Original Article

Shear Bond Strength of Zirconia Ceramic to the Primary Tooth Dentin

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ABSTRACT

Background: There is no information about the shear bond strengths (SBS) of zirconia ceramic to primary tooth dentin. **Aim:** To investigate the effect of different surface treatments and cements on the shear bond strength (SBS) of zirconia ceramic to primary tooth dentin. **Materials and Methods:** Prepared zirconia bars were distributed into four groups according to surface treatment procedure: control, sandblasting, CoJet and hot etching. The zirconia specimens in each group were further divided into subgroups according to cement (n = 13): self-adhesive resin (Rely-X Unicem), resin-modified glass ionomer (Ketac-Cem Plus), and universal bioactive (BioCem). Zirconia specimens were bonded to the primary tooth dentin surface by cement. SBS was measured, and the data were subjected to two-way ANOVA and Tukey's tests. **Results:** Statistical differences were observed in the surface treatment procedures for Rely-X Unicem ($P < 0.05$), but no statistically significant differences were found in the sandblasting, CoJet and hot-etching groups for Ketac-Cem Plus ($P > 0.05$). For BioCem, the SBS value for the hot etching group was significantly lower than those for the CoJet and sandblasting groups ($P < 0.05$). The SBS values for the Rely-X Unicem subgroups (sandblasting, CoJet and hot etching) were significantly higher than those for the other cements ($P < 0.05$). **Conclusion:** The bond strength of zirconia ceramic to primary tooth dentin is affected by surface treatments and cements.

KEYWORDS: *Cement, primary tooth, shear bond strength, zirconia*

INTRODUCTION

Early childhood caries (ECC), one of the most common types of dental caries, is a specific form of rampant caries observed in the primary teeth of children under the age of six.[1,2] Many problems can be observed in children with untreated or early extraction of teeth affected by ECC.[3] Therefore, restoring the primary teeth and keeping them in the mouth until the physiological fall process begins are important.

By considering the time spent on the teeth in the mouth and the cooperation of children, the primary teeth influenced by ECC can be restored with composite resins, conventional glass ionomer cements, resin-modified glass ionomer cements, and polyacid-modified composite resins.[4] Stainless steel crowns, polycarbonate crowns, or strip crowns are used in the teeth, which encounter excessive material loss and cannot be restored with composite resin or glass ionomer cements.[5] However, stainless steel crowns are not aesthetic, and they can be easily broken during the shaping of prefabricated veneered stainless steel crowns. Polycarbonate crowns require more cutting, their adaptation is difficult, and their wear resistance is low. Strip crowns can rupture when they are placed, their fracture resistance is low because of their composite structure, and they exhibit marginal area coloration.[5-9] Therefore, more aesthetic and durable restorative materials are needed in pediatric dentistry to reduce the number of application steps and the treatment time.

The problems related to the aesthetics and biocompatibility of metal-supported ceramics have led to the development of full ceramic systems without

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metal. Metal-free restorations may enable soft tissues to have better natural-like protection than metal-backed restorations. These restorations also show better color stability, long-term clinical success, and lower thermal conductivity.[10] Zirconium full ceramic crowns and bridges are preferred by dentists and adult patients because of their superior aesthetic and mechanical properties.[11] In recent years, it has also been used in the restoration of primary teeth influenced by ECC in pediatric dentistry.[12-16]

Although studies considering the bond strength of zirconia ceramic to permanent teeth can be found in the literature,[17-19] to our knowledge, no information about the shear bond strength (SBS) of zirconia ceramic to primary tooth dentin is available. Thus, this study aimed to investigate the SBS of zirconia ceramic to the primary tooth dentin and the effect of different surface treatments and cements on the SBS of zirconia ceramic to the primary tooth dentin. Two null hypotheses were tested: (1) surface treatment procedures do not affect the SBS of zirconia ceramic to the primary tooth dentin and (2) no significant differences are found in the SBS of the used cements.

