EFFECT OF SURFACE TREATMENT METHODS ON THE SHEAR BOND STRENGTH OF RESIN CEMENT TO ZIRCONIA CERAMIC

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Statement of problem. The aims of the study were to evaluate the effect of current surface conditioning methods on the bond strength of a resin composite luting cement bonded to ceramic surfaces and to identify the optimum cement type.

Material and methods. The sixty zirconia ceramic specimens(10 per group) with EVEREST milling machine and 60 tooth block were made. The zirconia ceramic surface was divided into two groups according to surface treatment: (1) airborne abrasion with 110μm aluminum oxide particles; (2) Rocatec system, tribochemical silica coating. The zirconia ceramic specimens were cemented to tooth block using resin cements. The tested resin cements were Rely X ARC, Panavia F and Superbond C&B. Each specimen was mounted in a jig of the universal testing machine for shear strength. The results were subjected to 2-way ANOVA and Post hoc tests was performed using Tukey, Scheffe, and Bonferroni test.

Results. The mean value of shear bond strength(MPa) were as follows:
RelyXARC(+Al_2O_3),5.35 ± 1.69; RelyXARC(+Rocatec),8.50 ± 2.13;
PanaviaF(+Al_2O_3),9.58 ± 1.13; PanaviaF(+Rocatec),12.98 ± 1.71;
SuperbondC&B(+Al_2O_3),8.27 ± 2.04; SuperbondC&B(+Rocatec),14.46 ± 2.39.
There was a significant increase in the shear bond strength when the ceramic surface was subjected to the tribochemical treatment(Rocatec 3M) in all cement groups($P<0.05$). Bonding strengths of cements applied to samples treated with Al_2O_3 were compared; Rely X ARC showed the lowest values, whereas Panavia F cement showed higher value than that of Superbond C&B group with no statistical significance. When the bond strength of cements with Rocatec treatment was compared, Rely X ARC showed lowest values. Overall, it was apparent that tribochemical treated Super-Bond possessed higher mean bond strength.(14.46MPa; $P<0.05$) than that of Panavia F cement group with no significance.

Conclusions. Silica coating followed silanization(Rocatec treatment) increase the bond strength between resin cement and zirconia ceramic. Panavia F containing phosphate monomer and Superbond C&B comprised of 4-META tend to bond chemically with zirconia ceramic, thus demonstrating higher bond strength compared to BisGMA resin cement. Superbond C&B has shown to have highest value of bonding strength to zirconia ceramic after Rocatec treatment compared to other cement.

Key Words
Zirconia ceramic, Bond strength, Surface treatment, Resin cement

In recent years, an increasing demand for esthetic restorations led to the development of several new ceramic systems with characteristics similar to natural teeth, such as translucency and fluorescence. New ceramic system involve reinforced ceramic cores through dispersion with leucite,[1-6] glass infiltration into sintered alumina(Al_2O_3),[7,8] the use of high purity alumina[9] or zirconia ceramics.[10]

Above all, zirconia ceramics is the most recently introduced dental all-ceramic material. It shows a much higher strength and toughness than all other commercially available dental ceramics.[11,12] Moreover due to the recent progress in CAD/CAM, it was also possible to manufacture crowns and multiple-unit dental bridge zirconia ceramics through in vitro[13] and clinical tests.[14]

To enhance the bond strength of luting cement to the ceramic surface, a number of techniques have been reported which mechanically facilitate resin-ceramic bonding. Hydrofluoric acid etching and silanization can enhance the mechanical bond strength of glass-ceramic materials to composite resin. The acid etching partially removes the glassy matrix and create mechanical retentive areas where silane molecules lodge and link inorganic molecules of the resinous material.[15-18]

However, these techniques are limited only to feldspathic ceramics. All-ceramic coping materials such as alumina and zirconia ceramic are not sufficiently roughened by airborne-particle abrasion or etched with hydofluoric acid, and do not sufficiently react with a silane coupling agent due to their low silica content.[19-24] The silica content of alumina ceramics is below 5 wt% and that of zirconia ceramics is below 1 wt%, compared with that of conventional feldspathic ceramics, which is 50 to 60 wt%. Silane agents, therefore, do not react effectively with alumina ceramic and zirconia ceramic materials.[25]

