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Effect of Self-adhesive Resin Cement and Tribochemical Treatment on Bond Strength to Zirconia

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Abstract

Aim To evaluate the interactive effects of different selfadhesive resin cements and tribochemical treatment on bond strength to zirconia.

Methodology The following self-adhesive resin cements for bonding two zirconia blocks were evaluated: Maxcem (MA), Smartcem (SM), Rely X Unicem Aplicap (UN), Breeze (BR), Biscem (BI), Set (SE), and Clearfil SA luting (CL). The specimens were grouped according to conditioning as follows: Group 1, polishing with 600 grit polishing paper; Group 2, silica coating with 110 μ m Al₂O₃ particles which modified with silica; and, Group 3, tribochemical treatment - silica coating + silanization.

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Introduction

All-ceramic restorations are metal-free alternatives to widespread metal-ceramic composite structures (Anusavice, 1993). The interest in using high-strength zirconium oxide ceramics for oral rehabilitation has been growing in recent years (Guazzato et al., 2004; Tsalouchou et al., 2008). These high-strength ceramics offer a wide variety of clinical applications, such as fixed-partial dentures (FPDs), posts or implant abutments in prosthetic dentistry. Zirconia has the most favourable properties, with a flexural strength of 900 to 1200 MPa, fracture resistance of more than 2 000 N, and fracture toughness of 9-10 MPam^{0.5}, which is almost twice that of alumina-based materials (Tinschert et al., 2000). CAD/CAM technologies have made working with this high crystalline material simpler, allowing the fabrication of fullSpecimens were stored in distilled water at 37°C for 24 hours before testing shear bond strength.

Results Silica coating and tribochemical treatment significantly increased the bond strength of the MA, UN, BR, BI, SE and CL to zirconia compared to #600 polishing. For both #600 polished and silica coating treatments, MDP-containing self-adhesive resin cement CL had the highest bond strengths to zirconia.

Conclusion Applying silica coating and tribochemical treatment improved the bond strength of self-adhesive resin cement to zirconia, especially for CL.

Keywords self-adhesive resin cement, zirconia, silica coating, tribochemical treatment, bond strength

coverage crowns or bridge frameworks (Luthardt et al., 1999).

Bonding properties are important for the adhesive stability of dental restorative materials. Reliable adhesion to ceramics conventionally requires surface pre-treatments. Although cementation of zirconia restorations with traditional luting cements (such as zinc phosphate or glass ionomer cements) may provide adequate clinical fixation, adhesive cementation is preferable to ensure better retention and marginal adaptation (Derand *et al.*, 2005; Atsu *et al.*, 2006).

Recently, so-called universal, all-purpose, or multipurpose self-adhesive resin cements have been available, each purportedly bonding to enamel, dentin, amalgam, metal, and porcelain (Blatz *et al.*, 2007; Ibarra *et al.*, 2007; Mazzitelli *et al.*, 2008; Fawzy *et al.*, 2009). Self-adhesive resin cements that rely on a single step application, have also been proposed for luting zirconia-based restorations (Ernst et al., 2005; Kumbuloglu et al., 2006). For the bonding of conventional silica-based ceramics, it has been well established that adhesive bonding (hydrofluoric etching and silanization) enhances the resin bond (Derand et al., 2000). It has been reported that applying a sandblast, combined with a phosphate monomer containing luting agent, is a recommended option for ensuring the success of bonding to zirconia, as it enhances the acidic functional monomers capability of chemically reacting with the substrate (Kern et al., 1998). Results with bonding of an untreated zirconia surface, however, remain controversial. The resin matrix of these self-adhesive resin cements consists of multifunctional acid methacrylates (De Munck et al., 2004). If a high content of acidic functional monomers can react with the substrate and achieve enough chemical bond strength, we can hypothesize that these self-adhesive resin cements could be used to bond successfully to zirconia without surface treatment. The aim of this study was to evaluate the effect of different self-adhesive resin cements and tribochemical treatment on bond strength to zirconia.