Materials and Methods

This study was approved by the Non-Interventional Clinical Research Ethics Council (approval date: 06/06/2016; approval number: 04). Before the tooth extraction, the patients/parents were informed about the use of their teeth for research purposes, and their consent was obtained.

Materials

Three different commercial cements, namely, self-adhesive resin cement (Rely-X Unicem), resin-modified glass ionomer cement (Ketac-Cem Plus), and universal bioactive cement (BioCem), were used in this study. All of the materials were applied according to the manufacturers' instructions. Details on the cements are presented in Table 1.

Preparation of dentin specimens

This study was conducted with 156 freshly extracted, caries-free, human primary molars that fell because of physiological resorption. The teeth were stored in 0.1% thymol solution prior to the experiment. Before the experimental procedures, the teeth were examined under a stereomicroscope (Nikon Eclipse E 600, Nikon Corp., Tokyo, Japan) at ×30 magnification, and those with cracks or stains were excluded.

The included teeth roots were removed. The occlusal surfaces of each tooth were ground with 320-grit silicon carbide paper (Leco, St. Joseph, Michigan, USA) to expose the dentin. The dentin specimens were embedded in acrylic resin with the dentin bonding site face-up in the mold. The outer surfaces of the dentin surfaces were ground under running water using a polishing machine (MetaServ, 250 Twin, Buehler, Germany) with 320-, 400- and 600-grit silicon carbide paper to create standardized flat dentin surfaces [Figure 1].

Preparation of zirconia specimens

A total of 156 zirconia bars (3 mm × 1 mm × 1 mm) were milled from yttrium-stabilized tetragonal zirconia (Y-TZP) blocks (Lot No. ZB6008A, ICE Zirkon Translucent, Zirkonzahn, GmbH, Bruneck, Italy). The bonding site of each bar was finished using 600-, 800-, 1000- and 1200-grit silicon carbide paper to obtain a flat surface. All the zirconia bars were then ultrasonically cleaned in deionized water for 5 min and divided into four groups according to surface treatment procedure (n = 39) as follows:

Group I (Control): No surface treatment was applied.

Group II (Sandblasting): The zirconia bars were sandblasted with 50 µm Al_2O_3 particles (Korox, Bego, Bremen, Germany) from a distance of 10 mm perpendicular to the specimen surface at 2.5 bar pressure for 15 s using a sandblasting device (Rocatec Junior, 3M ESPE, St. Paul, MN, USA).

Group III (tribochemical silica treatment, CoJet): The zirconia bars in this group were subjected to 30 µm Al_2O_3 particles coated with silica (CoJet Sand, St. Paul, MN, USA) from a distance of 10 mm perpendicular to the specimen surface at 2.8 bar pressure for 15 s using a sandblasting device (Rocatec Junior, 3M ESPE, St. Paul, MN, USA).

Group IV (Hot Etching): The zirconia bars were immersed in an experimental hot chemical etching solution composed of 800 ml methanol, 200 ml 37% HCl, and 2 g Fe_2Cl_3 at 100°C for 10 min.[20]

After the surface treatments procedures, all the zirconia bars were ultrasonically cleaned in deionized water for 5 min and gently air dried.

Cementation protocol

The zirconia specimens in each group were further divided into three subgroups according to cement (n = 13): self-adhesive resin cement (Rely X Unicem), resin-modified glass ionomer cement (Ketac-Cem Plus), and universal bioactive cement (BioCem). The zirconia specimens were bonded to the dentin specimens by cement prepared according to the manufacturer's instructions. Excess cement was removed and polymerized using a light-emitting diode curing unit (Elipar Free Light II, 3M ESPE, St. Paul, MN, USA) for 20 s on either side of the specimen. Specimens were also held in a fixed position without any movement for 5 min to achieve self-curing without motion [Figure 2].

