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Composite Resin to Yttria Stabilized Tetragonal Zirconia Polycrystal Bonding: Comparison of Repair Methods

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Clinical Relevance

Veneer chipping from yttria stabilized tetragonal zirconia polycrystal (Y-TZP) copings has become a common clinical concern. The present study presents information on the effect of different repair approaches on the bond strength of Y-TZP to a resin composite after aging. Among the assessed repair strategies, tribochemical silica coating provides the highest bond strength.

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SUMMARY

Purpose: The purpose of the current study was to evaluate different approaches for bonding composite to the surface of yttria stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics.

Methods: One hundred Y-TZP blocks were embedded in acrylic resin, had the free surface polished, and were randomly divided into 10 groups (n=10). The tested repair approaches included four surface treatments: tribochemical silica coating (TBS), methacryloxydecyldihidrogenphosphate (MDP)–containing primer/silane, sandblasting, and metal/zirconia primer. Alcohol cleaning was used as a "no treatment" control. Surface treatment was followed by the application (or lack thereof) of an MDP-containing resin cement liner. Subsequently, a composite resin was applied to the ceramic

surface using a cylindrical mold (4-mm diameter). After aging for 60 days in water storage, including 6000 thermal cycles, the specimens were submitted to a shear test. Analysis of variance and the Tukey test were used for statistical analyses (α=0.05).

Results: Surface treatment was a statistically significant factor (F=85.42; p<0.0001). The application of the MDP-containing liner had no effect on bond strength (p=0.1017). TBS was the only treatment that had a significantly positive effect on bond strength after aging.

Conclusion: Considering the evaluated approaches, TBS seems to be the best surface treatment for Y-TZP composite repairs. The use of an MDP-containing liner between the composite and Y-TZP surfaces is not effective.

INTRODUCTION

Yttria stabilized tetragonal zirconia polycrystals (Y-TZP) is a ceramic indicated for crowns and fixed partial denture frameworks as a result of its "high" toughness[1] and the good esthetic results it provides. One of the most common clinical complications involving ceramic restorations with Y-TZP frameworks is veneer chipping.[2] The immediate repair of this kind of failure is important to the well-being of the patient until the restoration can be replaced, if necessary. When the framework is exposed, effective bonding between composite resin and the Y-TZP framework is necessary.[3] However, the bonding of composite to Y-TZP has been shown to be a critical procedure, as indicated by the low bond strength values and reduced bond durability observed in *in vitro* tests.[4-6]

Surface conditioning (by mechanical or chemical methods) has been used to improve the bond strength of resin to the ceramic materials. Acid etching,[3,7-9] sandblasting (SAND),[8] the combination of both,[9] or silica coating,[8,10] followed by the application of silane produced high bond strength values of composite resin to feldspathic ceramics. The use of a resin cement containing methacryloxydecyldihydrogenphosphate (MDP),[4,11,12] SAND,[4,11] the combination of SAND and an MDP-containing primer/silane,[13] or tribochemical silica coating (TBS)[11,14-18] has been reported for bonding enhancement of composite resins to Y-TZP.

The investigation of different strategies for composite to Y-TZP bonding may yield some clinical direction when repair procedures are necessary. Therefore, the aim of this current study was to verify the effect of different repair approaches on the bond strength of Y-TZP to a resin composite. Different surface treatments—TBS, MDP-containing primer/silane system application (MDPS), SAND, metal/zirconia primer application (MZP), and no surface treatment (CRTL)—followed by the application (or lack thereof) of an MDP-containing resin cement liner application (RL), were evaluated. The hypotheses were as follows: 1) silica coating provides the highest bond strength values to Y-TZP among the evaluated surface treatments; 2) an MDP-based cement liner application increases bond strength values to Y-TZP.

MATERIALS AND METHODS

Y-TZP Block Preparation

One hundred 7.5 × 7.5 × 2.5–mm blocks of Y-TZP (In Ceram 2000 YZ cubes 40/15, Vita Zahnfabrik, Bad Säckingen, Germany) were cut with a diamond saw (#34570, Microdont, São Paulo, SP, Brazil) under water cooling in a customized machine. After sanding with #400 sandpaper the blocks were sintered in a VITA ZYRcomat furnace (Vita Zahnfabrik). After the recommended sintering cycle, the blocks presented with dimensions of approximately 5 × 5 × 2 mm as a result of the 20% to 25% sintering shrinkage.