There have been many studies on methods of zirconia ceramic bonding. Some studies has shown that a tribochemical silica coating may increase the bonding strength between resin cement and high-strength ceramics.[23-28] In this technique, the surfaces are air-abraded with aluminum oxide particles modified with sialic acid. The blasting pressure results in the embedding of silica particles on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents. Silane molecules, after being hydrolized to silanol, can form polysiloxane network of hydroxyl groups covering the silica surface. Monomeric ends of the silane molecules react with the metyacrylate groups of the adhesive resins by free radical polymerization process. In this way, it improves the bond between the silica adhered to the substrate and the resin matrix. On the other hands, Kern and Wegner[36] stated that phosphate monomer containing adhesive resin composite cement showed more durable bond by far regardless of tribochemical silica coating. Some studies show that airborne particle abrasion and use of a modified BisGMA resin luting agent, containing the adhesive phosphate (MDP), provided a long-term durable resin bond to zirconium-oxide ceramic.[20,36,43] While Uo et al.[29] found that a glass ionomer cement produced a more significant superior bonding to zirconia ceramics than a phosphate monomer containing adhesive resin composite. Also, pointed out that a 4-META containing adhesive resin (Superbond C&B, Sun Medical, Kyoto, Japan) has a bond strength superior by far to Panavia.[21,38]

As noted before, comparative studies exist in the literature, showing the advantages of various types of surface conditioning methods on various ceramics. There has been, however, no consensus in the literature regarding the best surface conditioning method for optimum bond strength depending on the luting cements or ceramics used. Also, the experiment reported here may be useful as other studies involved bonding strengths between cement and zirconia without the use of tooth-cement-zirconia ceramic.[31,32] Therefore, the aims of the study were to evaluate the effect of current surface conditioning methods on the bond strength of resin composite luting cement bonded to ceramic surfaces and to identify the optimum cement type.

MATERIAL AND METHODS

1. Tooth preparation

Intact caries-free human premolar and molar extracted from individuals 18-73 years old, were stored in 0.5% chloramine T solution (antimicrobial preservative). The teeth were used within 2 months after extraction. In this study, the age difference among the collected teeth was ignored since a previous study showed that age did not greatly influence the dentin bond strength.[30]

Each tooth was embeded in an autopolymerizing acrylic resin (Paladur; Heraeus Kluser, Hanau, Germany). Each tooth was sectioned perpendicular to its long axis, 2mm cervical to the occlusal pit using a low-speed diamond saw(Isomet; Buehler, Lake Bluff, IL, USA) under running water. The crown portion was discarded, and the remaining exposed section was inspected to ensure that all of the occlusal enamel had been removed and pulp horns had not been perforated. The flat occlusal dentin and circumferential enamel surface were abraded with 600-grit silicon carbide paper under running water for 10 seconds to create a standardized smear layer. Then, dentin specimens were stored in distilled water at 4℃. The dentin specimens from each region were randomly divided into the 6 groups 10 teeth for bonding tests.

2. Production of zirconia-specimens

The 60 zirconium oxide ceramic specimens (4mm diameter, 5mm height) were milled from presintered ceramic blocks with the Everest milling machine (Everest; KaVo, Germany).

3. Surface conditioning of zirconia-specimens

Conditioning 1 (110μm Al_2O_3 blasting)

Airborne-particle abrade aspects of zirconia ceramic with 110μm aluminum oxide at 2.8-bar for 40 seconds from a distance of 10mm.

Conditioning 2 (tribochemical silica coating)

The first step of the tribochemical treatment was commonly carried out with Rocatec-Pre (3M ESPE) to clean the material surface with 110 μm aluminum oxide airborne-particle abrasion. In the second step, Rocatec Plus (3M ESPE, Seefeld, Germany) airborne-particle abrasion was performed at 2.8-bar pressure with the instrument tip held perpendicular to the zirconia specimens at a distance of 10mm. Next, silane (Rocatec ESPE-Sil; 3M ESPE) was applied and allowed to air dry for 5 minutes.