Materials and Methods

Specimen preparation

Two zirconia blocks of different size ($10 \text{ mm} \times 10 \text{ mm} \times 20 \text{ mm}$ and $10 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$, 5.03% (by weight) Y₂O₃ - 94.67% (by weight) ZrO₂, Nikkato, Japan) were fabricated for the study (*N*=210). They were ground with 600 grit silicone carbide polishing paper (DCCS, Sankyo Fuji star, Japan) under water cooling and ultrasonically cleaned in acetone and distilled water for 15 minutes.

Zirconia blocks were randomly divided into three groups according to the different mechanical and/or chemical treatment performed: Group 1 (#600), no surface treatment; Group 2 (silica coating), the specimens were air-abraded with high-purity aluminium oxide 110 μ m, modified with silica (Rocatec Plus, 3M ESPE, USA) at a pressure of 0.28 MPa from a distance of approximately 10 mm for 20 seconds in circling movements; Group 3 (Tribochemical treatment), following the Silica coating, the remnants of sand particles were gently air blown, silane coupling agent (Clearfil ceramic primer, Kuraray medical, Japan) was applied, and then evaporation for 5 minutes was allowed.

Then, the specimens were divided into seven subgroups (*n*=10) according to the cement type. The following self-adhesive resin cements for bonding zirconia (Table 1) were used in this study: Maxcem (Kerr, USA; MA), Smartcem (Dentsply-Sankin, Japan; SM), Rely X Unicem Aplicap (3M ESPE, USA; UN), Breeze (Pentron clinical, USA; BR), Biscem (Bisco, USA; BI), Set (SDI, Australia; SE) and Clearfil SA luting (Kuraray, Japan; CL).

Bonding procedure

After appropriate surface treatment, each adhesive resin cement was applied according to the manufacturers' instructions at room temperature $(23.0 \pm 1.0^{\circ}\text{C})$ and relative humidity $(50\% \pm 5\%)$.

Application mode and chemical composition of the investigated materials are reported in Table 1. Two zirconia blocks of different size were bonded to each other under the load of 147 N (15 kg) for 15 minutes in order to standardize the applied pressure. The excess resin cement was removed by means of a laboratory knife. The resin cement was then light polymerized (G-Light, GC, Japan; 1 200 mW·cm⁻² light intensity) from each direction. Specimens were stored in distilled water at 37°C for 24 hours before testing shear bond strength.

Shear bond strength test

Shear bond strength was determined according to ISO/TS 11 405:2 003 using a Universal Testing Machine (Servopulser EHF-FDI, Shimadzu, Japan) at a crosshead speed of 0.5 mm·min⁻¹. The force at separation (N) was divided by the cross-section area (100 mm²) to provide results in units of stress (MPa).

Representative interfacial morphologies of surface treatment and debonded specimens were examined in a Scanning electron microscopy (SEM) (S-4000, Hitachi, Japan) with an acceleration voltage of 5 kV after sputtering using a gold-palladium alloy conductive layer (Ion sputter E-1030, Hitachi,