The blocks were embedded in acrylic resin so that only the 5 × 5–mm surface was free for bonding. The exposed surface for bonding was then flattened using #400, #600, and #1200 sandpapers. Adhesive tape was placed around the Y-TZP surface of each specimen, leaving a circular 4-mm-diameter area exposed for bonding.

Experimental Groups

The samples were randomly divided into 10 groups with 10 blocks of Y-TZP in each (n=10). Each group was submitted to one of the following approaches.

1. TBS-RL: Silica coating (TBS) + RL. The Y-TZP bonding area was submitted to tribochemical silica coating (CoJet system, 3M-ESPE, Saint Paul, MN, USA). Initially, the Y-TZP surfaces were air-abraded with 30-ìm–silica-coated alumina particles (COJET SAND, 3M-ESPE) using an intraoral air abrasion device at a pressure of 2.8 bar from a distance of 10 mm for 15 seconds. The conditioned surfaces were then coated with an MPS silane (ESPE Sil, 3M-ESPE) and left to dry at ambient conditions for five minutes. The Y-TZP surface then received a thin layer of dual

cure resin cement containing MDP (Panavia F, Kuraray, Ozaka, Japan). After cement polymerization, a divided cylindrical mold (4-mm diameter and 3-mm depth) was used for the incremental insertion and photocuring of a composite resin (Clearfil Majesty Esthetic, Kuraray) onto the ceramic surface.
2. TBS: The same procedures were used as for group 1, but there was no liner application.
3. MDPS-RL: MDPS + RL. The Y-TZP bonding surface was acid-etched with 37% phosphoric acid, washed, dried, and then had a mixture of Clearfil SE Primer (Kuraray) and Porcelain Bond Activator (Kuraray) applied on its surface. The resin cement liner and composite resin were applied as described for group 1.
4. MDPS: the same procedures were used as for group 3 (MDPS-RL), but with no liner application.
5. SAND-RL: SAND + RL. The Y-TZP bonding surface was submitted to sandblasting with 50-ìm alumina particles using an intraoral air abrasion device at a pressure of 2.8 bar from a distance of 10 mm for 15 seconds. The resin cement liner and composite resin were applied as described for group 1.
6. SAND: The same procedures were used as for group 5 (SAND-RL), but with no liner application.
7. MZP-RL: MZP + RL. An MZP (Ivoclar Vivadent, Schaan, Liechtenstein) was applied on the Y-TZP bonding surface. The resin cement liner and composite resin were applied as described for group 1.
8. MZP: The same procedures were used as for group 7 (MZP-RL), but with no liner application.
9. CRTL-RL: CRTL + RL. The Y-TZP bonding surface was rubbed with 96% isopropanol for 30 seconds. The resin cement liner and composite resin were applied as described for group 1.
10. CRTL: The same procedures were used as for group 9 (CRTL-RL), but with no liner application.

Table 1 shows how the groups were distributed and prepared, according to the different approaches for composite resin to Y-TZP bonding. The chemical descriptions for the materials used in the current study are presented in Table 2.

Table 1: *Experimental Groups Used in the Study*

Group Description	Identification Code
Tribochemical silica coating; MDP-based cement liner	TBS-*RL*
Tribochemical silica coating	TBS
MDP-containing primer/activator system; MDP-containing cement liner	MDPS-*RL*
MDP-containing primer/activator system	MDPS
Sandblasting (Al_2O_3); MDP-containing cement liner	SAND-*RL*
Sandblasting (Al_2O_3)	SAND
Metal/zirconia primer; MDP-containing cement liner	MZP-*RL*
Metal/zirconia primer	MZP
No treatment (alcohol cleaning); MDP-containing cement liner	CRTL-*RL*
No treatment (alcohol cleaning)	CRTL

Water Storage and Thermal Cycling

The specimens were stored in distilled water at 37°C for 60 days. They were submitted to thermal cycling for approximately 103 hours (6000 cycles; 5°C and 55°C baths; 30 seconds each bath; two seconds of transition) while they were water-stored.

Shear Testing

After storage and thermal cycling, the specimens were submitted to shear testing in the Universal testing machine, EMIC DL-1000 (EMIC, São José dos Pinhais, PR, Brazil). A knife-shaped indenter applied the load at a cross-head speed of 0.5 mm/min. A metal frame was used for holding each specimen to guarantee that the adhesive interface was parallel to the path of the knife and as near as possible to the long axis of the knife. The shear strength was recorded in N/mm^2 (MPa). The interfacial area (A) was 12.57 mm^2 ($A=\pi r^2$, where $\pi=3.1416$ and r=adhesive interfacial radius=2 mm).