4. Cementation

Throughout the experiments, the bonding procedures were carried out in accordance with manufactures' instructions. All materials were mixed and applied in a standardized way by the same operator(Table I).

Rely X ARC

Dispense the necessary amount of SE Bond Primer into a well of the mixing dish immediately before application and apply to the entire dentin of tooth block with a sponge. After conditioning the tooth surface for 20 seconds, evaporate the

Table I. List of used materials

Material	Type	Main composition	Manufacturer
Rocatec	Rocatec Pre	110㎛ Al_2O_3	3M Espe, Seefeld
	Rocatec Plus	Silica Cont. 110㎛ Al_2O_3	Germany
	Espe-Sil	Silane	
RelyX ARC	Base/Catalyst	BisGMA/TEGDMA/UDMA/BPEDMA, inorgnic fillers	3M Espe, Seefeld Germany
Panavia F	Base/Catalyst	BPEDMA/MDP/DMA, Ba-B-Si-glass, chemical and photoinitiators	Kuraray, Osaka, Japan
Superbond C&B	Base/Catalyst	4-META/TBB/PMMA containing resin	Sun Medical, Kyoto, Japan

4-META, 4-methacryloxyethyl-trimellitat-anhydrid; BPEDMA, Bisphenol-A-polyethoxy dimethacrylate; DMA, aliphatic dimethacrylate; MDP, 10-methacryloyloxy-decyl-dihydrogenphosphate; TBB, tri-n-butylborane; BisGMA, Bisphenol-A-Glycidyllmethacrylate; PMMA, polymethylmethacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

volatile ingredients with a mild oil free air stream, then apply BOND to the entire dentin of tooth block with a sponge. Light cure the Bond for 10 seconds. Two pastes were mixed and applied with apply thin, even layer to aspect of zirconia ceramic. The ceramic-cement-tooth set was placed into a press with the interface perpendicular to a vertical (800g/10min). Any excesses were removed before polymerization. The cement was light cured for 40 seconds at each margin(Elipar, 3M ESPE; light intensity 800mW/cm^2, distance 0). The set was then removed from the press, rinsed with air-water spray, and stored in distilled water at 37℃ for 24hours.

Panavia F

Panavia F dual-cure resin cement manipulated according manufacturer' s specification, was used to cement each ceramic block to its corresponding tooth block. The ceramic-cement-tooth set was placed into a press with the interface perpendicular to a vertical (800g/10min). Any excesses were removed before polymerization. The cement was light cured for 40 seconds at each margin(Elipar, 3M ESPE, Seefeld, Germany; light intensity 800mW/cm^2, distance 0), and Oxyguard (Kuraray, Osaka, Japan) was applied to the external edge of the interfaces. The set was then removed from the press, rinsed with air-water spray, and stored in distilled water at 37℃ for 24hours.

Superbond C&B

Dentin was etched with green activator for 10 seconds, rinse and air dry gently. The dentin of tooth block were prewet with 4META/MMA-TBB, mix liquid and powder with brush-on technique. Apply to dentin for bond the PMMA rod to dentin surface. Excesses, if any, were removed before polymerization. Then the ceramic-cement-tooth complex was placed into a press with the interface perpendicular to a vertical (800g/10min). Specimens were stored in distilled water at 37℃ for 24hours.

5. Shear bond strength testing

All 60 specimens were stored in distilled water at 37℃ for 24 hours before the shear strength testing. Specimens were mounted in a jig of the universal testing machine(Instron Corp, Canton, Massachusetts) and the shear force was applied to the adhesive interface until fracture occurred(Fig. 1). The specimens were loaded at a crosshead speed of 0.5mm / min.

The shear bond strengths were calculated and recorded using this formula :

σ (MPa)=L*9.8 / A

where 'σ' is the shear bond strength (MPa), 'L' is the load at failure(kgf) and 'A' is the adhesive area(mm^2).

6. Statistic Analysis

Statistical analysis was performed using SPSSs. The each shear bonding strength values were analyzed 2-way analysis of varience (ANOVA) and Post hoc tests was performed using Tukey, Scheffe, and Bonferroni test. P values less than 0.05 were considered to be statistically significant in all test.

