

The effect of ceramic primer on shear bond strength of resin composite cement to zirconia

A function of water storage and thermal cycling

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Different types of cement are available for use in dentistry: traditional cements (for example, glass ionomer cement, zinc oxyphosphate cement) and resin composite cements. Ceramic restorations, especially glass-ceramic-based restorations, must be cemented with a resin composite cement, because it improves the stability and clinical durability of the restoration.¹⁻⁴ On the basis of their esthetic properties, mechanical characteristics⁵ and high biocompatibility,⁶ zirconia restorations have become increasingly common in dentistry over the last few years.^{7,8} Researchers in various clinical studies reported that zirconia demonstrates great stability as a framework material.⁹⁻¹³ To date, researchers have reported few fractures of zirconia frameworks.^{14,15} Zirconia can be cemented with traditional cements or with resin composite cement. The advantage of using resin composite cement is reduced marginal microleakage.¹⁶⁻²⁰ Marginal discrepancies of dental restorations can result in a higher rate of ditching of the cement and, therefore, in secondary car-

ABSTRACT

Objectives. The authors investigated the use of ceramic primers combined with self-adhesive resin composite cements on the shear bond strength (SBS) to zirconia and compared them with one conventional resin composite cement.

Methods. The authors divided zirconia substrates (N = 550) into three groups: RelyX Unicem Aplicap self-adhesive universal resin composite cement (3M ESPE, St. Paul, Minn.) (group A) (n = 220); G-CEM Capsule self-adhesive resin composite cement (GC Europe, Leuven, Belgium) (group B) (n = 220); and Panavia 21 with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer (n = 110) (Kuraray Dental, Tokyo) used as a standard comparison (SC). The authors examined the self-adhesive resin composite cements without (0) and with (1) the use of a ceramic primer. They measured SBS initially (37°C for three hours), after water storage (37°C for one, four, nine, 16 or 25 days) and after thermal cycling (5°C and 55°C for 1,500, 6,000, 13,500, 24,000 or 37,500 cycles). The authors analyzed data by using descriptive statistics, the Mann-Whitney test, the Kruskal-Wallis test and a χ^2 test.

Results. Application of a ceramic primer did not result in a negative impact on SBS. Specimens in the A1 group (that is, RelyX Unicem Aplicap with ceramic primer) exhibited significantly higher SBS before and after water storage and thermal cycling compared with specimens that were not treated with a primer. The self-adhesive resin composite cements combined with ceramic primer exhibited similar or higher SBS values compared with those in the SC group at each aging duration (that is, water storage and thermal cycling). With respect to G-CEM Capsule, the authors observed a significantly positive effect of the primer after nine and 16 days' water storage and after one and four days' thermal cycling. They observed predominantly adhesive failures.

Conclusions. Ceramic primer in combination with self-adhesive resin composite cement demonstrated a positive effect on SBS to zirconia and should be used for cementation.

Practical Implications. Long-term adhesion to ceramic plays an important role in the longevity of dental restorations. Therefore, the authors recommend that clinicians use a ceramic primer when cementing zirconia restorations.

Key Words. Zirconia; bonding properties; zirconia primer; self-adhesive cements.

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ies or endodontic or periodontal complications.²¹ These biological complications may compromise the longevity of the tooth, as well as that of the dental restoration itself.

Resin composite cements can be divided further according to their compositions: the conventional resins based on bisphenol A–glycidyl methacrylate (bis-GMA), triethylene glycol dimethacrylate or urethane dimethacrylate or the resin composite cements containing acid groups. The latter can be divided into subgroups consisting of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and self-adhesive resin composite cements with multifunctional acidic methacrylates (for example, phosphoric acid ester, carboxylic acid or amino acid derivatives). Self-adhesive resin composite cements are simple to use and less time consuming than conventional resin composite cements, which require pretreatment of teeth with use of adhesive systems.¹⁶

The acidic methacrylates^{22–24} and the MDPs^{25–28} provide adhesion via direct interaction with the zirconia surface. The phosphate ester group of both bifunctional monomers may bond chemically to zirconia surface oxides, while the methacrylate group acts as a monomer matrix and is responsible for polymerization.²⁸

The bond strength of polished zirconia specimens pretreated with MDP ceramic primer resulted in an increase in chemical bonding.²⁹ Researchers in previous studies^{25,26} investigated the chemically cured Panavia 21 (Kuraray Dental, Tokyo) and the dual-cured Panavia F 2.0 (Kuraray Dental) resin composite cements, both of which contain MDP monomers. They observed higher and more durable bond strength values compared with those of other cements. The addition of a ceramic primer significantly improved the bond strength between zirconia and the resin composite cement.^{25,26,30} Three days of water storage at room temperature of zirconia specimens bonded with Panavia F 2.0 in combination with ceramic primer Clearfil SE Bond primer and Clearfil Porcelain Bond Activator resulted in a mean (standard deviation [SD]) shear bond strength [SBS] of 20.14 (2.59) megapascals; the mean (SD) SBS was 17.36 (3.05) MPa without application of a primer.²⁵ Blatz and colleagues²⁵ observed that after 180 days of water storage and 12,000 thermal cycles, zirconia surfaces that were pretreated with ceramic primer had a higher SBS (mean [SD], 16.85 [3.72] MPa) than did untreated surfaces (9.45 [5.06] MPa). We found no studies in the literature in which use of Clearfil SE Bond primer with Clearfil Porcelain Bond Activator in combination with Panavia 21 resulted in de-

creased SBS compared with that in specimens not treated with a primer.

Various test methods are available for laboratory analyses of bond strength.³¹ Investigators have shown that the SBS test is a reproducible method of testing resin composite cements. In a two-center study, Holderegger and colleagues³² tested the SBS of four different resin composite cements to dentin. In addition, Sailer and colleagues³¹ and Stawarczyk and colleagues³³ determined the SBS of different self-adhesive resin composite cements and reported values similar to those of Holderegger and colleagues.³² Furthermore, the SBS test is suitable for examining a large number of specimens, and it is a reliable and time-saving test. Consequently, because the SBS test methods exhibited reproducible results, we chose it for this investigation.

