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ORIGINAL RESEARCH ARTICLE

Evaluation of Shear Bond Strength of Different Resin Cements after Zirconia Surface Treatments

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Abstract

Purpose: The aim of the study was to evaluate the shear bond strength (SBS) of different resin cements after zirconia surface treatments.

Materials & Methods: A total of 60 zirconia discs (3x7mm) were prepared and divided into 3 main groups according to the surface treatments as control (C), sandblasting (SB), and tribochemical silica coating (TC). Main groups were divided into two subgroups according to two different resin cements were applied. No surface treatment was applied to the samples in C group. 50µm Al2O3 particles were applied to the samples in SB group for 10 s at a distance of 10 mm under 4 atm. TC group were tribochemically coated with alumina particles. Self-adhesive resin (ME) and multi-system dual-cure adhesive resin (NX3) was applied to the subgroups. After cementation, all samples were tested for SBS. SBS values were statistically analyzed by the Kruskal-Wallis and Mann-Whitney U tests.

Results: Regardless of the cement type, SBS values of the surface treated samples were statistically different (p < 0.001). Group SB was determined as the group with the highest SBS value. This group was followed by Group C and Group TC, respectively. The SBS values of the samples according to the resin cements and surface treatments were statistically significantly different (p<0.001). SBS values of the samples cemented with NX3 resin cement were found to be higher than the samples treated with ME resin cement.

Conclusion: SB increased resin bond to zirconia. It is more advantageous to use multi-system dual cure adhesive cements in zirconia cementation.

Key words: Resin cement; Shear bond strength; Surface Treatment; Tribochemical silica coating; Zirconia

Introduction

Ceramic fused to metal restorations have been used successfully for a long time. However, these restorations have disadvantages such as reflection of the porcelain-coated metal substructure on the gingiva, not allowing light transmittance in the anterior region, and corrosion. With the increased interest in esthetics, the use of zirconia material has increased due to its esthetic properties that reflect the natural tooth appearance. Zirconia is an attractive material for the fabrication of all-ceramic restorations due to its superior mechanical properties.¹ In addition to the esthetic properties and superior mechanical properties of zirconia material, it has come to the fore as a good alternative material to ceramic fused to metal restorations due to its good marginal compatibility, biocompatibility, and requiring less tooth preparation.²

Although it is superior in mechanical performance (strength, toughness, fatigue resistance), cementing zirconia with conventional cementation does not provide sufficient bond strength for most of the applications.³⁻⁵ Along with the durability of the material, the cementation process is very important for the success of the restoration.⁶ Örtorp et al.⁷ reported in their 3-year clinical follow-up study that the weakening of the connection of zirconia restorations was related to the cement used. Bond strength between tooth and zirconia can be improved by using resin cements. Thus, fracture resistance⁸, marginal adaptation², and retention of the restoration can be increased.⁹ Self-adhesive resin cements can provide a successful adhesion to enamel and dentin tissue without an adhesive system. Acid etching and silane application to conventional silica-based ceramics create a moist rough surface for successful ceramic-resin





Table 1. Characteristics of resin cements.

Code	Material	Туре	Delivery system	Monomer	Manufacturer
ME NX3	Maxcem Elite Self-adhesive Nexus third generation Dual cure-adhesive. Adhesive: OptiBond Solo Plus (total-etch) + gel-etchant		Automix-syringe Automix-syringe	HEMA / MA MA	Kerr, Bioggio, Switzerland Kerr, Bioggio, Switzerland

Abbrevations: HEMA: Hydroxyethyl Methacrylate, MA: methacrylic acid

adhesion.¹⁰ However, since zirconia is not silica-based, a solid silica-silane connection cannot be formed.¹¹ Therefore, alternative bonding techniques are required to obtain a strong, long-term, and durable resin bond with zirconia. Successful bonding between ceramic material and resin is possible by micromechanical locking and chemical bond formation on the ceramic surface.^{12,13} This success can only be achieved with a surface pretreatment method.^{14,15} Abrasion with sandblasting (SB) (Airborne-particle abrasion) applied using aluminum oxide (Al2O3) particles as a surface pretreatment method creates a rough area on the ceramic surface and changes the area at a micro level.¹⁶ Surface energy decreases and wettability on the ceramic surface increases as a result of abrasion with Al2O3 particles used in SB.¹⁷ SB, which strengthens the micromechanical connection as a result of irregular surface formation by increasing surface roughness and adhesion energy, is a conventional method that is frequently applied to increase the bonding of ceramic and resin.¹⁷