SBS testing

After the cementation procedure, all specimens were stored at 37°C in distilled water for 24 h. Then, the specimens were subjected to SBS testing (MOD Dental MIC-101, Esetron Smart Robotechnologies, Ankara, Turkey) by applying a shear load to the base of the zirconia bars at a crosshead speed of 1 mm/min until bond failure occurred [Figure 3].

Following debonding, the specimens were examined under a stereomicroscope (Nikon Eclipse E 600, Nikon Corp., Tokyo, Japan) at ×10 magnification to determine the mode of bond failure, which was recorded as adhesive (failure at the zirconia–dentin interface), cohesive (failure exclusively within the zirconia or dentin), or mixed (a mixture of adhesive and cohesive failure).

Statistical analysis

Two-way ANOVA was used to identify the significant differences in the SBS among the groups, and multiple comparisons were made using Tukey's test. The level of significance was set to $P < 0.05$. All statistical analyses were performed using IBM SPSS Statistics, Version 22 (SPSS Inc., Chicago, Illinois, USA).

Results

The means and standard deviations of the SBS for each group are listed in Table 2.

Among the surface treatment procedures, SBS values of all cements were the lowest in the control group. No statistically significant difference was found between the control and hot-etching groups in the BioCem group.

In the BioCem group, the mean SBS values of the CoJet group demonstrated were higher than those of the sandblasting group, but the differences were not statistically significant ($P > 0.05$). The SBS values of the hot etching group were significantly lower than those of the CoJet and sandblasting groups ($P < 0.05$).

In the Rely X Unicem group, statistically significant differences were found in the mean SBS values of all the surface treatment procedures ($P < 0.05$) (CoJet >sandblasting >hot etching).

In the Ketac-Cem Plus group, no significant differences were found in the SBS values of the sandblasting, CoJet, and hot-etching groups ($P > 0.05$).

In comparing the cements, the SBS values of the all Ketac-Cem Plus subgroups were significantly lower than those of the BioCem and Rely X Unicem groups ($P < 0.05$). Except for the control subgroups, the Rely X Unicem subgroups (sandblasting, CoJet, and hot etching) were significantly higher than the BioCem subgroups ($P < 0.05$).

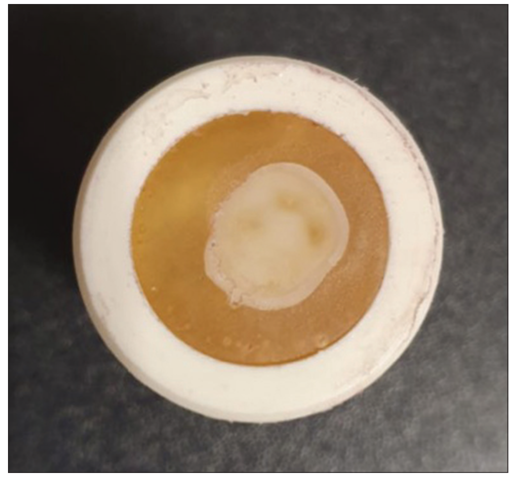

Figure 1: The view of the dentin samples embedded in acrylic resin

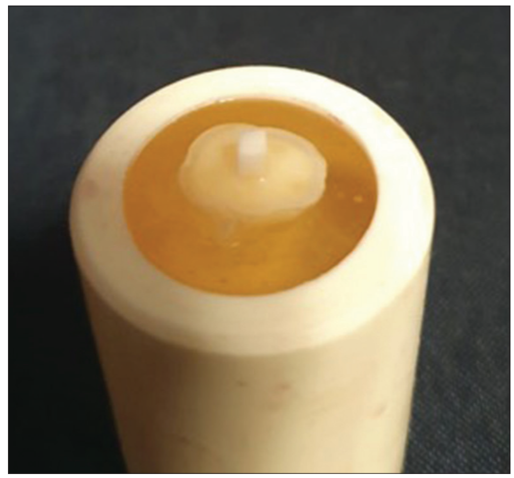

Figure 2: The view of the zirconia specimens bonded to the dentin specimens by cement