Failure Mode and Data Analysis

After testing, the specimens were analyzed in an optical microscope (Mitutoyo TM-505, Kanagawa, Japan), at 250× magnification to determine the predominant failure mode classification (adhesive = A; cohesive in ceramic = CC; cohesive in resin =

Table 2: *Chemical Composition and Use of the Materials Used in the Study*

Brand Mark	Manufacturer	Chemical Components	Use
CoJet sand	3M ESPE, Saint Paul, MN, USA	Aluminum oxide, amorphous silica	Blasting for silica coating
Metal/zirconia primer	Ivoclar Vivadent, Schaan, Liechtenstein	DMA, solvents, phosphonic acid acrylate, initiator and stabilizer	Primer
ESPE-Sil	3M ESPE, Seefeld, Germany	MPS, ethanol, methyl ethyl ketone	Silane coupling agent
Clearfil SE Bond Primer	Kuraray Medical Inc, Okayama, Japan	MDP, HEMA, hydrophilic dimethacrylates, DL-camphorquinone, *N,N*-diethanol-*p*-toluidine, H_2O	Self-etching primer
Clearfil Porcelain Bond Activator	Kuraray Medical Inc, Okayama, Japan	MPS, bisphenol-*a*-polyethoxy-dimethacrylate	Silane coupling agent
Aluminum oxide	—	Aluminum oxide	Sandblasting
Alcohol	Sigma Aldrich, Saint Louis, MO, USA	Isopropanol	Cleaning
Panavia F 2.0	Kuraray Medical Inc, Okayama, Japan	Paste A: MDP, DMA, silanated silica, DL-camphorquinone, others	Resin cement
		Paste B: DMA, silanated barium glass, sodium fluoride, others	

Abbreviations: DMA, dimethacrylates; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyldihydrogenphosphate; MPS, 3-methacryloyloxypropyl trimethoxysilane.

CR). The type of failure was confirmed under scanning electronic microscopy (SEM), using selected specimens.

Data were computed and submitted to analysis of variance (two-way ANOVA) and a 5% Tukey *post hoc* test.

RESULTS

The two-way ANOVA of the bond strength data is summarized in Table 3. Mean values, standard deviations, failure modes (%), and statistical homogeneity are described in Table 4.

There were different effects due to surface treatments ($p<0.0001$). The application of a MDP-containing RL was not related to an improvement in bond strength ($p=0.1017$) (Table 3).

TBS coating (used in groups TBS and TBS-RL) was the most effective method among the tested approaches, resulting in statistically significant higher bond strengths of composite to Y-TZP after thermal cycling and storage. The MDPS group produced intermediate bond strengths, greater than those associated with the SAND, MZP and MZP-RL, and the CRTL and CRTL-RL groups (Figure 1; Table 4).

DISCUSSION

The shear test is an acceptable alternative for bonding tests when other methods have little viability. In a pilot study, the attempt to produce bar specimens for microtensile bond testing resulted in de-bonding between the composite and Y-TZP during the cutting procedures, which was attributed to the difficulty in cutting the Y-TZP ceramic (excessive vibration, heating and damage to the adhesive interface) and the low bond strength values between the composite and Y-TZP.

The literature[4,6,11,14,15] shows that aging has an important role with regard to the longevity of composite to Y-TZP bonding. Therefore, water storage and thermal cycling were performed in the present study before the shear test in order to simulate oral conditions.

Table 3: *Sources of Variation and Two-Way Analysis of Variance (ANOVA) for the Bond Strength Results (MPa)*

Sources of Variation	Degrees of Freedom	Sum of Squares	Mean Square	*F*-Value	*p*-Value
Resin liner	1	17.06	17.06	2.73	0.1017
Surface treatment	4	2130.97	532.74	85.42	0.0000
Resin liner × surface treatment	4	100.84	25.21	4.04	0.0046
Error	90	561.31	6.2		
Total	99	2810.18			

The use of no-treatment groups (CRTL and CRTL-RL) was necessary so that we had a baseline for the bond strength between an untreated Y-TZP surface and composite under the study conditions. Alcohol cleaning was performed to clean grease residues and other possible contaminants off the Y-TZP surface. "No treatment" resulted in null and very low (2.17-MPa) bond strengths (for the CRTL and CRTL-RL groups, respectively). Similar results had already been reported in earlier studies.[4-6] This indicates the necessity of procedures designed to improve the bond strength between composite resins and Y-TZP. This weak bond strength is attributed to the absence of mechanical interlocking and chemical bonding,[19,20] which can favor water penetration and de-bonding after a certain storage time.[21] However, in a clinical situation, in which the Y-TZP surface has some degree of roughness caused by machining, bonding might not be as weak as indicated in the current results, which were obtained using a polished Y-TZP surface.