In this study, we aimed to evaluate the influence of ceramic primers in combination with self-adhesive resin composite cements on the SBS to zirconia after various durations of water storage and thermal cycling, as well as to compare the results with those for an MDP-based standard comparison (SC) group. Owing to the stable bond strength reported in previous studies, we chose Panavia 21 combined with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer (all Kuraray Dental) for the SC in this study. The scientific hypothesis tested was that applying a ceramic primer to zirconia specimens would result in higher SBS values than those in specimens not treated with a ceramic primer, regardless of the type of self-adhesive resin composite cement used.

METHODS

We included in this study two dual-cured, self-adhesive resin composite cements (RelyX Unicem Aplicap self-adhesive universal resin composite cement [3M ESPE, St. Paul, Minn.] and G-CEM Capsule self-adhesive resin composite cement [GC Europe, Leuven, Belgium]) and one conventional dual-cured resin composite cement (Panavia 21) with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer used as the SC group. We tested the self-adhesive cements with and without application of the ceramic

ABBREVIATION KEY. **bis-GMA:** Bisphenol A–glycidyl methacrylate. **2-HEMA:** 2-hydroxyethyl methacrylate. **MD:** Mean difference. **MDP:** 10-methacryloyloxydecyl dihydrogen phosphate. **4-META:** Methacryloxyethyl trimellitate anhydride. **SBS:** Shear bond strength. **SC:** Standard comparison. **SEM:** Scanning electron microscope, scanning electron microscopic. **UDMA:** Urethane dimethacrylate.

TABLE 1

Summary of cements and primers tested.

GROUP	PRODUCTS USED	COMPOSITION*	LOT NO.	APPLICATION	MANUFACTURER
Standard Comparison	Clearfil Porcelain Bond Activator, Clearfil SE Bond primer and Panavia 21 resin composite cement	<ul style="list-style-type: none"> • Clearfil Porcelain Bond Activator: 3-trimethoxysilylpropyl methacrylate, hydrophobic aromatic dimethacrylate, others • Clearfil SE Bond primer: 2-HEMA,[†] MDP,[‡] hydrophilic aliphatic dimethacrylate, dl-camphorquinone, water accelerators, dyes, others • Panavia 21–catalyst paste: MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated silica filler, colloidal silica, catalysts, others • Panavia 21–universal paste: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated titanium dioxide, silanated barium glass filler, catalysts, accelerators, pigments, others 	00208B/00769A and 00406C UNI TC/00647C CAT	Mix one drop of Clearfil Porcelain Bond Activator with one drop of Clearfil SE Bond primer for five seconds; apply the mixture to zirconia surface; dry surface sufficiently by blowing soft, oil-free air; apply equal amounts of Panavia 21 catalyst and universal pastes mixed by a dispenser	Kuraray Dental, Tokyo (www.kuraraydental.com)
A0	RelyX Unicem Aplicap self-adhesive universal resin composite cement	<ul style="list-style-type: none"> • Powder: silanized glass powder, silane-treated silica, substituted pyrimidine, calcium hydroxide, sodium persulfate, titanium dioxide • Liquid: mixture of mono, di- and tri-glycerin-dimethacrylate-ester of phosphoric acid, triethylene glycol dimethacrylate, substituted dimethacrylate, copper acetate 	352388	Insert capsule into activator, press handle and hold for 2 to 4 seconds; mix for 10 seconds with RotoMix Capsule Mixing Unit; insert capsule into applicator	3M ESPE, St. Paul, Minn. (http://solutions.3m.com/wps/portal/3M/en_US/3M-ESPE-NA/dental-professionals/contact-us/about-3M-ESPE/)
A1	RelyX Ceramic Primer and RelyX Unicem Aplicap self-adhesive universal resin composite cement	RelyX Ceramic Primer: ethyl alcohol, water, methacryloxypropyl trimethoxysilane	7XY 352388	Apply by using microbrushes and air dry softly	
B0	G-CEM Capsule self-adhesive resin composite cement	<p>Powder: fluoro-alumino-silicate glass (amorphous), camphorquinone</p> <p>Liquid: dimethacrylate, 4-META,[§] UDMA,[¶] phosphoric ester monomer</p>	803061	Shake the capsule and push the plunger until it is flush with the body of the capsule; place capsule into an applicator and click the lever once; mix for 10 seconds; insert capsule into applicator	GC Europe, Leuven, Belgium (www.gceurope.com)
B1	GC Ceramic Primer and G-CEM Capsule self-adhesive resin composite cement	GC Ceramic Primer A: ethanol GC Ceramic Primer B: methyl methacrylate, ethanol, UDMA, 2-HEMA	803061/ 901272	Mix ceramic primers A and B, apply to zirconia surface and dry softly with air	

* Sources: Kuraray Clearfil Porcelain Bond Activator MSDS,³⁴ Kuraray Clearfil SE Bond: Primer MSDS USA,³⁵ Kuraray Panavia 21: Catalyst Paste MSDS USA,³⁶ Kuraray Panavia 21: Universal Paste MSDS USA,³⁷ 3M Material Safety Data Sheet: 3M ESPE RelyX Unicem Aplicap/Maxicap,³⁸ 3M Material Safety Data Sheet: 3M ESPE RelyX Ceramic Primer,³⁹ G-CEM Capsule-Powder: Material Safety Data Sheet,⁴⁰ G-CEM Capsule-Liquid: Material Safety Data Sheet,⁴¹ Ceramic Primer A: Material Safety Data Sheet,⁴² Ceramic Primer B: Material Safety Data Sheet.⁴³

[†] 2-HEMA: 2-hydroxyethyl methacrylate.

[‡] MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

[§] 4-META: Methacryloxyethyl trimellitate anhydride.

[¶] UDMA: Urethane dimethacrylate.

primer on the zirconia surface, according to the manufacturer's recommendations (Table 1³⁴⁻⁴³).