Different surface conditioning processes have been used as an alternative to SB for the last 20 years.¹⁸ Among these, one of the popular methods is the tribochemical silica coating (TC), which can be easily applied intraorally on the chair–side, chem– ically activating the ceramic surface and roughening the surface. This process can be applied to increase bonding in acid– resistant, high–crystal content ceramics.¹⁹ TC is based on the development of micromechanical retention by using Al2O3 particles modified with silica and coating the porcelain surfaces with a thin and glassy silica layer. During the silane with the TC, a covalent bond is formed between the methacrylate group of the resin cement and the silica particle and it is stated in the literature that the bonding between zirconia and resin cement is strengthened related to this bond.^{18,20,21}

The study aimed to evaluate the effect of different surface conditioning processes for zirconia on its bond strength to selfadhesive resin cement and multi-system dual-cure adhesive resin cement and to compare the shear bond strengths of these different resin cements. The first null hypothesis was that TC would further increase the bonding strength of zirconia to resin cement rather than SB. The second null hypothesis was that the multi-system dual-cure adhesive cement would exhibit better SBS than self-adhesive cement.

Materials and Methods

In the study, two different surface treatments; SB and TC and two different resin cements; self-adhesive resin cement and multi-system dual curing adhesive resin cement were applied to the zirconia (Upcera Co. Ltd., Liaoning, China) surface. The characteristics of applied resin cements are showed in Table 1 and surface treatment methods and applied luting cements for each group are shown in Figure 1. Ethical approval was obtained from the Clinical Research Ethics Committee of the Faculty of Medicine of Tokat Gaziosmanpasa University (20– KAEK-065).

Preparation of samples

Zirconia samples were divided into three main groups with 20 samples in each group (n=20) according to the surface treatments applied as control (C) (no-treatment), SB, and TC. Each of these three main groups was divided into two subgroups according to two different resin cements that were applied. In this way, 6 groups were formed in total. Power analysis (G* power 3.1.9.4) was performed first to determine the number of samples to be used in the study. The number of samples for 6 groups was determined as 60 samples in total, 10 in each group with 80% power, 5% margin of error, and 0.5 effect size.²²

In the study, 60 molar teeth extracted for reasons such as periodontal or orthodontic were used. The teeth, which were cleaned with a curette and made sure that no additions were left on them, were stored in 0.01% thymol solution at room temperature. Autopolymerizing acrylic (Ortho-Jet Resin Acrylic; LangDental Manufacturing Co, Illinois, USA) was filled into 3 cm high and 2 cm diameter polyvinyl chloride cylinders. Before the acrylic resin polymerized, the teeth were fixed in the block so that it was 1 mm higher than the cementoenamel junction. A large water-cooled low-speed diamond cutting saw (Metkon Microcut 201, Htp High Tech Products, Istanbul, Turkey) was used to expose the superficial coronal dentin surface of the fixed teeth. The teeth were cut 3 mm below the occlusal surface. The occlusal surface exposed with the help of a diskshaped medium-grit rotary tool (Model 902; Brasseler USA) was smoothed. A total of 60 zirconia (Upcera Co. Ltd., Liaoning, China) disc-shaped samples were designed 25% larger in a CAD/CAM device (DEMASTER, Htp High Tech Products, Istanbul, Turkey) and sintering shrinkage was compensated. The final size of the discs was designed to have a height of 3 mm and a diameter of 7 mm. Ceramics were sintered in a sintering furnace (Protherm; B&D Dental Origin Milling, USA) for 8 h at 1500°C after milling. The sintered samples were measured with the help of digital calipers (Mitutoyo, Tokyo, Japan). Samples were polished for 60 s with the help of 600 and 1200 grid silicon carbide sandpaper. Samples were cleaned with distilled water and then cleaned with isopropyl alcohol for 3 min.

