Abstract: The bond strength of universal adhesives to air-abraded zirconia ceramic was evaluated. Overall, 40 zirconia ceramic blocks with dimensions of 6 × 6 × 4 mm were cut from pre-sintered blanks. The sintered blocks were embedded in self-cured acrylic resin. The zirconia blocks were then randomly allocated to four groups (n = 10) in which different universal adhesives were used, except for the control group in which no universal adhesive was used. A silicon mold was used to build the resin cement. All specimens were stored in distilled water for 24 h at 37°C and mounted on a universal testing machine. They were then subjected to shear bond strength testing at a cross-speed of 0.5 mm/min until failure occurred. The failure modes were analyzed using a digital microscope at 50× magnification. Univariate one-way analysis of variance and Tukey’s post-hoc test were used for statistical analysis. Compared with the control group, the groups with universal adhesives showed statistically significant differences (P < 0.05). In addition, there was no statistically significant difference in the bond strengths of the groups with universal adhesives (P > 0.05). After 24 h of storage, the cementation bond to air-abraded zirconia ceramic was improved by the application of a universal adhesive.

Keywords: zirconia; universal adhesives; ceramics; resin cements.

Introduction

All-ceramic materials are used for partial restorations, crowns, fixed partial dentures, and many other clinical applications (1). Full ceramic restorations are categorized into glass-based ceramics and polycrystalline ceramics, which contain no glass (2). Yttria-stabilized tetragonal zirconium dioxide polycrystal (or zirconia) is one example of a polycrystalline ceramic (2). Zirconia restorations have become increasingly common in dentistry over the past few years because of their favorable esthetic, mechanical, and biocompatible properties (3,4).

The cementation protocol for an all-ceramic restoration is essential to its success (5). The adhesive cementation procedure for glass-based ceramics is well defined: it involves hydrofluoric acid etching and silanization of the restoration for better chemical bonding (6). Although conventional and adhesive cements were previously thought to be effective for zirconia ceramics (7-9), more recent studies (10,11) suggested that zirconia restorations should be adhesively, rather than conventionally,
cemented. The proper clinical protocol for using adhesive cementation with zirconium crowns and bridges remains a controversy (12), possibly owing to the chemical inertia of this material, which may negatively affect the establishment of effective chemical bonding with resin cements (13).

Different methods have been suggested to enhance resin cements’ bond strength to zirconia (14), such as airborne particle abrasion with alumina to facilitate resin-ceramic bonding by micromechanical means (15-17), physicochemical activation of the ceramic surfaces using silica-coated alumina particles followed by silanization (18-20), and chemical activation with functional monomer-containing adhesive promoters or resin cements. Other methods include selective infiltration etching (21), Er:YAG-laser irradiation (22), CO₂ laser treatment (23), and fluorination techniques (24).

Primers play an important role in adhesive procedures for all-ceramic restorations (12). However, the availability of different primers makes it difficult for clinicians to select the appropriate system for specific clinical situations (12). In fact, the ideal method to enhance bonding to ceramics should be simple and inexpensive (13). Following a simplified strategy, new universal adhesives have been developed for either direct or indirect use with multiple restorative materials such as composite, glass ceramics, and zirconia. One of these universal adhesives has shown promising results regarding the enhancement of bonding to zirconia, even in the absence of air abrasion pretreatment (12,25). In this study, three universal adhesives were tested.

The purpose of this study was to evaluate the effects of universal adhesives on the 24-h bond strength of resin cement to zirconia that had been subjected to air abrasion. The first null hypothesis was that universal adhesives have no effect on bonding to zirconia. The second null hypothesis was that there is no difference between universal adhesives.

### Materials and Methods

#### Specimen preparation

Overall, 40 zirconia blocks (Lava Zirconia, 3M ESPE, St. Paul, MN, USA) with dimensions of 6 × 6 × 4 mm were cut from pre-sintered blanks. All specimens were ground for 2 min using #600-800 silicon carbide papers (CrbMet Abrasive Disks, Buehler, Lake Bluff, IL, USA) on a 300 rev/min grinding machine (Automata, Jean Wirtz, Dusseldorf, Germany) under running water. The blocks were then sintered in a sintering oven (Lava Furnace 200, 3M ESPE) according to the manufacturer’s instructions. The dimensions of the sintered blocks decreased to (4.7 ± 0.2) × (4.7 ± 0.2) × (3.1 ± 0.2) mm.