Product/Code/Lot No./Manufacturer	Main composition	Application		
Resin cements				
Maxcem/MA/2772209/Kerr (California,	Bis-GMA, UDMA, TEGDMA, GPDM, barium	Mix cement through a dual-barrel syringe.		
USA)	glass filler, fluoroaluminosilicate glass filler, fumed silica	Apply, light-cure for 20 seconds from each side.		
Smartcem/SM/431-022/Dentsply-	methacrylic acid, HEMA, trimellitic acid,	Mix cement through a dual-barrel syringe.		
Sankin (Tokyo, Japan)	4-MET, PENTA, PEM-F, BDMA, sulfinate	Dispense cement onto mixing pad and mix for 20 seconds. Apply, light-cure for 30 seconds from each side.		
Rely X Unicem Aplicap/UN/304133/	dimethacrylate, acetate , methacrylated	Insert capsule into activator. Press down		
3M ESPE (Minnesota, USA)	phosphoric ester, glass powder, silica,	handle completely. Insert activated capsule		
	calcium hydroxide	into mixing device. Mix 10 seconds. Apply,		
		light-cure for 30 seconds from each side.		
Breeze/BR/162835/Pentron clinical	Bis-GMA, UDMA, TEGDMA, HEMA, 4-MET,	Mix cement through a dual-barrel syringe.		
(Connecticut, USA)	silane treated barium glass, silica (amorphous), Ca-Al-F-silicate	Apply, light-cure for 20 seconds from each side.		
Biscem/BI/700009638/Bisco (Illinois,	TEGDMA, HEMA, phosphate, dental glass	Mix cement through a dual-barrel syringe.		
USA)		Apply, light-cure for 20 seconds from each side.		
Set/SE/ S0711272/SDI (Victoria,	UDMA, phosphate, fluoroaluminosilicate	Insert capsule into activator. Press down		
Australia)	glass, silica,	handle completely. Insert activated capsule		
		into mixing device. Mix 15 seconds. Apply,		
		light-cure for 20 seconds from each side.		
Clearfil SA luting/CL/0005AA/ Kuraray	Bis-GMA, TEGDMA, MDP, barium glass,	Mix cement through a dual-barrel syringe.		
medical (Tokyo, Japan)	silica, sodium fluoride	Apply, light-cure for 10 seconds from each side.		
Silane coupling agent				
Clearfil ceramic primer/ /0001AA/	γ-MPTS, MDP, ethanol	Apply primer on zirconia block and air-dry		
Kuraray medical (Tokyo, Japan)		for 5 seconds.		

GPDM: glycerol dimethacrylate dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; 4-MET: 4-methacryloxyethyl trimellitic acid; PENTA: phosphoric acid modified acrylate resin; PEM-F: monofluoro phosphazene modified methacrylate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; BDMA: 1,3-butyleneglycol dimethacrylate; MDP: 10-methacryloyloxy-decyl dihydrogenphosphate; γ-MPTS: γ-methacryloxypropyltrimethoxysilane.

Japan).

Statistical analysis

Statistical analyses of the test results were undertaken with the Kruskal–Wallis test followed by multiple pair-wise comparisons of the groups using the Wilcoxon rank sum test for independent samples. Significance levels were adjusted with the Bonferroni–Holm correction for multiple testing. Non-parametric statistical test procedures were used for group comparisons, because Bartlett's test showed inhomogeneity of variances between groups and the data was found to be non-normally distributed. *P*-values less than 0.05 were considered to be statistically significant in all tests. The statistical analysis was performed using SPSS 10.0 software for Windows (SPSS 10.0, SPSS, USA).

Results

Kruskal–Wallis test indicated that bond strengths were significantly influenced by the resin cement

and surface treatment (P < 0.05). The shear bond strength values and the results of multiple comparisons were summarized in Table 2 for all seven resin cements and three surface treatments (P < 0.05). The mean values were (7.27 ± 0.66)– (17.69 ± 2.81) MPa for #600, (11.26 ± 2.76)– (31.38 ± 3.59) MPa for silica coating, and (15.32 ± 2.81)–(50.91 ± 6.56) MPa for tribochemical treatment. The bond strength of the MDP-containing self-adhesive cement (CL) to #600 polished zirconia was relatively high with a mean of (17.69 ± 2.81) MPa. The use of the BR on tribochemical treatment resulted in the highest bond strength values (50.91 ± 6.56) MPa.

Specimens conditioned with aluminium oxide $110 \mu m$, modified with silica showed a change in surface texture with a silica layer (Figure 1A). Fractured surfaces of #600, silica coating and tribochemical treatment luted with BR were shown in Figure 1B, C and D, respectively.