We used a cutting machine (Accutom, Struers, Ballerup, Denmark) to cut the zirconia substrates from square blanks (YZ-20/15, Vita In-Ceram YZ, Vita Zahnfabrik, Bad Säckingen, Germany; lot 20961) into 2-millimeter slices (N = 550). The substrates subsequently were sintered in a furnace (model LHT 02/16, Nabertherm, Lilienthal, Germany), with a heating rate of 17°C/minute and a final temperature of 1,530°C. The holding time was 120 minutes, and the cooling rate was 10°C/minute until the substrates reached room temperature. We then embedded the substrates in a self-curing acrylic resin (ScandiQuick, Scan-Dia, Hagen, Germany). After three hours, we polished the substrates in a standardized manner by using silicon carbide polishing paper with a grade of up to P2400 (800 grit) and by using water cooling (Abramin machine, Struers). We cleaned the specimens with alcohol and dried them with oil-free air.

We divided the polished specimens into three main groups: the SC group (n = 110) and two self-adhesive resin composite cement groups (n = 220 per group). We pretreated one-half of the specimens in both self-adhesive cement groups with the ceramic primer (n = 110 per group), and we cemented the other one-half of the specimens without pretreating them (n = 110 per group). We conducted the initial SBS test after storing the specimens in water for three hours at 37°C for both water storage and thermal cycling. We further divided the specimens into subgroups according to the following durations of water storage (one day, four days, nine days, 16 days and 25 days) or number of thermal cycles (1,500, 6,000, 13,500, 24,000 and 37,500 cycles). Each of the 55 subgroups contained 10 specimens.

Bonding procedure. In groups that received the ceramic primer pretreatment, we applied the agent to the surface of the specimen according to the manufacturer's instructions (Table 1³⁴⁻⁴³). Before applying the cement, we fixed an acrylic cylinder with an inner diameter of 2.9 mm (DR-TEC, Birmensdorf, Switzerland) to the zirconia surface in the holding device. The experimental setup has been described in detail elsewhere.³³ The acrylic cylinder acted as a place holder, with a defined area for the resin composite cement. We placed the cement into the aperture of the acrylic cylinder. To attain a consistent cement layer of 0.5 mm, we inserted in the center of the acrylic cylinder a steel screw with an outer diameter of 2.8 mm parallel to

the axis of the acrylic cylinder. After positioning the screw, we loaded it with a weight of 100 grams (Figure 1A). We removed excess cement thoroughly and polymerized the specimens by using a light-curing unit (Elipar S10 LED Curing Light, 3M ESPE).

Aging regimens. We stored the specimens in distilled water for three hours, one day, four days, nine days, 16 days and 25 days at 37°C in an incubator (UMS, Memmert, Schwabach, Germany). Before performing thermal cycling, we stored all specimens for 24 hours in distilled water at 37°C in the incubator. We performed thermal cycling for 1,500, 6,000, 13,500, 24,000 and 37,500 cycles in a thermal cycling machine (Thermocycler, Willytec, Gräfelfing, Germany [distributed by SD Mechatronik, Feldkirchen-Westerham]) at temperatures of 5°C and 55°C and at a dwell time of 20 seconds in each bath. We converted the thermal cycles into days (1,500 cycles equates to 24 hours and 11 minutes).

SBS measurement. We measured SBS by using a universal testing machine (Z010, Zwick/Roell, Ulm, Germany), with a crosshead speed of 1 mm/minute. We positioned the specimens vertically and parallel to the loading piston in a special device, as described elsewhere.³³ We applied the load to the outer surface of the cylinder at a distance of 300 micrometers from the specimen surface (Figure 1B). We recorded values at the debonding time of the acrylic cylinders. We expressed the SBS values by using the following formula: force/area (that is, newtons/square millimeter).

Fracture mode. To determine the fracture mode, we examined the specimen under a binocular microscope (Wild M3B stereomicroscope, Heerbrugg, Switzerland) with a magnification of ×25, as well as under a scanning electron microscope (SEM) (Tescan Vega TS 5136 XM, Elektronen-Optik-Service, Dortmund, Germany). We recorded two types of fractures: adhesive (no cement remnants on the specimen surface) and cohesive (specimen surface covered with cement).

Statistical analysis. We performed analyses by using descriptive statistics for SBS data (SPSS 15, IBM SPSS, Armonk, N.Y.). We tested the approximate normality of data distribution by using the Kolmogorov-Smirnov and Shapiro-Wilk tests. Using Mann-Whitney and Kruskal-Wallis tests, we compared all groups that were nonparametric with respect to the use of a ceramic primer, resin composite cement and aging level. To evaluate the effect of the ceramic primer combined with self-adhesive resin composite