The sintered blocks were embedded in self-cured acrylic resin (Dentsply Degudent GmbH, Hanau, Germany) and ground for 2 min, following the same polishing protocol. The specimens were then cleaned in isopropyl alcohol for 5 min using an ultrasonicator (Sonicer, Yoshida Dental Mfg. Co., Ltd., Tokyo, Japan). A power calculation was performed using means and standard deviations from similar previous studies, and the sample size identified at 80% power was less than 10. However, a sample size of 10 for each group was identified to be adequate. The blocks were randomly divided into four groups (n = 10 each); three experimental groups and one control group (Table 1), to which different universal adhesives were applied for bonding. Air abrasion was performed using Al₂O₃ particles (50 μm; 0.2 MPa; 20 s at 10 mm; 45° angle) in a chairside air abrasion device (BEGO Bremer Goldschlägerei Wilh. Herbst GmbH & Co. KG, Bremen, Germany) attached to a metal holder. The specimens were ultrasonically cleaned again in isopropyl alcohol for 10 min. They were then washed using distilled water and air dried for 5 min.

#### Bonding procedures

A piece of double-sided adhesive tape with a 4-mm-diameter hole was firmly attached to each zirconia block to define its adhesive area. A universal adhesive was applied to the entire ceramic surface using a micro-brush and air dried according to the manufacturer’s instructions (Table 2). Different type of adhesives namely, Scotchbond Universal Adhesive (SBU), Tetric N-Bond Universal (TNU), and Clearfil Universal Bond (CUB) was used for each experimental group of specimen blocks, and no adhesive was applied to the control group. The universal adhesives were not light cured, and each glossy, uniform,
and immobile adhesive film was visually inspected to ensure even distribution and adequate solvent evaporation. Resin cement (RelyX Ultimate Clicker, 3M ESPE, Neuss, Germany) was incrementally built on the ceramic surface using a silicone mold (diameter: 3 mm; height: 3 mm). Each resin cement layer was light cured for 20 s (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at an intensity of 1,000 mW/cm². After the mold was removed, additional light curing was performed for 20 s on different (four) sides of the resin cement buildup. The light-curing tip was kept as close as possible to the specimen surface.

Shear bond strength testing
All specimens were stored in distilled water for 24 h at 37°C. They were then mounted onto a universal testing machine (Instron 5965, Instron Corporation, Norwood, MN, USA) and subjected to a shear force using a notched chisel at a cross-speed of 0.5 mm/min until failure occurred.

Failure mode assessment
After debonding, the failure modes were assessed using a digital microscope at 50× magnification (Hirox Co., Ltd., Tokyo, Japan).

Statistical analysis
Univariate one-way analysis of variance and Tukey’s post-hoc tests (SPSS, Version 22.0, IBM, Armonk, NY, USA; α = 0.05) were used to analyze the significance of the shear bond strength (MPa). All data were subjected to Levene’s test of homogeneity of variance (α = 0.05), following the assumption of equal variance.

Results
The mean values and standard deviations of the shear bond strength (SBS) are shown in Table 3. The SBS values ranged from 10.74 MPa for the control group to 20.21 MPa for the TNU group. Compared with the control group, the three universal adhesive groups showed statistically significant differences (P < 0.05). Moreover, there was no statistically significant difference between the bond strength of the three universal adhesives to the...
zirconia ceramic blocks ($P > 0.05$).

The failure modes and their frequency are shown in Fig. 1. They were classified as follows: adhesive failure, cohesive failure in the resin cement, cohesive failure in the ceramic, and admixed failure (adhesive failure together with cohesive failure in the cement; Fig. 2). No pre-test failures were noted. Admix interface failures were the most common type of failures observed in the study, with a frequency of 100% for the SBU, TNU, and CUB groups. In the control group, 80% of the specimens showed adhesive failures, whereas 20% of the specimens showed cohesive failure.

**Discussion**

Effective adhesion of resin cements to zirconia is essential for the longevity of zirconia restorations (26). There are two aspects involved in the adhesion of zirconia: adhesion between the restoration surface and resin cement and adhesion of the resin cement to the tooth surface (14). Clinically, the weakest interface is observed between zirconia and the resin cement (27). This study was designed to evaluate the effect of the three different universal adhesives on 24-h bond strength between zirconia and the resin cement. The conclusions of this study have to be extrapolated with caution, because future studies are still needed regarding long-term bond strength of these materials, such as artificial aging.