RESULTS

Detailed shear bond strength values and standard deviation are listed in Table II. There was statistical significance in the test of between-subject effect cement, treatment and cement * treatment(Table III). * indicates interaction between cement and treatment. There was a significant increase in the shear bond strength when the ceramic surface was subjected to the tribochemical treatment(Rocatec 3M) in all cement groups(Table III).

In the multiple comparison of each cement, there was no statistical significant between Panavia F and Superbond C&B, but there was statistical significance between Rely X ARC and both cements(Table IV).

More positive interaction and greater bonding strength was found in Superbond C&B (14.46± 2.39MPa) rather than Panavia F(12.98±1.71MPa) when both cement was treated with Rocatec(Table III).

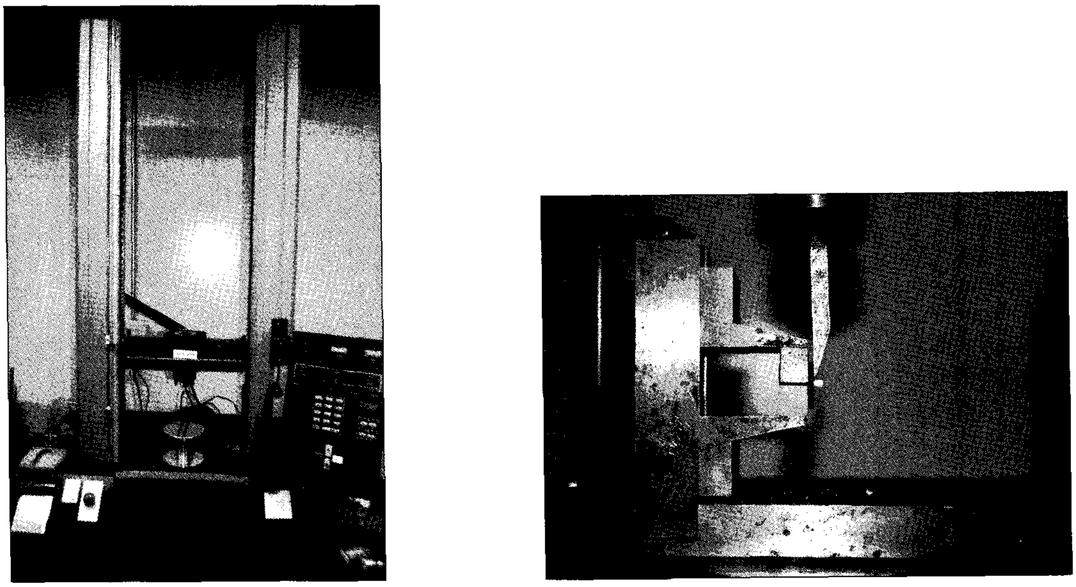

Fig. 1. Universal testing machine and shear bond test.

Table II. Mean bond strength(MPa) and standard deviation of tooth-cement-zirconia complex

cement	treatment	Mean	Std. Deviation	Number
Rely X	Al_2O_3	5.3550	1.69509	10
	Rocatec	8.5043	2.13645	10
	Total	6.9296	2.47654	20
Panavia	Al_2O_3	9.5800	1.13590	10
	Rocatec	12.9876	1.71795	10
	Total	11.2838	2.25054	20
Superbond	Al_2O_3	8.2738	2.04702	10
	Rocatec	14.4628	2.39682	10
	Total	11.3683	3.84525	20
Total	Al_2O_3	7.7363	2.41242	30
	Rocatec	11.9849	3.27974	30
	Total	9.8606	3.56889	60

Dependent Variable : Bonding Strength(Mpa)

Table III. Test of between-subject effects(cement, treatment, cement*treatment)

Source	df	Mean Squre	F	Sig.
Corrected Model	5	111.390	30.921	.000
Intercept	1	5833.855	1619.431	.000
CEMENT	2	128.892	35.779	.000
TREATMENT	1	270.765	75.162	.000
CEMENT*TREATMENT	2	14.202	3.942	.025
Error	54	3.942		
Total	60			
Corrected Total	59			

a. R Squared = .703(Adjusted R Squared =.687)
* Means interaction between cement and treatment