Surface treatment of zirconia blocks

The disk surfaces of the samples in the Group SB were treated by using 50 µm Al2O3 particles under 4 atm pressure at a distance of 10 mm to the sample at an angle of 90 degrees for 10 s. An appliance was prepared to apply a standard procedure to all samples. By fixing the tip of the sample and SB device on this apparatus, it was ensured that the SB process was performed at the same distance and the same angle for each sample. The samples in the TC group were abraded with an airborne-particle abrasion device (Cojet System; 3M ESPE, Seefeld, Germany) filled with alumina particles coated with 50micron particle size silica (Cojetsand; 3M ESPE, Seefeld, Germany). Abrasive particles were applied to the samples at a right angle from the application tip from a distance of 10 mm with 4 atm air pressure for 10 s. SB apparatus was used to apply the SB process to each sample at a fixed distance and angle. After



Figure 1. Surface treatment methods and applied luting cements for each group (n=10)

the application, the samples were washed with distilled water and then dried.

Adhesive cementation

Two different resin cements, Maxcem Elite (ME) self-adhesive resin cement (Kerr, Bioggio, Switzerland) and Nexus third generation universal dental cement (NX3) (Kerr, Bioggio, Switzerland) were applied to the subgroups (Table 1). ME resin cement was applied as a thin layer to the adherent surface of the zirconia disc with the auto-mix syringe following the manufacturer's recommendations and pressed onto the dried dentin with light finger pressure. The ceramic-resin luting agent-dentin combination was placed under a load of 750 g in a press (Articolo 719/00, Carlo de Giorgi, Milano, Italy).²³ After self-cure set time for 2-3 min and residual cements were cleaned with an applicator. Then, the samples were light polymerized for 10 s with the help of visible blue light LED device (Woodpecker LED, Guilin Woodpecker Medical Instrument Co Ltd., Guilin National High Tech Zone Information Industry Park, CHINA) at a wavelength of 460- 490 nm. Before the application of NX3 cement, first, 37.5% phosphoric acid (Kerr Gel Etchant, Orange, CA, USA) was applied for 15 s to the dentin surfaces. The dentin surfaces were then thoroughly rinsed and air-dried. With a light brushing motion, OptiBond Solo Plus (Kerr, Bioggio, Switzerland) was applied to the dentin surface with an applicator tip for 15 s and air-dried for 3 s. The excess adhesive was removed using an applicator and light-cured for 10 s. NX3 auto mix was squeezed onto the ceramic with a syringe and pressed on the dentine with light finger pressure. As in the other group, 750 g load was applied in the press to the cemented samples. The samples were light polymerized for 20 s with LED curing unit (Woodpecker LED, Guilin Woodpecker Medical Instrument Co. Ltd., Guilin National High Tech Zone Information Industry Park, CHINA) at a wavelength of 460-490 nm.

Shear bond strength

After cementation, all samples were kept in distilled water at 37°C for 24 h. After 24 h, samples were mounted on the SBS test device (Shimadzu AGS-X, Shimadzu Scientific Instruments, Columbia, North Carolina, USA) and tested to failure in shear with a pressure of 5 kN at a head speed of 1mm/min, with a 90-degree angle in the vertical direction. SBS values recorded were calculated as MPa.

Statistical analysis

The data were analyzed in terms of normal distribution by the Kolmogorov–Smirnov test. Mann–Whitney U test was used to compare within–group data in the mean ± standard deviation of the luting cements with the normal distribution. Kruskal Wallis Variance analysis was used to compare the groups formed according to the surface treatments of the study data. In the determination of statistically significant groups, the relationship between groups was examined with Tamhane's T2 test. In calculations, the significance level was accepted as p < 0.05. Ready–made statistical software (IBM Statistical Pack-age for the Social Sciences (SPSS) Version 20, SPSS inc., IBM Co., Somers, NY) was used in the analysis of the data.