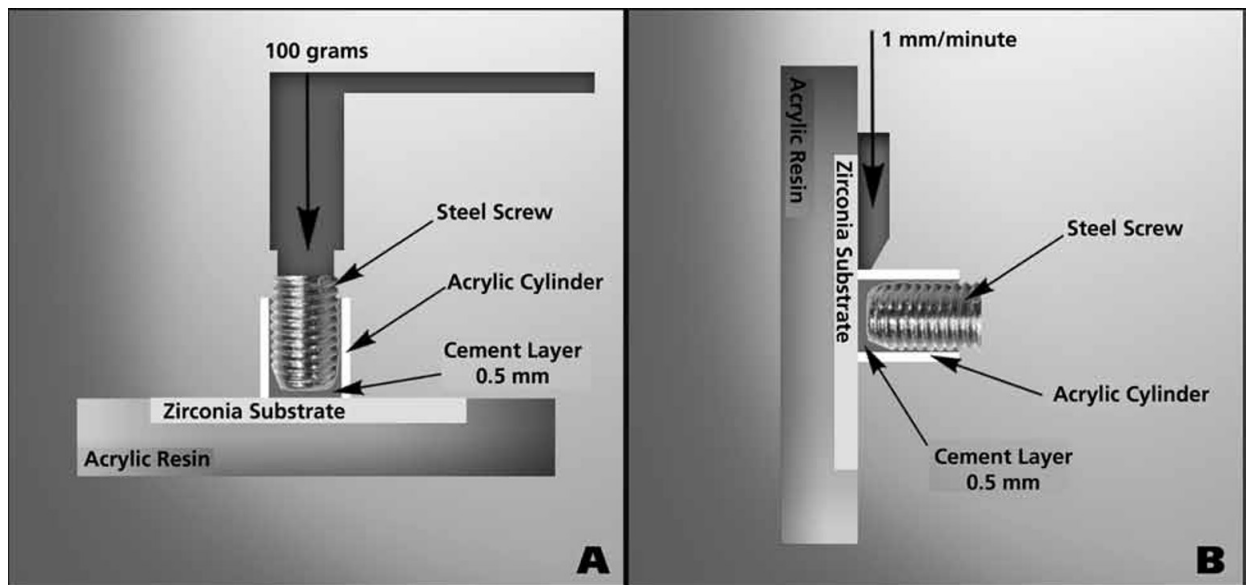


Figure 1. Shear bond strength test design. **A.** Specimen preparation with acrylic cylinder and cement layer at the bottom of the cylinder. The steel screw is located in the center, with the loading device on top of the screw. **B.** Experimental assay for the shear bond strength test. mm: Millimeter.

cement, we calculated mean differences (A1–A0 or B1–B0) between groups with and without the ceramic primer, as well as 95 percent confidence intervals (CIs) and *P* values.

We used the χ^2 test to analyze differences between the tested groups with respect to failure types. We gathered the 95 percent CI for the true relative frequencies of the failure types from the Ciba Geigy table.⁴⁴ We considered all results of the statistical analyses with *P* values smaller than .05 to be statistically significant.

RESULTS

We referred to specimens that were fractured during the aging process as 0 MPa. We used all specimens for statistical analysis. The results of Kolmogorov-Smirnov and Shapiro-Wilk tests indicated that the SBS groups were not normally distributed. Hence, we performed nonparametric statistical analyses. Table 2 presents the descriptive statistics, such as mean and SD, for water storage results and measurements after thermal cycling. Figure 2 (page 1267) shows the mean (SD) bond strength (in megapascals) after water storage, and Figure 3 (page 1267) shows the results after thermal cycling.

Water storage. We found no negative effects of water storage on any tested resin composite cements. In contrast, specimens in the SC group and in groups B0 and B1 showed an increase in SBS according to the aging duration (Table 2; $P < .001$).

Thermal cycling. Specimens in groups A0 and A1 (RelyX Unicem Aplicap self-adhesive

universal resin composite cement, without and with primer, respectively) exhibited a significant decrease in SBS ($P = .013$) after 25 days of thermal cycling. However, we found no influence of thermal cycling on specimens in the SC group (Table 2; $P > .05$).

Impact of aging regimens. Regardless of the type of resin composite cement used or whether a ceramic primer was used, our study findings showed that one day of water storage or thermal cycling had no impact on SBS ($P = .087$ to $.963$) (Table 3, page 1268).

Specimens in the following groups exhibited a significantly lower SBS after thermal cycling compared with specimens aged in water: after four days, both self-adhesive resin composite cements without ceramic primer, as well as RelyX Unicem Aplicap with ceramic primer ($P < .001$ to $P = .025$); after nine days, the SC group (Panavia 21) and the G-CEM Capsule group, both with ceramic primer ($P < .001$ and $P = .001$, respectively); after 16 days, all cements with ceramic primer and G-CEM Capsule without ceramic primer ($P < .001$ to $P = .011$); after 25 days, all groups ($P < .001$ to $P = .002$).

Impact of ceramic primer. In general, the use of a ceramic primer combined with both tested self-adhesive resin composite cements had no negative effect on the SBS of zirconia specimens (Table 4, page 1268). After water storage and thermal cycling, the SBS in the RelyX Unicem groups increased after pretreatment with RelyX Ceramic Primer ($P < .001$ to $P = .020$). Within the G-CEM groups, the study

TABLE 2

Shear bond strength test results after water storage and thermal cycling.