MDP presents ester phosphate groups, which supposedly can bond directly to oxides of the ceramic surface and to the methacrylate groups of the composite matrix.[22] Some authors[4,11,12] report good bonding results to Y-TZP surfaces when using resin cements containing MDP. These good results are the reason for the inclusion of the RL and MDPS treatment options in the current study (prior to the application of the composite as a repairing material).

It was expected that the RL would allow both an improvement in bonding to Y-TZP and a chemical bond to the resin composite. However, RL did not have a significant effect on the composite/Y-TZP bonding in all groups. Table 4 shows that when comparing groups with the same treatment with and without RL, all pairs were statistically similar. Lüthy et al.[23] also found a small, but not significant, increase in the bond strength between an MDP-based dual resin cement and Y-TZP. Additionally, MDPS did not have a positive effect on the

Table 4: *Mean and Standard Deviation Values of Bond Strength (MPa) for the Tested Y-TZP–Composite Bonding Approaches, Failure Modes (%), and Homogeneous Groups*

Surface Treatment	Bond Strength, MPa	Failure Mode, %	Homogeneous Groups[a]
TBS-RL	13.97 ± 4.58	100 CR	A
TBS	11.61 ± 2.12	90 CR; 10 A	A
MDPS-RL	2.98 ± 1.32	100 A	BC
MDPS	5.75 ± 4.13	80 CR; 20 A	B
SAND-RL	3.03 ± 3.02	100 CR	BC
SAND	0.65 ± 1.77	90 CR; 10 A	C
MZP-RL	0	100 A	C
MZP	0	100 A	C
CRTL-RL	2.17 ± 2.41	100 A	C
CRTL	0	100 A	C

Abbreviations: A, adhesive failure; CR, cohesive failure into the composite resin; CRTL, "no treatment" control; MDPS, methacryloxydecyldihidrogenphosphate (MDP)–containing primer/silane; MZP, metal/zirconia primer; RL, MDP-containing resin cement liner; SAND, sandblasting; TBS, tribochemical silica coating.
[a] Tukey ($p<0.05$): different online small-capital letters indicate statistical difference.

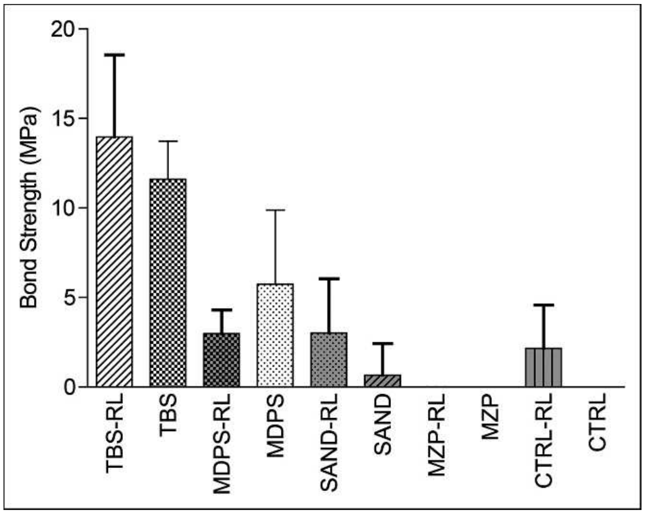

Figure 1. *Mean shear bond strength (MPa) and standard deviation (bars) of composite to Y-TZP for the different bonding approaches.*

composite/Y-TZP bonding. Similar findings were reported by Kern et al.,[24] for whom the application of an MDP-containing primer resulted in spontaneous failure between composite and Y-TZP during 150 days of storage. These findings seem to indicate that any possible reaction between MDP (from cement liner or primer/silane) and Y-TZP oxides is not enough to promote a significant increase in the bond strength. Therefore, the application of RL would only represent one more step in the repairing procedure, without a significant improvement in bonding.