Results

As a result of the normality test, it was observed that the data were distributed normally (p=0.893). Regardless of the cement type, SBS values of the surface treated samples were statistically different (p<0.001) (Table 2). Group SB was determined as the group with the highest SBS value (191.85±13.86). This group was followed by Group C (124.99±26.92) and Group TC (76.22±34.27), respectively.

The SBS values of the resin cements after zirconia surface treatments were shown in Table 3. The effect of surface treatments on the adhesion of NX3 and ME to zirconia was found to be statistically significant (p<0.001). The highest SBS was determined in SBNX3 group. SBNE, CNX3, TCNX3, CME, and TCME groups followed this respectively. Compared to Group C, the SB process increased the SBS value of both resin groups, while the TC process decreased the SBS values of all groups.

The effect of surface treatments on the SBS of different cements was found statistically significant except for the Group SB (p<0.001). SBS values of the samples cemented with NX3 resin were found to be higher than those treated with ME resin.

Table 2. Shear bond strength values of the surface treated sample	es.
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	Surface Treatment			n
	Group C (n=20)	Group SB (n=20)	Group TC (n=20)	– p
SBS (MPa)	124.99±26.92 ^(a)	191.85±13.86 ^(b)	76.22±34.27 ^(c)	<0.001

Abbrevations: SBS: Shear bond strength

Values with different superscripts indicate the difference between groups.

Table 3. The shear bond strength values of the resin cements after zirconia surface treatments.

Group C		Group SB		Group TC		ME	NX3
CME	CNX3	SBME	SBNX3	TCME	TCNX3		
100.7±12,08 ^(A,a)	149.2±8,8 ^(B,a)	186.08±9.1 ^(A,b)	197.6±15.7 ^(B,D)	43.9±3.9 ^(A,c)	108.4±12.3 ^(B,C)	p#	p#
p<0.001		p=0.151		p<0.001		<0.001	<0.001

Common superscript indicates the statistical insignificance. Capital letters were used for intragroup comparison. Lower case letters were used for comparison between groups.

* p value was considered significant at the 0.05 level.

Discussion

According to the study results, while the TC surface treatment for zirconia decreased the bond strength to the resins, the SB surface treatment increased the bond strength to the resins and the multi-system dual-cured adhesive cement exhibited better SBS than the self-adhesive cement. Therefore, the first hypothesis was rejected and the second hypothesis was accepted.

The clinical success and longevity of the restoration depends on a strong bond between resin cement and zirconia.²⁴ This is possible through the formation of both chemical bonds and micromechanical bonds. Over the years, various pretreatment methods have been studied on zirconia surfaces to ensure a better and longer-lasting bond between cement and ceramic. Since many factors such as the type of ceramic, the monomer content of the cement,²⁵ the particle size of the cement's filler,²⁶ and the mechanical properties of the luting cement²⁶ may interfere with bonding strength, conducting pretreatment studies is rather complicated.

Conventional Bis-GMA resin cements do not exhibit a durable, long-term bond to high-strength ceramic materials such as zirconia.^{5,27} Application of phosphoric or hydrofluoric acid applied to increase micromechanical retention in silicabased ceramics does not provide an acceptable surface roughness for high strength ceramics.²⁸ The surface roughness in high-strength ceramics can be achieved by sintering or exposing the restoration surface to abrasive particles such as sand.²⁹⁻³¹ Thus, in the current study, the SBS values of two different resin cements (self-etch adhesive resin without Bis-GMA content and multi-system dual-cure adhesive resin) were evaluated after SB and TC surface treatments. Airborne particle abrasion with Al2O3 is the preferred surface treatment method for high strength ceramic materials. 5,12,27,32-34 Al2O3 sand is used in various sizes and different methods in studies and laboratory applications. 4,29,35,36 When the bond strength between zirconia and different resin cements was evaluated, it was observed that blasting with Al2O3 of 50 and 100 μm under the same pressure did not create a significantly different bond strength.²⁶ However, Yang et al.³⁶ reported that SB with 50 µm Al2O3 is an effective method in long-term zirconia cementation. For this reason, Al2O3 with a size of 50µm was used in the current study. SB with Al2O3 significantly increased the SBS value of both resin groups, while TC treatment significantly decreased the SBS of them. However, it was determined that the effect of the SB process on the SBS value of the resins was insignificant. Oyagüe et al.³⁵ showed in their study that the SB process creates more micro retentive pits on the zirconia surface than the TC process. In the current study, the reason for the higher SBS values of the sandblasted samples compared