DISCUSSION

A requirement for the successful function of ceramic restorations over the years has been focused on adaquate adhesion between ceramic and tooth substance. Bond strengths are influenced by several factors one of which is the luting cement type.[40] Bonding of ceramic to tooth substance is based on the adhesion of luting cement and its bonding resin to the ceramic substrate together with the adhesion of luting cement to enamel and dentin. This study measured bond strength using zirconia ceramic-cement-tooth complex.

In this present study, the bonding of conventional BisGMA resin cement Rely X ARC to zirconia ceramic with no Rocatec treatment was weaker than other cements (5.35±1.69MPa). In contrast, using Rocatec treatment of the zirconia ceramic surface resulted in a significant increase in the

Table IV. Multiple comparision of each cement
Defendent Variables: Bonding Strength(MPa)

	(I)CEMENT	(J)CEMENT	Mean Difference(I-J)	Std. Error	Sig.
Tukey HSD	Rely X	Panavia	-4.3541*	.6002	.000
		Superbond	-4.4387*	.6002	.000
	Panavia	Rely X	4.3541*	.6002	.000
		Superbond	-8.45E-02	.6002	.989
	Superbond	Rely X	4.4387*	.6002	.000
		Panavia	8.45E-02	.6002	.989
Scheffe	Rely X	Panavia	-4.3541*	.6002	.000
		Superbond	-4.4387*	.6002	.000
	Panavia	Rely X	4.3541*	.6002	.000
		Superbond	-8.45E-02	.6002	.990
	Superbond	Rely X	4.4387*	.6002	.000
		Panavia	8.45E-02	.6002	.990
Bonferroni	Rely X	Panavia	-4.3541*	.6002	.000
		Superbond	-4.4387*	.6002	.000
	Panavia	Rely X	4.3541*	.6002	.000
		Superbond	-8.45E-02	.6002	1.000
	Superbond	Rely X	4.4387*	.6002	.000
		Panavia	8.45E-02	.6002	1.000

The mean difference is significant at the 0.5 level.

bonding strength of Rely X ARC to zirconia ceramic (8.50±2.13MPa). In specific, Rocatec treatment of zirconia ceramic significantly increase bond strength. 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. These findings of the present study are in agreement with those reported in the literature.[39-44] Kim[44] reported that for zirconia ceramic tested, airborne-particle abrasion with alumina particles and acid etching had little influence on providing reliable bond strengths between composite resin and ceramic materials. Silica coating technique, however, yielded the highest bond strength values to the composite resin. And, Luthy[43] noted that using Rocatec-treatment of the zirconia surface resulted in a significant of BisGMA to zirconia. After thermocycling, the bond strength did not decrease significantly. Wegner and Kern[39] reported that tribochemical silica coating of samples in Rocatec group resulted in a significant increase in bonding strength of the conventional bis-GMA resin composite to YPSZ ceramic. After 2years of water storage with thermocycling, however, the resin bond strength decreased significantly. On the other hand, the bond strength of Rely X ARC to zirconia was the lowest among other bonding strength specimens, with or without Rocatec treament. This results from conventional BisGMA resin cement does not adhesive monomer different from their cement which used this study.