Based on the size of the bonded area, the bond strength tests may be divided into “macro” methods, in which the bonded area is wider than 3 mm$^2$, and “micro” methods, in which the bonded area is below 2 mm$^2$ (28,29). Generally, the use of macro test methods has been somewhat replaced by the micro test methods, particularly the micro tensile bond strength test (30). However, SBS test remains the most commonly used macro bonding test method because of its unrivaled ease and speed of use (29). SBS test assembly has great influence on the stress distribution and is an important source of variability in shear bond measurements (31). A notched chisel was used instead of a knife-edge chisel in this study, because it is believed to impart more uniform stress distribution at the edge of the bonding area (32).

The zirconia surfaces were subjected to air abrasion prior to the study. Air abrasion of zirconia can be performed using Al$_2$O$_3$ or silica-coated Al$_2$O$_3$ particles (33,34). Al$_2$O$_3$ was used to exclude possible effects of silicatization of zirconia. The abrasive particle size and air pressure were selected based on the recommendations of the manufacturer and suggestion by Kern (11). Air abrasion increases surface roughness, improves micro-mechanical interlocking (35), and modifies the surface energy and wettability of the ceramic (25). However, it is thought to leave loose residues on the ceramic surface, and ultrasonic treatment is reported to be helpful in cleaning the surface prior to bonding (36,37). In our study, all specimens were subjected to the same surface treatment, with one resin cement used in all groups; therefore, the only variable was the universal adhesive used. The limitations of the present study included the relatively short period (24 h) of water storage. In addition, *in vitro* testing of a dry, clean field may not appropriately simulate the actual conditions in an oral cavity.

The current study results suggested that the bond strength of the resin cement to air-abraded zirconia is significantly affected by the use of a universal adhesive. Therefore, the first null hypothesis was rejected. Our results were in accordance with those of recent studies (12,25) that have reported a positive effect of the applica-
tion of universal adhesives on bonding to zirconia.

The definitive clinically durable bond strength between a resin cement and zirconia remains unknown (25). However, 20 MPa has been widely considered clinically sufficient for durable resin-zirconia adhesion (38). The mean bond strength values were 9 MPa for the control group and 17.8-20.21 MPa for the groups in which a universal adhesive was applied prior to resin cement application.

The increase in bond strength may be attributed to the effect of the functional monomer, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), in all the adhesives. MDP was first added to resin cements (e.g., Panavia F, Kuraray, Japan) for improved adhesion when bonding to polycrystalline ceramics (20,39). MDP bonds directly to the zirconia surface (40), and the formation of a covalent bond between oxygen, phosphorus, and zirconia (P-O-Zr) was confirmed in a recent study (41). Organophosphate monomers contain polymerizable functional groups that can polymerize with the matrix of methacrylate-based dental resin cements (42). In addition, because resin cement has a higher viscosity than adhesives in general, the adhesives could have penetrated more into irregularities created by air abrasion.

Without a universal adhesive, air abrasion alone was not enough to bring about satisfactory bonding between the resin cement and zirconia. The lack of chemical bonding between the methacrylate monomer of the resin cement and zirconia may explain the lower bond strength of the control group, whereas the role of air abrasion should not be underestimated, because without micro-mechanical modification, MDP may not provide durable adhesion to zirconia (43).

There was no statistically significant difference in the bond strengths of the specimens employing the three universal adhesives. Therefore, the second null hypothesis was valid. The similar bonding performance of the three adhesives may have resulted from their similar composition.

The lack of cohesive failure in the ceramic in all the specimen groups may be explained by its good mechanical properties (3). In contrast, cohesive failure in resin cement may have been caused by errors made during buildup of the resin cement. The high percentage of adhesive failure in the control group may have been caused by the lower bond strength values. Conversely, the higher bond strength values obtained for the test groups employing universal adhesives and the inhomogeneous stress distribution during shear testing (32) may be plausible explanations for the admixed failure in the specimens. Further long-term studies are recommended to evaluate the durability of this bonding technique after aging.

In conclusion, the application of universal adhesives can enhance the strength of the 24-h post-cementation bond to air-abraded zirconia ceramic.

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Conflict of interest

None declared.

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