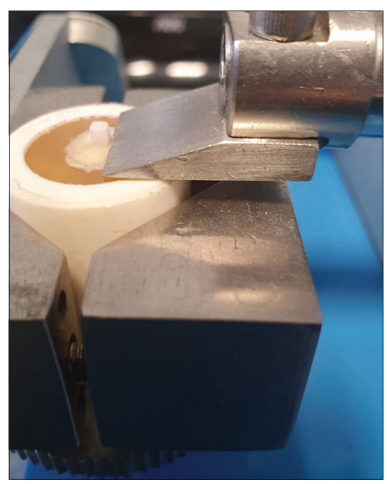

Figure 3: The view of the specimens subjected to SBS testing

Table 1: Cements used in this study

Materials	Composition	Manufacturer	Lot Number
Self-adhesive resin cement (Rely-X Unicem)	Base: Glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator	3M/ESPE, Neuss, Germany	642241
	Catalyst: Methacrylated, phosphoric esters, dimethacrylate, acetate, stabilizers, self-cure initiators, light-cure initiators		
Resin-modified glass ionomer cement (Ketac-Cem Plus)	Paste A: Fluoroaluminosilicate glass, proprietary reducing agent, HEMA, water, opacifying agent	3M/ESPE, Neuss, Germany	790597
	Paste B: Methacrylated polycarboxylic acid, BisGMA, HEMA, water, potassium persulfate, zirconia silica filler		
Universal bioactive cement (BioCem)	Blend of diurethane and other methacrylates with modified polyacrylic acid, silica, amorphous, sodium fluoride	NuSmile LTD, USA	151201

Bis-GMA=bisphenol-Aglycidylmethacrylate, HEMA=Hydroxyethyl methacrylate

Table 2: Mean±SD of SBS of different surface treated zirconia bars bonded to primary dentin using cements

Groups	Mean±SD		
	Rely X Unicem	Ketac-Cem Plus	BioCem
Control	5.50±0.74^{d,1}	1.68±0.26^{b,2}	6.04±0.63^{b,1}
Sandblasting	12.85±0.90^{b,1}	4.34±0.65^{a,3}	11.15±0.70^{a,2}
CoJet	14.54±0.92^{a,1}	4.37±0.58^{a,3}	11.60±0.84^{a,2}
Hot etching	11.07±0.92^{c,1}	4.17±0.59^{a,3}	6.22±0.30^{b,2}

*Differences in superscript letters indicate statistically significant differences within columns, and differences in superscript numbers indicate significant differences within rows (p<0.05) (1, a=best values)

All specimens showed adhesive failure at the zirconia–dentin interface.

Discussion

Zirconium, which provides a natural tooth-like image, is a biocompatible material with gingival tissues.[21] Zirconia full ceramic crowns and bridges are preferred by dentists and adult patients because of their superior aesthetic and mechanical properties.[11] However, the use of zirconium crowns in primary teeth is scarce in the literature.[12-16,22-24]

The long-term success of zirconium restorations was found to be related to the preparation technique of the inner surfaces before the cementation, the properties of the cement, the bond strength, and the durability between the zirconium ceramic and the cement.[25-27] The cementation process is one of the most important steps in ensuring the retention, sealing, and continuity of restorations.[28] In the cementation of zirconium crowns, the use of resin cements is generally preferred.[29] Zirconium surfaces need a suitable surface preparation process to form a stable and repeatable bond with cement.[30] To increase the bond strength between the zirconium and the tooth surface, laser treatment,[31] sandblasting,[25,31-35] hot chemical etching,[20,35-37] selective infiltration etching,[20,36,37] tribochemical silica coating,[25,32,33,38,39] silane application,[25] primer application,[40] nano-structured alumina coating,[40] and gas-phase fluorination[41] methods are suitable methods. In dental literature, although there are studies considering the bond strengths of zirconia ceramic to permanent teeth,[17-19] to our knowledge, no studies have been conducted on the SBS of zirconia ceramic to primary tooth dentin. Therefore, this study aimed to investigate the SBS to the primary tooth dentin of zirconia ceramic and the effect of different surface treatments and cements on the SBS of zirconia ceramic to the primary tooth dentin.