GROUP	NO. OF DAYS OF WATER STORAGE OR THERMAL CYCLING	SHEAR BOND STRENGTH, IN MEGAPASCALS*					
		Water Storage			Thermal Cycling		
		Mean (SD) [†]	Median (IQR) [‡]	Minimum/maximum	Mean (SD)	Median (IQR)	Minimum/maximum
SC [§]	0	6.1 (1.9) ^a	5.6 (3.5)	3.2/8.7	6.1 (1.9) ^a	5.6 (3.5)	3.2/8.7
	1	6.0 (2.3) ^a	6.8 (4.7)	2.4/9.1	5.4 (2.7) ^a	4.9 (4.2)	2.1/10.1
	4	7.7 (2.0) ^{ab}	7.4 (3.1)	4.7/11.1	6.6 (1.0) ^a	6.7 (1.2)	4.4/7.9
	9	8.1 (0.9) ^{ab}	8.0 (1.4)	6.9/10.0	4.8 (1.6) ^a	5.3 (3.3)	2.1/6.6
	16	9.4 (2.2) ^b	9.6 (3.4)	5.6/12.6	4.9 (2.3) ^a	5.2 (2.9)	0/8.4
25	8.2 (1.7) ^{ab}	8.2 (2.7)	5.3/10.2	4.6 (2.6) ^a	3.8 (5.3)	1.5/7.9	
A0 [¶]	0	7.5 (1.3) ^a	7.9 (2.1)	5.7/9.9	7.5 (1.3) ^b	7.9 (2.1)	5.7/9.9
	1	8.6 (2.4) ^a	8.6 (5.1)	5.1/11.5	6.2 (3.6) ^b	7.3 (3.8)	0/11.0
	4	10.1 (2.2) ^a	9.9 (4.4)	7.3/12.7	4.6 (2.1) ^b	4.6 (2.8)	0/7.1
	9	8.1 (2.5) ^a	8.7 (3.7)	4.1/11.9	6.6 (3.3) ^b	6.6 (4.0)	0/11.2
	16	9.1 (3.4) ^a	10.1 (5.3)	1.4/12.4	6.3 (3.8) ^b	6.7 (5.4)	0/11.1
	25	8.4 (3.6) ^a	9.2 (4.3)	0/12.9	2.7 (2.9) ^a	2.7 (4.7)	0/7.4
A1 [#]	0	11.4 (1.3) ^a	11.0 (2.4)	9.7/13.5	11.4 (1.3) ^b	11.0 (2.4)	9.7/13.5
	1	11.4 (2.4) ^a	10.8 (4.0)	7.6/15.1	11.4 (1.1) ^b	10.9 (1.9)	10.3/13.6
	4	13.8 (3.7) ^a	12.5 (7.8)	9.3/18.9	8.5 (1.0) ^b	8.9 (1.8)	6.9/9.8
	9	13.6 (1.8) ^a	14.0 (2.2)	9.6/15.7	13.2 (2.7) ^c	12.2 (5.6)	10.2/17.1
	16	13.4 (2.9) ^a	13.2 (3.8)	8.9/18.8	9.8 (2.0) ^{ab}	10.2 (3.2)	7.1/12.8
	25	13.3 (1.7) ^a	13.2 (2.0)	11.7/15.0	8.2 (1.6) ^a	8.7 (2.7)	5.3/10.3
B0 ^{**}	0	6.2 (1.7) ^a	6.5 (2.3)	3.6/9.4	6.2 (1.7) ^a	6.5 (2.3)	3.6/9.4
	1	8.5 (1.3) ^b	8.1 (1.7)	6.3/10.7	7.0 (2.2) ^a	7.5 (3.6)	4.4/10.4
	4	8.8 (1.8) ^b	8.6 (3.0)	6.8/12.7	7.3 (0.7) ^a	7.3 (1.4)	6.0/8.2
	9	8.8 (1.4) ^b	8.8 (2.8)	6.9/10.9	10.1 (3.3) ^b	10.0 (5.1)	5.7/15.3
	16	8.2 (2.0) ^b	8.4 (2.2)	4.1/11.4	5.4 (2.5) ^a	4.8 (3.5)	2.7/9.8
	25	9.7 (1.8) ^b	10.1 (2.9)	6.0/12.0	4.2 (4.5) ^a	3.2 (8.6)	0/10.1
B1 ^{††}	0	7.1 (1.3) ^a	6.9 (2.2)	5.2/9.3	7.1 (1.3) ^{ab}	6.9 (2.2)	5.2/9.3
	1	8.6 (1.4) ^{ab}	9.4 (2.1)	5.9/9.7	9.8 (3.0) ^{bc}	9.5 (5.3)	5.3/14.5
	4	10.0 (1.8) ^{bc}	9.7 (2.4)	7.2/13.2	10.6 (2.3) ^c	11.5 (3.6)	6.1/13.0
	9	12.8 (1.7) ^{cd}	12.9 (3.2)	10.1/15.3	8.5 (2.7) ^{abc}	9.0 (4.4)	3.5/12.0
	16	11.7 (1.3) ^{cd}	12.2 (1.7)	8.8/12.9	7.1 (2.7) ^{ab}	7.1 (3.3)	2.7/12.0
	25	11.7 (3.3) ^{cd}	11.2 (5.6)	0.9/17.8	6.4 (3.1) ^a	5.9 (4.3)	2.5/11.5

* Different superscript letters indicate statistically significant differences between aging levels within a group.

† SD: Standard deviation.

‡ IQR: Interquartile range.

§ SC: Standard comparison, consisting of Clearfil Porcelain Bond Activator, Clearfil SE Bond primer and Panavia 21 resin composite cement (manufactured by Kuraray Dental, Tokyo).

¶ A0: RelyX Unicem Aplicap self-adhesive universal resin composite cement (manufactured by 3M ESPE, St. Paul, Minn.).

A1: RelyX Ceramic Primer (3M ESPE) and RelyX Unicem Aplicap self-adhesive universal resin composite cement.

** B0: G-CEM Capsule self-adhesive resin composite cement (manufactured by GC Europe, Leuven, Belgium).

†† B1: GC Ceramic Primer (GC Europe) and G-CEM Capsule self-adhesive resin composite cement.

results showed a significant positive impact of GC Ceramic Primer for specimens that underwent nine and 16 days of water storage ($P < .001$), as well as for those that underwent one ($P = .032$) and four ($P < .001$) days of thermal cycling.

Failure types. Although most of the groups exhibited adhesive failures, we observed cohesive failures in resin composite cement in the following groups: Panavia 21 (SC) after 16 days of thermal cycling, RelyX Unicem Aplicap self-adhesive universal resin composite cement without ceramic

primer (A0) at all thermal cycling levels and G-CEM Capsule without ceramic primer (B0) after 25 days of water storage at 37°C.

Figure 4 (page 1269) shows an SEM image of a cohesive fracture in resin composite cement after debonding.

DISCUSSION

The effective adhesion of cements to zirconia is a major prerequisite for the longevity of dental restorations. In this study, we evaluated the effect of applying ceramic primer on the SBS of

different self-adhesive resin composite cements to zirconia. The findings showed that pretreatment of the zirconia surface with a ceramic primer resulted in significantly higher SBS values for RelyX Unicem Aplicap self-adhesive universal resin composite cement. For G-CEM Capsule, only similar or slightly higher SBS values were attained after pretreatment of zirconia specimens with ceramic primer. Therefore, we accepted the tested hypothesis—that the application of ceramic primer would result in higher SBS—only for RelyX Unicem Aplicap self-adhesive universal resin composite cement.