TBS resulted in significantly higher bond strengths of composite to Y-TZP (13.97 MPa and 11.61 MPa for TBS-RL and TBS groups, respectively). Silica coating is a surface treatment in which aluminum-oxide particles modified with silica are blasted under pressure onto the ceramic surface, resulting in the embedding of silica particles on the ceramic surface.[25] The TBS system includes this air abrasion procedure associated with the application of a coupling agent (silane), which may result in chemical bonding between the silica-coated ceramic surface and the resin composite through cross-links with the methacrylate groups. Silane agents may also increase the ceramic surface energy, improving the wettability of the resin.[19,26,27] TBS has been reported to improve the bond strength of resin cements to alumina[25,28] and zirconia[18,25,29] ceramics. When using 90 days of water storage and thermal cycling, May et al.[14] and Passos et al.[15] verified stable and higher bond strength values between resin cements and Y-TZP when TBS was used, but lower or null bond strength values when no TBS was used. These findings substantiate the results found in the current study and indicate that TBS can be very useful for improving composite bonding to Y-TZP–exposed surfaces in cases that require an immediate repair of chipped restorations. The mechanisms likely associated with this enhancement of bonding are the fine surface roughness, enlargement of the bonding area,[26,30,31] and the chemical bond between the silica layer on the Y-TZP surface and the silane agent.[19,21,25,26,30-32] Figure 2c shows a micrograph of the Y-TZP surface after TBS treatment in which a fine complex of embedded silica particles were silanated and are available for micromechanical interlocking and chemical bonding to the composite.

Airborne alumina particle abrasion was not effective in improving the bond strength to Y-TZP (3.03 MPa and 0.65 MPa for SAND-RL and SAND groups, respectively). When comparing sandblasting to TBS for surface treatment of glass-infiltrated and polycrystalline ceramics, Bottino et al.[19] and Valandro et al.[33] found higher bond strength values when TBS was used. However, Wolfart et al.,[4] Oyagüe et al.,[11] Blatz et al.,[12] and Yoshida et al.[13] reported that SAND improved composite bonding to Y-TZP in short-term studies. Casucci et al.[34] showed that sandblasting with 125-μm particles (under 60 to 100 psi) did not alter the Y-TZP surface roughness. In the current study, the alumina particle size was 50 μm. Clearly, there were some topographic changes in the Y-TZP surface, as can be seen in Figure 2b. However, these changes were not significant enough to cause an increase on the bond strength after aging. Kern et al.[24] showed that the isolated treatment with alumina sandblasting (50-μm particle size) caused an immediate increase in bond strength between resin cement and Y-TZP; however, the bond strength decreased to 0 MPa after 150 days of storage. Sandblasting with the 50-μm alumina size seems to have an effect in the short term as a result of surface changes. However, this effect does not seem to be lasting. Lack of chemical bonding and possible water leakage in the adhesive interfaces could be involved.

MZP is a single-component primer that contains a phosphonic acid compound (phosphonic acid acrylate) as the active ingredient. According to the manufacturer's information, MZP establishes a chemical bond to oxidic surfaces, such as metal alloys or oxide ceramics (zirconium oxide, aluminum oxide) and methacrylate-based luting composites. In the current study, the eventual chemical bond was not enough to tolerate the storage/thermal cycling conditions, since all specimens treated with MZP