Sandblasting with Al_2O_3 particles[25,32-34] and the silica-coating process using silica modified Al_2O_3 (CoJet) particles[25,32,33,38,39] are surface treatment methods commonly used in zirconia ceramic. The abrasive particles used in these surface treatments remove the contamination layer on the ceramic surface, increase the surface area required for the bonding, and increase the bond strength of resin cement by facilitating the wetting of the zirconium surface.[4,32-34,38,39,42] In our study, in accordance with other studies,[25,32-34,38,39,42] the SBS of zirconia to the primary tooth dentin increased for all the cement groups when 50 µm Al_2O_3 and the CoJet system were used as the surface treatment process. Thus, the first null hypothesis of the study was rejected.

Although the surface treatment procedures with abrasive Al_2O_3 particles are generally used in increasing the bond strength of zirconium to resin cement,[25,32-34,38,39] the mechanical properties of zirconia ceramic can be adversely affected during abrasion with Al_2O_3 particles. Therefore, the search continues for alternative methods to increase the bond strength between cement and zirconia without damaging the zirconia surface.[43,44] The hot chemical (acid) solution etching[20,35-37] method dissolves only the surface particle structure of zirconia ceramic and creates nanometer dimensions of roughness.[20,36] This method creates less internal stress than etching with Al_2O_3 particles.[20,36] Previous studies showed that the SBS increased when hot acid solution etching was used as the surface treatment process.[20,35-37] Consistent with previous studies,[20,35-37] when the hot acid solution etching was used as the surface treatment process in all cement groups in our study, the SBS of zirconia to the primary tooth dentin increased in comparison with that of the control group.

In this study, self-adhesive resin cement (Rely X Unicem), resin-modified glass ionomer cement (Ketac-Cem Plus), and universal bioactive cement (BioCem) were used to compare the efficiency of the cements. The findings showed that self-adhesive resin cement offered highest SBS of zirconia ceramic to the primary tooth dentin among the other cements; thus, the second null hypothesis was also rejected. This result may be explained by the fact that the Rely X Unicem contains methacrylated phosphoric ester as a functional monomer. Phosphate ester monomer-containing cements have been reported to result in high, durable bond strengths because the phosphate ester group chemically bonds to metal oxides such as zirconium dioxide.[45]

According to the results of the study, the bond strength of zirconia ceramic to the primary tooth dentin is affected by the surface treatments and cements. However, to our knowledge, no information on the SBS of zirconia ceramic to the primary tooth dentin is available to date. Therefore, to confirm the data obtained from this *in vitro* study and to investigate the effects of the different surface treatments and cements, further clinical and scanning electron microscopy trials are required.

Conclusion

Based on the results of the study, the following conclusions can be drawn:

1. The different surface treatment methods tested were all found to have positive effects on the SBS of zirconia to the primary tooth dentin. CoJet was the most effective in increasing the SBS.
2. The SBS values of self-adhesive resin cement in all the subgroups were found to be higher than those of the other cements.

Disclosure and Acknowledgments

This study has been presented in International Association of Paediatric Dentistry Regional Meeting and 25th Congress of Turkish Society of Paediatric Dentistry October 12-14 2018 Istanbul, Turkey.

Declaration of patient consent

The authors certify that they have obtained all appropriate patient consent forms. In the form the patient(s) has/have given his/her/their consent for his/her/their images and other clinical information to be reported in the journal. The patients understand that their names and initials will not be published and due efforts will be made to conceal their identity, but anonymity cannot be guaranteed.

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Conflicts of interest

There are no conflicts of interest.

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