Investigators in previous studies²²⁻²⁸ characterized the MDP monomer as an important molecule for achieving successful bond strength between zirconia and resin composite cement. However, opinions differ with regard to the bond between conventional resin cements and zirconia. Although resin composite cements with bifunctional monomers, such as MDP, or multifunctional phosphor methacrylates are designed to result directly in chemical interaction with the zirconia surface,²²⁻²⁸ some authors^{45,46} have stated that resin composite cements that contain bis-GMA work mainly through micromechanical anchorage. Because we could find no published studies in which investigators reported a negative impact of Clearfil SE Bond primer and Clearfil Porcelain Bond Activator in combination with Panavia 21 on the bond strength, and owing to

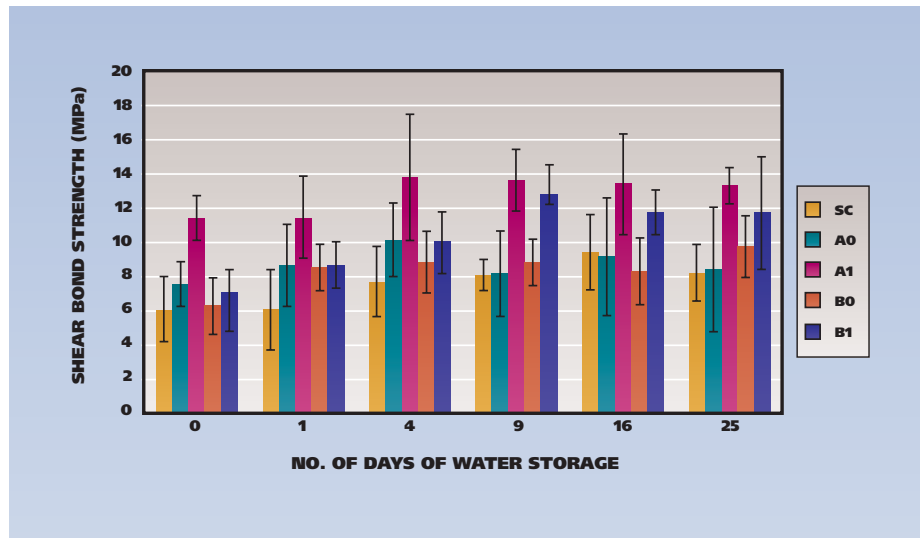


Figure 2. Mean (standard deviation) shear bond strength of specimens after water storage. SC: Standard comparison (Panavia 21 with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer, Kuraray Dental, Tokyo). A0: RelyX Unicem Aplicap self-adhesive universal resin composite cement (3M ESPE, St. Paul, Minn.). A1: RelyX Unicem Aplicap self-adhesive universal resin composite cement with RelyX Ceramic Primer (3M ESPE). B0: G-CEM Capsule self-adhesive resin composite cement (GC Europe, Leuven, Belgium). B1: G-CEM Capsule self-adhesive resin composite cement with GC Ceramic Primer (GC Europe). MPa: Megapascals.

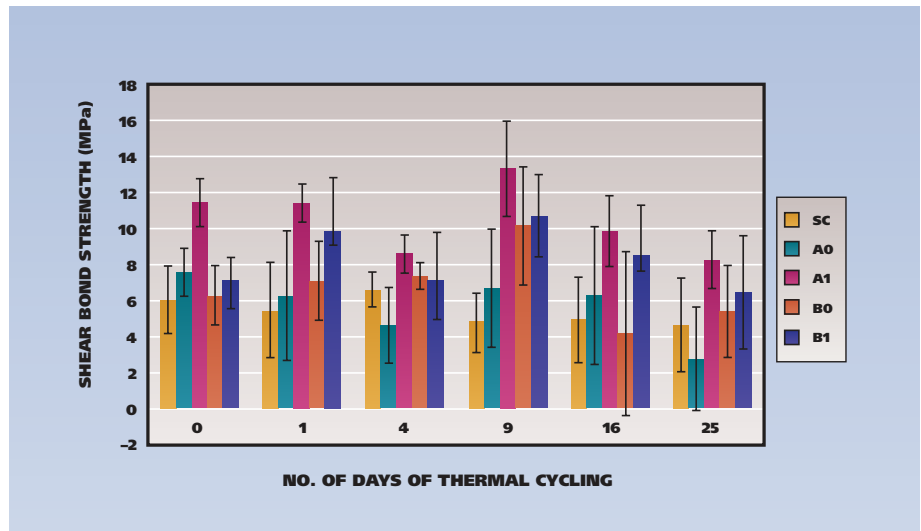


Figure 3. Mean (standard deviation) shear bond strength of specimens after thermal cycling. SC: Standard comparison (Panavia 21 with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer, Kuraray Dental, Tokyo). A0: RelyX Unicem Aplicap self-adhesive universal resin composite cement (3M ESPE, St. Paul, Minn.). A1: RelyX Unicem Aplicap self-adhesive universal resin composite cement with RelyX Ceramic Primer (3M ESPE). B0: G-CEM Capsule self-adhesive resin composite cement (GC Europe, Leuven, Belgium). B1: G-CEM Capsule self-adhesive resin composite cement with GC Ceramic Primer (GC Europe). MPa: Megapascals.

the stable results reported in several previous studies,^{25,26} we used Panavia 21 combined with Clearfil SE Bond primer and Clearfil Porcelain Bond Activator as a standard group for comparison in this study. Both the primer and cement in this combination of materials contain MDP monomers.

In this study, we investigated two different

TABLE 3

Comparison between water storage and thermal cycling.

