Bonding performance of two newly developed self-adhering materials between zirconium and dentin

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Abstract

Purpose: This study evaluated the effect of four resin materials on the shear bond strength (SBS) of a ceramic core material to dentin.

Materials and Methods: Sixty molar teeth were embedded in a self-curing acrylic resin. All specimens were randomly divided into four groups of teeth, each according to the resin cement used. Sixty cylinders were then luted with one of the four resin materials to dentin (GC EQUIA, Panavia F, Variolink II and Vertise). Then, specimens were stored in distilled water at 37°C for one day. Shear bond strength of each specimen was measured using a universal testing machine at a crosshead speed of 0.5 mm/minute. The bond strength values were calculated in N, and the results were statistically analyzed using a Kruskal–Wallis and Bonferroni corrected Mann–Whitney U tests.

Results: The shear bond strength varied significantly depending on the resin materials used ($P < 0.05$). The specimens luted with GC EQUIA showed the highest shear bond strength (25.19 ± 6.12), whereas, the specimens luted with Vertise flow (8.1 ± 2.75) and Panavia F (11.17 ± 3.89) showed the lowest.

Conclusion: GC EQUIA material showed a higher shear bond strength value than other resin materials.

Key words: Shear bond strength, self-adhering, zirconia

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Introduction

Interest in zirconia has been increasing over the years in all fields of dentistry. On account of its optical properties, biocompatibility, and mechanical properties, zirconia has been chosen as a metal-free alternative to conventional dental materials.[1-3]

The long-term success of zirconia ceramic restorations depends on the cementation procedure.[4] Zirconium has no conventional silica and glass phase; therefore, acid etching and silanation are not effective in cementation procedures.[5] This issue is the major limiting factor in the use of zirconia in dental restorations and has been discussed in the literature.[6-8] Different types of cements can be used for the cementation of zirconia restorations among which adhesive resins are the most preferred because they increase fracture resistance and have better marginal adaptation and retention.[9,10] In adhesive systems, cements infiltrate the dentin tubules and form a hybrid layer between the dentin and resin cement.[11] Due to this bonding, the adhesive systems are called active materials. Conversely, in conventional cements, a mechanical interlock occurs between the dentin and restoration and these materials are referred to as passive.[12]

Nowadays, all resin cements are based on the use of self-etching or an etch-and-rinse adhesive together with a low-viscosity resin composite. This multistep application is complex and precise and contains many critical and

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time-consuming steps that could impair the effectiveness of the adhesion.

There are two types of tooth surface treatments. Etching can be applied using either a total-etch or a self-etch dentin adhesive depending on the clinician's preference and to increase bonding effectiveness.[13,14]

In recent years, self-adhering flowable composites and glass-ionomer cements have been released in the market. Generally, crown or restoration pretreatments are necessary for cementation, but these materials have the advantage of direct application to the tooth surface without requiring any pretreatment. However, there is little information about the performance of self-adhering composite and glass-ionomer cement in the bonding of zirconium restorations without surface pretreatment. Therefore, the aim of the present study was to compare the shear bond strength (SBS) of self-adhesive and conventional adhesive cements to dentin. The proposed hypothesis is that self-adhering resin has higher bond strength than adhesive systems.

Materials and Methods

The study was performed using 60 extracted (for periodontal reasons), non-carious, permanent human molars that had not been previously endodontically treated or fractured. After extraction, the teeth were immediately cleaned and stored in distilled water at room temperature for no longer than four weeks according to the International Organization for Standardization (ISO).[15] The occlusal thirds of the crowns were sectioned with a water-cooled, slow-speed, diamond saw-sectioning machine (IsoMet; Buehler, Lake Bluff, IL). The teeth were fixed in an autopolymerizing acrylic resin (Meliodent; Bayer Dental Ltd., Newbury, UK) with the ground surface upward and parallel to the support. Dentin surfaces were polished with 600 and 800Grit Silicon Carbide abrasive paper under water cooling for 30 seconds to standardize the smear layer. The specimens were then divided randomly into four groups of 15 teeth each according to the resin cements used (Vertise Flow, Panavia F, Variolink II, and GC EQUIA). Resin cement materials used in present study are shown in Table 1.

