE) in $10^{-6}K^{-1}$ between 100 °C and 500 °C.

** Based on manufacturer's information by technical profile

Table 2. Groups according to Y-TZP surface treatment.

Groups	Surface treatment
LC	Liner Control: only liner application after final sintering
JOX	Treated with particles of aluminum oxide (micro-jet - BIOART - São Carlos, SP, Brazil) after final sintering
S40	Sonochemical treatment with nominal power of 40 % with (Sonics VCX - 750) before final sintering
S70	Sonochemical treatment with nominal power of 70 % with (Sonics VCX – 750) before final sintering

Table 3. Recommended cycles for zirconia firing.

Heating schedule	IPS e.max ZirCAD ™
Standby temperature	403 °C
Drying time	12ºC/min
Heating rate	65 °C/min
Maximum firing temperature	1050 °C
Holding time	15min

3 Discussion

3 DISCUSSION

The enhance in zirconia restorations clinical performance is achieved by means of an increased adhesion between zirconia substrate and veneering ceramic,[13] since delamination of veneering porcelain, fracture of the veneering ceramic, and fracture of the zirconia substructure are the main complications of porcelain-veneer Y-TZP fixed dental prostheses.[4,55] Therefore, many studies evaluate surface treatments to improve zirconia's adhesion to veneering ceramics.[19,41]

Studies have shown that zirconia surface treatments tend to weaken it, increase fracture risk and also increase the content of monoclinic phase.[17,35] Therefore, the present study was designed to evaluate and test the effectiveness of roughing treatments between Y-TZP and veneering ceramic before liner application. The null hypothesis tested was accepted. There was no difference in Y-TZP/veneering ceramic bond strength associated with roughening treatments.

Shear bond test was used in the present study to evaluate bond strength of zirconia/veneering ceramic after different surface treatments, because it is shown to have a wide application in the literature[4,16,24,36] and usually can be applied to bilayered zirconia-based ceramic systems.[4,16]

A proportional relationship between increased roughness and bond strength was not confirmed in the current study. In general, average surface roughness of zirconia substrates was statistically similar to all groups, but presented surface modifications as revealed by Confocal and AFM evaluations (Fig. 5-6). Results of the present study show that although there was different surface characteristics for each surface treatment, the bond strength did not considerably increase. Fischer et al.[13] showed that increased surface roughness did not enhance shear bond strength.

Effects of liner application and sandblasting on zirconia cores bond strength have shown controversial results in literature. Some studies have shown that liner have decreased the bond strength[2,13,18,36] or have demonstrated that it has either no effect[13] or has caused an increase.[3] Also, sandblasting can increase the shear bond strength with veneering porcelain[30] or not.[12,13]

Among the surface treatments proposed in studies, results indicate that liner application showed the highest bond strength values when compared to other groups (LC, S40 and S70), which might be due to the sufficient wetting of liner over the zirconia surface.[56] However, sandblasted groups did not have better veneering ceramics bond strength when compared to non-sandblasted groups. These results could be confirmed in a related study, where there was no influence of sandblasting on shear strength for all zirconia and corresponding veneering ceramics investigated.[16] In contrast to the findings of the present study,[41] has shown that sandblasting followed by liner application over conventionally sintered zirconia core yielded to the highest bond strength.

Innovative sonochemical therapy can potentially modify the treated ceramic surface, even though it did not increase zirconia/veneer shear bond strength compared to other treatments. This result can be explained based on investigations of different adhesion mechanisms of zirconia/veneering ceramic, since the micromechanical interactions were merely assumed.[16]

Phase transformation from tetragonal to monoclinic phase implies a volume expansion of 3-4 %.[5,21] Grains expansion makes them push each other, creating roughness on zirconia surface.[31]

In literature, some studies show that, regardless of the conditions, sandblasting can induce phase transformation on zirconia surface,[13,44,45] increasing monoclinic phase amount can lead to the development of micro-cracks in the glass phase of the veneer and to a decline in strength.[3,9] Examination of phase transformation zone revealed that all proposed treatments caused a crystallographic transition. Therefore, crystalline structure instability may also be a negative factor in the maintenance of zirconia/porcelain integration. Thereby, it would be more desirable, procedures which provide an initially weaker material, but more stable through time.[15]

Among failures that core-veneered zirconia restorations may suffer (cohesive and adhesive)[25], the most frequent failures are by delamination and chipping[42]. Although the adhesion mechanism is not known, it is assumed that it could be the result of diffusion of liner components into the surface of zirconia.[2,13]

In this study, fractured veneer/zirconia surfaces showed most specimens in all groups exhibited mixed failure modes and the remaining specimens had shown adhesive failures. Few cohesive failures were observed and none of the samples fractured within zirconia. In most mixed type fractures, fracture began as an adhesive type at load application side, and terminated as a cohesive type on the opposite side. Surfaces treated with sandblasting showing higher percentage of mixed failures have been reported previously.[53,57]

According to results of this study, roughening treatments seem unnecessary to provide enough bond strength at Y-TZP/veneering ceramic interface. However, the limitation of this study was that specimen design and dimensions may not represent the clinical shape of dental restorations, but provide a geometry that allows measurements of shear bond strength. It is believed that the result of the present study will help to define a surface treatment protocol for zirconia. Nevertheless, more clinical and in vitro research is needed to confirm the validity of these results.