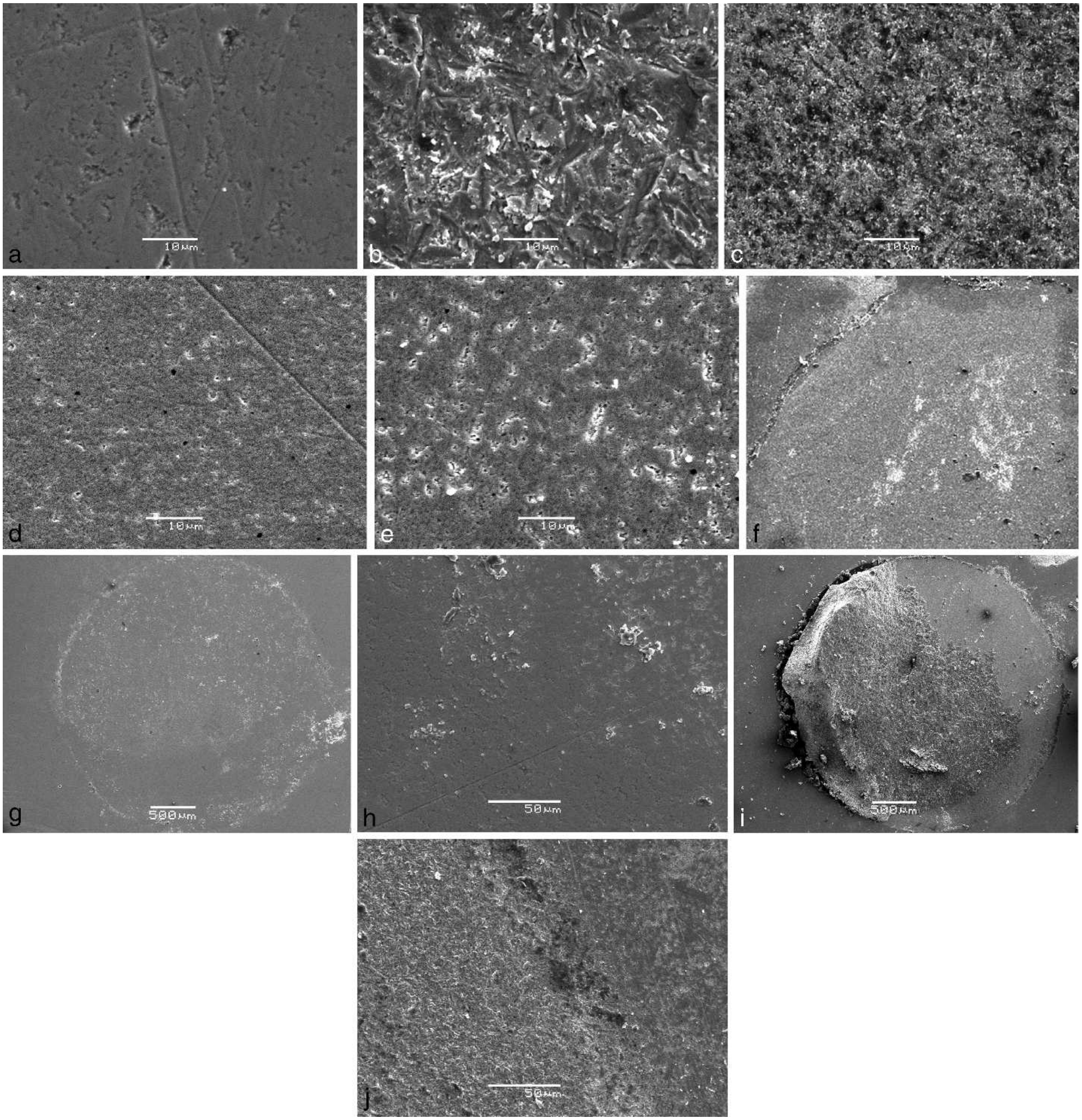

Figure 2. *SEM images: (a) polished Y-TZP surface; (b) sandblasted Y-TZP surface; (c) silica-coated Y-TZP surface; (d) MDPS-treated Y-TZP surface; (e) MZP-treated Y-TZP surface; (g, h) adhesive failure, MZP group; (i) resin cohesive failure, TBS-RL group; (j) resin cohesive failure (left side), TBS group. Dashed line indicates bond boundary.*

presented de-bonding before the shear test, producing null bond strength values for both the MZP and MZP-RL groups.

All surface treatments evaluated in this current study, with or without the application of an RL, with the exception of TBS, presented low or null values for bond strength after thermal cycling and 60-day water storage. These results confirm the weak bond between composite and Y-TZP and seem to indicate that tribochemical silica coating is the best strategy for promoting bonding when a composite repair procedure is possible and indicated in order to restore the function of a chipped restoration and delay the exposure of the Y-TZP framework to water from the oral environment. In addition to the positive effect of TBS on the bond strength, this treatment seems to not affect the fatigue strength of Y-TZP.[35]

When looking at the failure types in Table 4, adhesive failures occurred mainly in the CRTL and MZP groups. These groups presented a high incidence of spontaneous de-bonding during the storage/thermal cycling period. The TBS, TBS-RL, MDPS, SAND, and SAND-RL groups presented cohesive failures in resin. It is supposed that the higher bond strength values between the Y-TZP surface and the resin liner or the repairing composite increased the possibility of cohesive failure occurrence, typical of a shear test configuration.

The first hypothesis was accepted, as the TBS groups provided the highest bond strengths for composite to Y-TZP. This treatment and MDPS had an effect that was different than that associated with the CRTL groups. However, the liner application had no significant effect on the bond strength between composite and Y-TZP. Therefore, the second hypothesis was rejected.

CONCLUSIONS

Considering the current study conditions, the use of an MDP-containing cement liner between a composite and Y-TZP surface is not effective and not recommended. Tribochemical silica coating seems to offer the best strategy as a Y-TZP surface treatment for composite repairs when there is an exposure of this ceramic to the oral environment.

Conflict of Interest Declaration

The authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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