-	#600		Silica coating		Tribochemical treatment	
Groups	Medians	Means ± SD	Medians	Means ± SD	Medians	Means ± SD
MA	8.79 ^{A,B} a	9.15 ± 1.70	13.98 ^B b	13.79 ± 2.62	31.13 ^A c	31.94 ± 6.49
SM	8.24 ^{A,B} a	8.21 ± 1.81	11.25 ^B b	11.26 ± 2.79	15.53 ^D c	15.32 ± 2.81
UN	7.36 ^A a	7.54 ± 0.64	18.75 ^D b	19.01 ± 3.48	44.34 ^{ABC} c	42.47 ± 6.98
BR	9.89 ^B a	10.19 ± 1.81	24.48 ^A b	25.76 ± 4.88	50.21 ^C c	50.91 ± 6.56
BI	7.82 ^A a	7.58 ± 1.17	27.28 ^A b	25.35 ± 4.26	45.02 ^{BC} c	46.39 ± 8.61
SE	7.47 ^A a	7.27 ± 0.66	28.06 ^{A,C} b	27.39 ± 4.07	36.59 ^{AB} c	36.48 ± 6.69
CL	16.70 ^C a	17.69 ± 2.81	31.17 ^C ♭	31.38 ± 3.59	38.45 ^{AB} b	37.05 ± 6.46

Table 2 Bond strength of self-adhesive resin cements to zirconia. Medians, means and standard deviations in MPa (n=10)

SD: standard deviations. Within the same column, medians with the same upper case superscript letter are not statistically different (P>0.05). Within the same row, medians with the same lower case subscript letter are not statistically different (P>0.05). Kruskal–Wallis test followed by pairwise comparison using the Wilcoxon test modified by Bonferroni.



Figure 1 SEM microphotographs (× 2 000) of silica coating and fractured surfaces luted with BR

(A): Silica coating. Specimens conditioned with aluminium oxide 110 µm, modified with silica showed a change in surface texture with a silica layer. (B): Fractured surfaces of #600 luted with BR. A complete detachment from the substrate occurred. (C),(D): Fractured surfaces of silica coating and tribochemical treatment luted with BR, respectively. Cement residuals are detectable on the bonded zirconia surface.

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Discussion

Resin cement selection seems to be a more relevant factor in bonding to zirconia. In the #600 group, the application of MDP-containing selfadhesive cement (CL) attained the best bonding results. CL specimens containing MDP monomer exhibited significantly higher bond strength than MA, SM, UN, BI and SE specimens containing phosphoric ester, as well as BR specimens not containing phosphoric ester. The adhesive potential of MDP to zirconia may depend on the presence of a passive coating of zirconium oxide on the ceramic surface. Chemical reactions involving the hydroxyl groups of the layer and the phosphate ester monomers of the MDP may occur at the interfacial level (Kern et al., 1998; Wolfart et al., 2007). Moreover, such a promising behavior is reported the effectiveness of Panavia 21 with similar chemical composition (Palacios et al., 2006; Valandro et al., 2006; Valandro et al., 2007). Therefore, the MDP containing self-adhesive resin cement CL is more suitable to bond the zirconia surface than other self-adhesive resin cements. Laboratory tests are important to determine some characteristics of the materials, however, as these results cannot be directly related to the clinical situation. Moreover, the groups of silica coating and tribochemical treatment showed higher bond strengths than #600. Accordingly, final long-term clinical evaluations of these self-adhesive resin cements are needed.

Significant differences in bond strength were found between 4-MET containing BR specimen and GPDM containing MA specimen, in spite of the fact that these two specimens have very similar resin matrix compositions and fillers. The difference may be attributable to the adhesive monomer each self-adhesive resin cement contains.