4 Final Considerations

4 FINAL CONSIDERATIONS

Within the limitations of this in vitro study, it was concluded that the additional surface treatments for Y-TZP, ie, sandblasting and 70 % power sonochemical treatment, presented similar results, but it did not increase shear bond strength to a feldspathic ceramic. Therefore, a rougher surface seems unnecessary to provide enough bond strength at Y-TZP/veneering ceramic interfaces. However, considerable refinements are required in order to overcome existing incompatibilities between Y-TZP and veneering ceramics, and minimize chipping of veneering ceramics. Future in vivo long-term studies should evaluate the clinical performance of the interface between Y-TZP and veneering ceramics.

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ANNEXES

ANNEX A – Guidelines for The Journal of Prosthetic Dentistry:

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It is important that the file be saved in the native format of the MS Word program. The text should be in single-column format. Keep the layout of the text as simple as possible. Most formatting codes will be removed and replaced on processing the article. In particular, do not use the word processor's options to justify text or to hyphenate words. However, do use bold face, italics, subscripts, superscripts etc. When preparing tables, if you are using a table grid, use only one grid for each individual table and not a grid for each row. If no grid is used, use tabs, not spaces, to align columns. The electronic text should be prepared in a way very similar to that of conventional manuscripts (see also the Guide to Publishing with Elsevier: http://www.elsevier.com/guidepublication). Note that source files of figures, tables and text graphics will be required whether or not you embed your figures in the text. See also the section on Electronic artwork.

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Formatting of funding sources
List funding sources in this standard way to facilitate compliance to funder's requirements:

Funding: This work was supported by the National Institutes of Health [grant numbers xxxx, yyyy]; the Bill & Melinda Gates Foundation, Seattle, WA [grant number zzzz]; and the United States Institutes of Peace [grant number aaaa].

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Follow internationally accepted rules and conventions: use the international system of units (SI). If other units are mentioned, please give their equivalent in SI.

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Figure Submission

JPD takes pride in publishing only the highest quality figures in its journal. All incoming figures must pass a thorough examination in Photoshop before the review process can begin. With more than 1,000 manuscripts submitted yearly, the manuscripts with few to no submission errors move through the system quickly. Figures that do not meet the guidelines will be sent back to the author for correction and moved to the bottom of the queue, creating a delay in the publishing process.

File Format
All figures should be submitted as TIF files or JPEG files only.

Image File Specifications

Figure dimensions must be 5.75 × 3.85 inches.
Figures should be size-matched (the same physical size) unless the image type prohibits size matching to other figures within the manuscript, as in the case of panoramic or periapical radiographs, SEM images, or graphs and screen shots. Do not "label" the faces of the figures with letters or numbers to indicate the order in which the figures should appear; such labels will be inserted during the publication process. Do not add wide borders to increase size.

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The figures should be of professional quality and high resolution. The following are resolution requirements:
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If text is to appear within the figure, labeled and unlabeled versions of the figures must be provided. Text appearing within the labeled versions of the figures should be in **Arial font and a minimum of 10 pt.** The text should be sized for readability if the figure is reduced for production in the Journal. Lettering should be in proportion to the drawing, graph, or photograph. A consistent font size should be used throughout each figure, and for all figures, Please note: Titles and captions should not appear within the figure file, but should be provided in the manuscript text (see Figure Legends).

If a key to an illustration requires artwork (screen lines, dots, unusual symbols), the key should be incorporated into the drawing instead of included in the typed legend. All symbols should be done professionally, be visible against the background, and be of legible proportion should the illustration be reduced for publication.

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Color Figures

Generally, a maximum of 8 figures will be accepted for clinical report and dental technique articles, and 2 figures will be accepted for tips from our reader articles. However, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.
Clinical figures should be color balanced. Color images should be in CMYK (Cyan/Magenta/Yellow/Black) color format as opposed to RGB (Red/Green/Blue) color format.

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Graphs should be numbered as figures, and the fill for bar graphs should be distinctive and solid; no shading or patterns. Thick, solid lines should be used and bold, solid lettering. Arial font is preferred. Place lettering on white background is preferred to reverse type (white lettering on a dark background). Line drawing should be a minimum of 600 dpi. Screen Captures should be a minimum of 300 dpi and as close to 5.75 and 3.85 as possible.

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The figure legends should appear within the text of the manuscript on a separate page after Tables and should appear under the heading FIGURES. Journal style requires that the articles (a, an, and the) are omitted from the figure legends. If an illustration is taken from previously published material, the legend must give full credit to the source (see Permissions).

File Naming

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In the article, clearly reference each Figure and Table by including its number in parentheses at the end of the appropriate sentence before closing punctuation. For example: The sutures were removed after 3 weeks (Fig. 4). Or: are illustrated in Table 4.
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Place thumbnails (reduced size versions) of your figures in Figures section below each appropriate legend.
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All files are saved as TIFFs or JPEGs (only).Figure size: 5.75" × 3.85" (radiographs, SEMS, and screen captures may vary but they must all be size-matched). Figures are 300 dpi; line or combo line/photo illustrations are minimum 600 dpi.For text in figures use Ariel font.Label the Figure files according to their sequence in the text.Provide figure legends in the manuscript Figure section.Place thumbnails (small versions of figure files approx. 2" × 1.5") in Figure section below each legend.Submit composite figure parts as separate files.
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• Use a logical naming convention for your artwork files.
• Provide figure legends in the Figure section.
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A detailed guide on electronic artwork is available on our website:
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The complete reference list, double-spaced and in numerical order, should follow the Conclusions section but start on a separate page. Only references cited in the text should appear in the reference list.

Reference formatting should conform to **Vancouver style** as set forth in "Uniform Requirements for Manuscripts Submitted to Biomedical Journals" (Ann Intern Med 1997;126:36-47).

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List up to six authors. If there are seven or more, after the sixth author's name, add et al.

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