Sixty cylindrical-shaped, 2.5-mm-wide, 3-mm-high wax patterns were prepared, spruced, and invested (Zirkonzahn, Bruneck, Italy). The core cylinders were divested and all surfaces were carefully airborne-particle abraded (Miniblaster; Belle de St. Claire, Encino, CA) with 50-μm particles at a pressure of 80 psi. The tip of the micro etcher was kept 1 mm away from the surface of the specimens and was applied for three seconds. Before cementation, excess water was removed with a gentle puff of compressed air after which the core cylinders were luted to the dentin with one of the four resin cements.[16]

In the Vertise Flow resin group, the dentin surfaces were cleaned with water and dried with air and the ceramic specimens were cleaned in an ultrasonic cleaner (BioSonic JR; Whaledent Int., NY). A 0.5-mm-thick layer of Vertise Flow self-adhering cement (Kerr, Orange, CA) was applied to the dentin surface and rubbed for 15- 20 seconds with the proprietary Microbrush. Then, a small amount of resin composite was placed onto the zirconium specimen base applied to the dentin surfaces. The ceramic core cylinders were seated on the dentin surface with light finger pressure and excess cement was removed with an explorer.[17] Photo-polymerization was performed with a light polymerizing unit (Elipar S10; 3M ESPE, Seefeld, Germany) at 550 mW/cm^2 (at a light tip-to-specimen distance of 0 mm, 90° apart) for 20 seconds.

In the Panavia F group, the ceramic core cylinders were etched with 40% phosphoric acid gel (K Etchant; Kuraray Co., Ltd., Osaka, Japan) for five seconds. A layer of silane–coupling agent combination (Clearfil Porcelain Bond Activator and Clearfil SE; Kuraray Co., Ltd., Osaka, Japan) was applied to the ceramic bonding surfaces for five seconds and then air dried. Panavia F ED, the self-etching primer, was applied to the dentin surface for 60 seconds and gently air- dried. Panavia F was mixed for 20 seconds and applied to the dentin surface and the bonding surface of the ceramic core disk. The cementation procedure and photo-polymerization were performed as previously described.

In the Variolink II group, the ceramic core cylinders were treated with 37% fluoric acid (Ceramic Etchant; Ceramco, Burlington, NJ) for one minute and neutralized (Ceramic Etchant Neutralizer; Ceramco) in accordance with the manufacturer's instructions. Silane (Monobond-S; Ivoclar Vivadent, Schaan, Liechtenstein) was applied with a brush to the ceramic core disks for 60 seconds, after which a bonding agent (Heliobond; Ivoclar) was applied. After the dentin was etched, a primer (Syntac Primer; Ivoclar) was applied to the dentin surface for 15 seconds, an adhesive (Syntac Adhesive; Ivoclar) was applied for 10 seconds, and then the bonding agent (Heliobond) was applied with a brush. The cement (Variolink II, Vivadent, and Ivoclar), consisting of a combination of 25% Variolink II yellow base, 25% Variolink II white base, and 50% catalyst was hand-mixed following the manufacturer's directions and applied to both the dentin surface and the ceramic core cylinder. The cementation procedure and photo-polymerization were performed as previously described.

In the GC EQUIA group, the dentin surfaces were cleaned with water and dried with air, and the ceramic specimens were cleaned in the ultrasonic cleaner. The GC EQUIA self-adhering glass-ionomer cement was placed in a mixer and mixed for 10 seconds after which it was applied to both the dentin surface and the ceramic core cylinder. The cementation procedure and photo-polymerization were performed as previously described.

The specimens were placed on a universal testing machine (Shimadzu AG-X, Tokyo, Japan), and the load was applied at a crosshead speed of 0.5 mm/minute according to the American Society for Testing and Materials Standard Test Method E8M – 00.[18] Load at failure was recorded. One sample per group was randomly selected for assessment under a scanning electron microscope (SEM) (Noran Instruments JSM 6400, Middleton, WI).[19]

Fracture analysis

After the specimens were tested and removed from the testing apparatus, the fracture sites were observed using a stereomicroscope (LG-P52; Olympus, Tokyo, Japan) at 22°ø magnification to identify the mode of failure. Fractured surfaces were classified according to the following types: (1) adhesive failure at the interface between the ceramic and resin luting agent or between the resin luting agent and the composite resin interface; (2) cohesive failure within the ceramic, within the resin luting agent, or within the composite resin only; and (3) adhesive and cohesive failure at the same site, or a mixed failure.[20]

Statistical analysis

The data were entered into a spreadsheet (Excel version 4.0; Microsoft, Seattle, WA) for calculation of the descriptive statistics. The results of Levene's test ($P < 0.05$) and the Shapiro-Wilk test ($P < 0.05$) in all of the groups demonstrated that there was no variance homogeneity. Therefore, the bond strength data were statistically compared with the Kruskal-Wallis test, complemented by Bonferroni's correction and the Mann-Whitney U test. A chi- square test was used to compare the incidence of the different failure modes among the resin materials. The data were analyzed using SPSS 20 for Mac statistical program software. The level of significance was 5% ($P < 0.05$).