Though SM contains adhesive monomer (4-MET) and polymerization initiators such as BPO/ tertiary amine, sulfinic acid, the bonding interface of SM specimen after 24-hour water storage was easily removed with a laboratory knife, displaying a white powdery state. It might be that SM showed the lowest bond strengths and a greater degree of leakage because of inadequate curing.

Untreated zirconium oxide ceramic is a rela-

tively inert substrate with low surface energy and wettability (Ozcan, 2003). It is reported that atomic force microscopy analysis reveals a significant increase in surface roughness after sandblasting with 125 µm aluminum-oxide particles (de Oyague et al., 2009). Silica coating deposited a silica layer on the ceramic surface due to the high-pressure impaction of alumina particles modified by silica on the conditioned substrate. Silica coating should promote chemical bonding at the ceramic-resin cement interface (Kumbuloglu et al., 2006; Valandro et al., 2007). A study to survey improvement to the resin-ceramic (a silica-based ceramic) bond with the aid of silane suggests no need for HF acid etching or other pre-treatment for the ceramic part when appropriate silane is used, as even the siloxane bond was inert against boiling water (Hooshmand et al., 2002; Matinlinna et al., 2004).

The bond strengths of the phosphate monomer containing resin composites to silica-coated, untreated zircona surface were statistically significantly higher when compared with the bond strength of the Bis-GMA resin cement MA which contains GPDM. The CL, SE, BR BI and UN specimens treated with silica coating showed bond strength values approximately 2-4 times as much as those specimens treated with #600. This increased bond strength might be explained by combined effects of mechanical interlocking, and the adhesiveness of phosphate monomercontaining cements or carboxylic acid monomer (4-META) increased bond strength, revealing the capability of acidic functional monomers to chemically react with the silica coating substrate.

In this study, using a tribochemical treatment resulted in a significant increase in the bond strength of the mostly resin cements to zirconia. Roughening the zirconia surfaces with silica coating followed by silanization evidently enhanced the bond between the self-adhesive resin cements and the ceramic surfaces. The silica layer left by silica coating on the ceramic surface provides a basis for silane to react. In the ceramic-resin bond, silane functions as a coupling agent, which adsorbs onto and alters the surface of the ceramic, thereby facilitating chemical interaction (Lu *et al.*, 2001; Ozcan, 2003).

When comparing bond strength of UN, BR and BI specimens, tribochemically treated specimens



showed 2-2.5 times higher bond strength values than those of silica coating specimens, exhibiting higher bond strength than CL specimen. CL specimens which obtained a mean bond strength value of (31.38 ± 3.59) MPa in silica coating did not show any improvement in spite of the additional application of silane coupling agent, demonstrating a minor increase of (37.05 ± 6.46) MPa. The formulations of MDP, Bis-GMA, UDMA are shown in Figure 2. Bis-GMA, UDMA are multifunctional methacrylate, and have two C=C groups at the end of the chair. However, MDP only have one C=C group. So, the polymerization of MDP will be a linear polymer and Bis-GMA, UDMA will be cross-linked polymer. Crosslinking has a profound effect on the properties of a polymer. Higher content of MDP monomer may have affected adversely on the mechanical strength of CL specimen.

Some earlier studies reported high and stable bond strength to the zirconia reinforced ceramic after airborne particle abrasion using Al_2O_3 particles in combination with phosphate monomer based resin cement (Andersson *et al.*, 1993; Piconi *et al.*, 1999; Strub *et al.*, 1988). Comparing the results of these studies with this present study, it can be suggested that the tribochemical treatment may allow a better bond strength to the zirconia with this resin cement.

Conclusion

Within the limitations of the present *in vitro* study, the following conclusions can be drawn:

(1) Applying silica coating and tribochemical treatment improves the bond strength of self-adhesive resin cement to zirconia.

(2) In the surface treatment of #600 polished and silica coating, MDP containing self-adhesive resin cement Clearfil SA luting shows the highest bond strengths to zirconia. The high content of MDP may have an effect on the mechanical strength of the cement.

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