In the present study, it was observed that the bond strength of Panavia F cement to zirconia cement with no Rocatec treatment was the highest (9.58±1.13MPa), but less significant than Super-bond C&B (8.27±2.04MPa). The Rocatec pre-treatment of the zirconium resulted in statistically significant increase in the bond strength values of more than 27%. In contrast to the conventional BisGMA resin composite, the bond strength of the MDP-containing resin composites was statistically significantly high, due to this active monomer. The phosphate ester group of the monomer bonds chemically to zirconium oxides. These findings of the present study are also in agreement with those reported previously in the literature. Bottino et al,[35] observed that silica coating, followed by silianization, increases the bond strength of zirconium-oxide ceramic to an MDP-containing composite resin relative to the use of airborne-particle abrasion with zirconium-oxide ceramic. Blatz et al,[37] demonstrated the application of an MDP-containing bonding /silane coupling agent mixture to zirconium-oxide ceramic restorations abraded with airborne Al_2O_3 particles can yield a superior shear bond strength. But, Kern and Wagner[36] observe that the use of monomer-phosphate-based resin cement allows a higher and stable bond strength to zirconia ceramic blasted with Al_2O_3 particles. These observations suggest that there could be an additional effect of the silica coating and chemical bond of the monomer phosphate to the zirconium oxide. Further studies are necessary to confirm these results. Superbond C&B treated with Al_2O_3 showed higher value than with Rely X ARC, which was statistically significant, whereas showed lower value than that of Panavia F. When an additional Rocatec pretreatment was used, Superbond C&B obtained the maximum bond strength. As shown in Table IV, there was interaction between treatment and cement. Superbond C&B was found to have higher values of bonding strength after Rocatec treatment compared to that of Panavia F. According to the result of this study, Superbond C&B has shown to have highest value of bonding strength to zirconia ceramic after Rocatec treatment compared to other cements.

The most plausible explanation for this high value is that Superbond contains 4-META/MMA-TBB resin. The anhydrid group of 4-META is supposed to have a chemical affinity to zirconia oxides and affinity to Rocatec treatment. Few studies have evaluated the effect of Rocatec treatment on the bond strength of zirconium oxide to Superbond C&B. Derand and Derand[38] evaluated that different surface treatments, as well as resin luting agent (Superbond C&B, Sun Medical) produced significantly higher bond strengths, regardless of surface treatment (silica coating, airborne-particle abrasion, HF etching, or grinding with a diamond bur). However, the present study showed that the groups treated with the Rocatec systems combined with Superbond C&B presented higher bond strength compared to the samples blasted with Al_2O_3 particles.

In this study, shear bond tests have been used, a commonly used bond strength test, fast and easy to perform and also reflects the clinical situation. It can be questioned if tension tests are more appropriate for evaluation the adhesive capabilities of resin agents to ceramics.[33] The fact that the cementation process was carried out using hand loading pressure without any type of standardized device might also have increased the overall SD of the results. On the other hand, this procedure was comparable to a true clinical situation in which the cementation pressure is generally controlled manually. The ceramic-material composition and intalgio surface configuration are specific for each commercial system. Therefore, conclusion drawn for one zirconia ceramic system may not be applicable to other systems that have different chemical composition and surface morphology. A study of the bond strength of the intalgio surface of Everest zirconia found that the combination of Rocatec treatment with Superbond C&B and Panavia F is best choice.

The present study suggested that the silica-coated surface (Rocatec system) could develop a better bond strength between the zirconia ceramic and resin cements because of the increase of silica content and the interaction with the siliane agent(Rocatec-sil) and, later, with resinous materials. Also, Panavia F resin cement containing adhesive phosphate monomer, and Superbond C&B resin cement containing 4-META could increase the bond strength to zirconia ceramic, and Superbond C&B has shown to have highest value of bonding strength to zirconia ceramic after Rocatec treatment compared to other cements.

The limitations of in vitro studies, which may not completely simulate in vivo performance, should be appreciated. Further research is needed to examine various surface treatments, longterm storage, thermomechanical cycling, zirconium oxide systems, and different luting cemcents, In addition long-term prospective. Randomized clinical trials are needed to evaluate the benefits of certain clinical procedures, including this innovative type of all-ceramic restoration.

CONCLUSION

Within the limitations of this study, the following conclusion were drawn :

1. Silica coating followed silanization(Rocatec treatment) increase the bond strength between resin cement and zirconia ceramic when compared with airborne particle abrasion using Al_2O_3.
2. Panavia F containing phosphate monomer and Superbond C&B comprised of 4-META tend to bond chemically with zirconia ceramic, thus demonstrating higher bond strength compared to BisGMA resin cement.
3. Superbond C&B has shown to have highest value of bonding strength to zirconia ceramic after Rocatec treatment compared to other cements.

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