GROUP	NO. OF DAYS OF WATER STORAGE OR THERMAL CYCLING									
	One		Four		Nine		16		25	
	P	MD* (95% CI) [†] , in MPa [‡]	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa
SC [§]	.576	0.6 (-1.7 to 3.0)	.146	1.1 (-0.4 to 2.6)	< .001	3.3 (2.0 to 4.6)	< .001	4.5 (2.3 to 6.6)	.002	3.6 (1.4 to 5.6)
A0 [¶]	.101	2.4 (-0.5 to 5.2)	< .001	5.5 (3.5 to 7.5)	.275	1.5 (-1.2 to 4.2)	.092	2.9 (-0.5 to 6.3)	.001	5.7 (2.5 to 8.7)
A1 [#]	.963	0.04 (-1.7 to 1.8)	< .001	5.2 (2.6 to 7.8)	.738	0.3 (-1.7 to 2.5)	.006	3.6 (1.2 to 5.9)	< .001	5.1 (3.7 to 6.3)
B0 ^{**}	.087	1.5 (-0.2 to 3.2)	.025	1.5 (0.1 to 2.8)	.253	-1.3 (-3.6 to 1.0)	.011	2.9 (0.7 to 5.0)	.002	5.5 (2.1 to 8.9)
B1 ^{††}	.269	-1.2 (-3.3 to 1.0)	.500	-0.6 (-2.5 to 1.3)	.001	4.3 (2.1 to 6.5)	< .001	4.7 (2.6 to 6.7)	.002	5.2 (2.2 to 8.3)

* MD: Mean difference (water storage value minus thermal cycling value). A positive number indicates water storage results higher than thermal cycling results. A negative number indicates thermal cycling results higher than water storage results.
[†] CI: Confidence interval.
[‡] MPa: Megapascals.
[§] SC: Standard comparison, consisting of Clearfil Porcelain Bond Activator, Clearfil SE Bond primer and Panavia 21 resin composite cement (manufactured by Kuraray Dental, Tokyo).
[¶] A0: RelyX Unicem Aplicap self-adhesive universal resin composite cement (manufactured by 3M ESPE, St. Paul, Minn.).
[#] A1: RelyX Ceramic Primer (3M ESPE) and RelyX Unicem Aplicap self-adhesive universal resin composite cement.
^{**} B0: G-CEM Capsule self-adhesive resin composite cement (manufactured by GC Europe, Leuven, Belgium).
^{††} B1: GC Ceramic Primer (GC Europe) and G-CEM Capsule self-adhesive resin composite cement.

TABLE 4

Impact of ceramic primer in combination with self-adhesive resin composite cement.

VARIABLE	NO. OF DAYS OF WATER STORAGE OR THERMAL CYCLING											
	0		One		Four		Nine		16		25	
	P	MD* (95% CI) [†] , in MPa [‡]	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa	P	MD (95% CI), in MPa
Water Storage												
A0 [§] versus A1 [¶]	< .001	3.8 (2.6 to 5.1)	.018	2.8 (0.5 to 5.1)	.017	3.7 (0.7 to 6.6)	< .001	5.5 (3.4 to 7.5)	.008	4.2 (1.2 to 7.2)	.020	4.9 (2.2 to 7.6)
B0 [#] versus B1 ^{**}	.228	0.8 (-0.5 to 2.2)	.876	0.1 (-1.1 to 1.4)	.152	1.2 (-0.4 to 2.9)	< .001	4.0 (2.5 to 5.4)	< .001	3.5 (1.8 to 5.0)	.117	2.0 (-0.5 to 4.5)
Thermal Cycling												
A0 versus A1	< .001	3.8 (2.6 to 5.1)	.001	5.1 (2.4 to 7.7)	< .001	3.9 (2.3 to 5.5)	< .001	6.6 (3.7 to 9.4)	.020	3.5 (0.6 to 6.4)	< .001	5.5 (3.2 to 7.7)
B0 versus B1	.228	0.8 (-0.5 to 2.2)	.032	2.7 (0.2 to 5.2)	< .001	3.3 (1.6 to 5.0)	.239	-0.2 (-4.4 to 1.2)	.164	1.7 (-0.7 to 4.1)	.210	2.3 (-1.4 to 5.9)

* MD: Mean difference (value for specimens with primer minus value for specimens with no primer). A positive number indicates higher results for specimens with primer than for specimens with no primer. A negative number indicates higher results for specimens with no primer than for specimens with primer.
[†] CI: Confidence interval.
[‡] MPa: Megapascals.
[§] A0: RelyX Unicem Aplicap self-adhesive universal resin composite cement (manufactured by 3M ESPE, St. Paul, Minn.).
[¶] A1: RelyX Ceramic Primer (3M ESPE) and RelyX Unicem Aplicap self-adhesive universal resin composite cement.
[#] B0: G-CEM Capsule self-adhesive resin composite cement (manufactured by GC Europe, Leuven, Belgium).
^{**} B1: GC Ceramic Primer (GC Europe) and G-CEM Capsule self-adhesive resin composite cement.

self-adhesive resin composite cements used in combination with a primer. Neither of these ceramic primer and cement systems contains

the functional MDP monomer. The dual-curing RelyX Unicem is a phosphorylated methacrylate-based liquid-powder system

containing phosphoric acid groups. Several authors²²⁻²⁴ have reported that as a result of hydrogen bonding of these phosphoric acid groups, chemical bonding to the zirconia surface occurred. RelyX Ceramic Primer is an alcohol solution composed of a stabilized, active silane coupling agent. According to the manufacturer's instructions for RelyX Unicem Aplicap, the operator can use the resin composite cement with or without coating the substrate with silicate.⁴⁷ In contrast, G-CEM Capsule contains 4-methacryloxyethyl trimellitate anhydride, which may allow an adhesive bond to metal oxides.⁴⁸ The adhesive GC Ceramic Primer is composed of methyl methacrylates and 2-hydroxyethyl methacrylate (2-HEMA) in an alcohol solution. Because both cements can be used without pretreatment of zirconia restorations, our objective was to investigate the impact of pretreatment with a ceramic primer.

The results of this study show that higher SBS values were achieved through the application of a ceramic primer on the zirconia surface. VITA In-Ceram YZ zirconia substrate contains less than 1 weight percent of silicon dioxide, which may explain why we observed a small but statistically significant increase in SBS for specimens treated with RelyX Ceramic Primer in combination with RelyX Unicem Aplicap for all aging durations. For G-CEM Capsule self-adhesive resin composite cement, we measured a statistically significant positive effect of the GC Ceramic Primer only for specimens tested after nine days and 16 days of water storage, as well as after one day and four days of thermal cycling. The study results showed no negative impact of G-CEM Capsule self-adhesive resin composite cement combined with GC Ceramic Primer except after nine days of thermal cycling.