to the TC applied samples is thought to be due to the increased micro retention related to the filling of the micro-holes on the sandblasted surfaces with more resin particles. Similar to the various studies, in the current study, 15,30,37,38 regardless of the cement material used, it was also reported that SB with Al2O3 applied on the zirconia surface provided higher SBS than the TC. TC is used to improve the silica content on the surface of non-silica components such as zirconia and alumina-based ceramics.³⁹ The present study results showed that the TC reduced the SBS of the resins in all groups. In particular, a dramatic decrease was observed in the SBS value of the self-etch adhesive resin cement. The TC is expected to cover the zirconia surface ideally completely with silica. However, in a study, it was observed that some areas on the zirconia surface were not covered with silica.¹⁹ In the same study, it was stated that some silica particles were not in contact with the zirconia surface or were not fused with other particles and therefore the tribochemical reaction was not completed. However, various studies reported that Al residues remained on the surface despite cleaning the surface with air spray or ultrasonic cleaner after TC treatment. ^{40,41} According to the results of the current study, the surface of zirconia may not be completely covered with silica and that the residual silica particles on the zirconia surfaces may prevent the bonding of luting cement to zirconia sufficiently. Zirconia stabilized with Yttria was reported to offer more hardness than systems with a glassy structure and to prevent silica impregnation on the surface. ^{42,43} However, further studies are required to understand exactly why TC surface treatment reduces SBS of the resins.

The effect of surface treatments on the SBS of resin cements was found statistically significant except for the Group SB. However, it was observed that the groups using multi-system dual-cure adhesive resin cement displayed higher SBS values than the groups using self-etch adhesive cement. Agreed with the results of the current study, Lee et al. 44 reported in their study that NX3 cement showed greater bond strength than ME cement. Unlike NX3, ME cement requires the dentin surface to be acidified and adhesive application before it is applied. The reason for the higher SBS values of the groups using NX3 may be explained by the pickling process that created more microretention by roughening the surface and the applied adhesive further increased the bonding. ME resin, on the other hand, consists of multifunctional monomers, unlike NX3 resin. However, the multifunctional monomers in ME resin are not as effective as NX3 for micromechanical bonding and chemical interaction on the tooth surface.

Adequate bond strength is defined by values equal to or higher than 20 MPa, regardless of the test methods used.⁴⁵ In this regard, although the lowest SBS was observed in TC applied groups, all samples exhibited sufficient bonding strength. In evaluating the bond strength of in vitro studies, there is no consensus on the studies' methodology, the interpretation of the results, and their application to clinical results. Since different materials are tested in different ways in the studies, no conclusions can be drawn based on the findings of the studies. 4^{6}

According to the results of the present study, it is observed that the SB process significantly increased the SBS of the resins while TC was a less effective method for increasing the SBS of resins. There were several limitations of this study. First was the lack of a failure mode or SEM analysis, which could help to understand the effect of TC better. Other limitations were the determination of short-term SBS, using 1 type of zirconia and 2 types of cement materials, and the in vitro study design. The effect of surface treatments on zirconia's mechanical properties should also be investigated before making any clinical recommendations.

Conclusion

Tribochemical silica coating decreased the shear bond strength of the resins while the sandblasting with Al2O3 increased the shear bond strength of resins. Different surface treatments affected the shear bond strength of resin cements. Within the limits of the study, it has been determined that it is more advantageous to use multi-system dual-cure adhesive cements for zirconia restorations instead of self-etch adhesive cements.

Author Contributions

Concept: Y.H., I.S., Design: Y.H., S.K., Data Collection or Processing: Y.H., S.K., I.S., Analysis or Interpretation: Y.H., S.K., Literature Search: Y.H., S.K., I.S., Writing: Y. H.

Conflict of Interest

The authors declare that they have no conflict of interest.

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