Variations in SBS values (including high SDs) may be caused by the water absorption of 2-HEMA in the GC Ceramic Primer. However, we did not observe any negative impact on SBS after use of primers in combination with both of the tested self-adhesive resin composite cements.

Magne and colleagues⁴⁶ investigated the effect of an experimental primer containing organophosphate monomer and carboxylic acid monomers on zirconia-to-resin SBS. They reported mean (SD) SBS values of 21.89 (6.11) MPa and 26.68 (6.20) MPa for BisCem self-adhesive cement (BISCO, Schaumburg, Ill.) and Duo-Link universal cement (BISCO), respectively. In contrast, for specimens that were not treated with the experimental primer, the mean

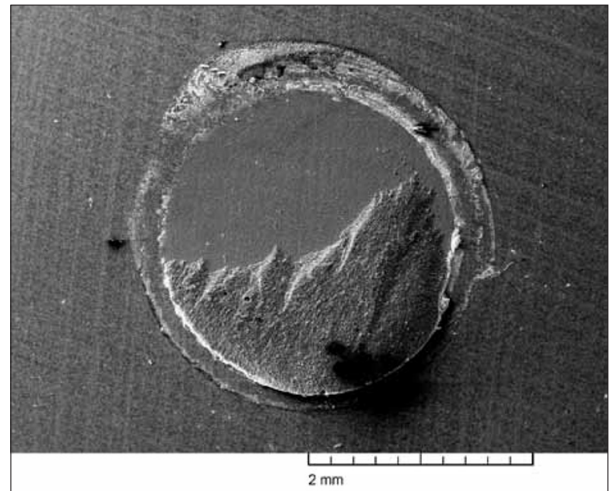


Figure 4. Scanning electron microscopic image of a cohesive fracture in resin composite cement after debonding (magnification $\times 120$). mm: Millimeters.

(SD) SBS values were 9.79 (2.18) MPa and 5.95 (1.21) MPa for BisCem and Duo-Link cements, respectively. In their study of zirconia ceramics treated with a zirconia primer, Chen and colleagues⁴⁹ reported that the application of zirconia primers resulted in a statistically significant improvement in SBS (unprimed zirconia, 4 MPa; primed zirconia, 17-23 MPa).

For in vitro studies, thermal cycling at different temperatures or long-term water storage under steady temperatures are the techniques typically used to evaluate SBS. Temperature changes play an important role in the bond strength of dental restorations. Intraoral thermal variations in vivo are caused by the alternating daily occurrences of eating, drinking and breathing.⁵⁰⁻⁵² To simulate intraoral conditions in terms of temperature changes, we used thermal cycling of 1,500 to 37,500 cycles at 5°C and 55°C, with a dwell time of 20 seconds in each bath. Investigators in previous studies observed different effects of thermal cycling. On the one hand, postpolymerization reaction of the luting area may result in higher bond strength,⁵³ whereas volumetric changes may lead to cracks and result in lower bond strength.⁵⁴ Nevertheless, the process of conducting thermal cycling in the laboratory is only a simulation of the steadily changing oral environment, without clear relation to clinical studies.

Gale and Darvell⁵⁵ conducted an overview of many studies in which investigators used a wide range of temperature changes during the thermal cycling of extracted teeth with restorations. The authors stated that 55°C is the maximum comfortable tooth temperature. For all reviewed studies, 5°C and 55°C were the median temper-

atures (range, 0°C-36°C [low] and 40°C-100°C [high]). Investigators in the reviewed studies reported a wide range of thermal cycles and dwell times (median of 500 cycles and 30 seconds' dwell time).

In addition, the conversion of 1,500 thermal cycles to one day should be seen as a limiting factor, because this number of thermal changes does not occur in one's daily routine. For our study, the thermal cycling machine performed 1,500 cycles in 24 hours and 11 minutes. There is no consensus among studies⁵⁵ with respect to the use of artificial aging procedures, as both water storage and thermal cycling were used in laboratory tests.

In this study, water storage resulted in no negative effect on any tested resin composite cement. For Panavia 21 with Clearfil Porcelain Bond Activator and Clearfil SE Bond primer and for G-CEM Capsule (with and without GC Ceramic Primer), we observed an increase in SBS according to the duration of water storage. In contrast to the Panavia 21 and G-CEM groups (with and without GC Ceramic Primer), we observed a significant decrease in SBS in the RelyX Unicem Aplicap self-adhesive universal resin composite cement groups (with and without primer) after 25 days of thermal cycling. Both aging methods, when considered individually, resulted in a different impact on SBS.

When directly comparing thermal cycling with water storage, we observed a noticeable influence of temperature changes. After 25 days of thermal cycling, specimens in all groups exhibited significantly lower SBS values than those in specimens in the groups aged in water. As water storage seemed to have no significant deteriorating impact on the SBS values, researchers in future studies should perform thermal cycling to simulate intraoral temperature ranges.

Kelly and colleagues⁵⁶ reported an incorrect loading during the SBS tests, which caused cohesive failures, and they also tested the flexural strength of the substrate or luting material. However, in our study, we observed predominantly adhesive failures. Hence, the observations of Kelly and colleagues⁵⁶ cannot be confirmed.

Investigators in future studies also should assess the effect of other ceramic primers on zirconia bonding properties. The results of our study require clinical verification.

CONCLUSIONS

Within the limitations of this *in vitro* study, we conclude that the use of a ceramic primer had a positive impact on the bond-strength

properties of zirconia to RelyX Unicem Aplicap self-adhesive universal resin composite cement. For zirconia specimens bonded with G-CEM Capsule self-adhesive resin composite cement, we observed a positive effect of the ceramic primer only for single stages of water storage and thermal cycling. Because we observed no negative impact of the ceramic primers overall, we recommend that clinicians apply a ceramic primer before cementing zirconia restorations. In addition, researchers in future studies of bond strength should use thermal cycling for specimen aging. ■

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