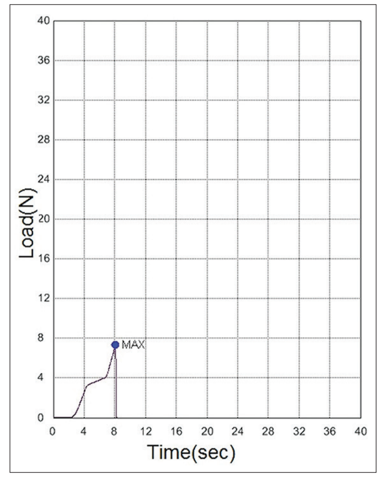

Figure 1: Load versus time curves obtained for vertise flow group

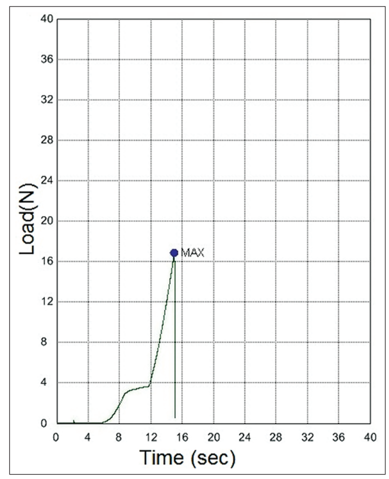

Figure 2: Load versus time curves obtained for Variolink group

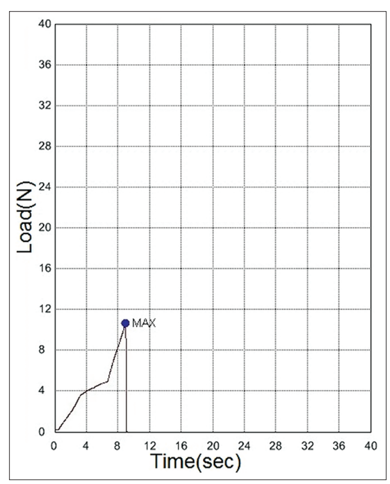

Figure 3: Load versus time curves obtained for panavia Fgroup

Results

The load versus time curves obtained from the tests is reported in Figures 1 to 4 for all the groups. The Kruskal–Wallis test indicated that the bond strengths were significantly influenced by the resin cement ($P < 0.05$). The shear bond strength values and the results of multiple comparisons of all four resin cements are summarized in Table 2. GC EQUIA exhibited the highest bond strength values (25.19 ± 6.12). Representative scanning electron microscope (SEM) photographs of the fracture interfaces after tensile testing are shown in Figures 5-8.

Fracture analysis

The specimen failure modes were evaluated and are shown in Figure 9. As expected, cohesive failure and mix failure were seen in all specimens in the Panavia F, Vertise Flow, and Variolink II bond groups. In the GC EQUIA group, cohesive failure was observed more than the other failure modes. Statistical analysis (Chi-square test) showed no statistically significant differences in failure modes among the groups ($P > 0.05$).

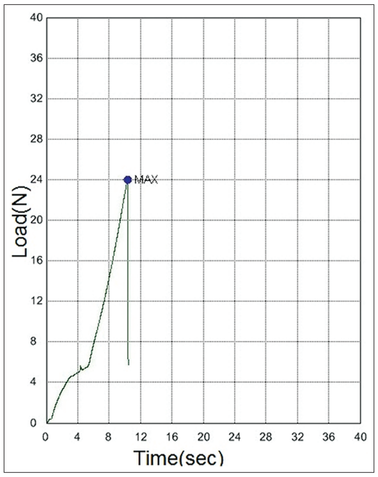

Figure 4: Load versus time curves obtained for GC EQUIA group

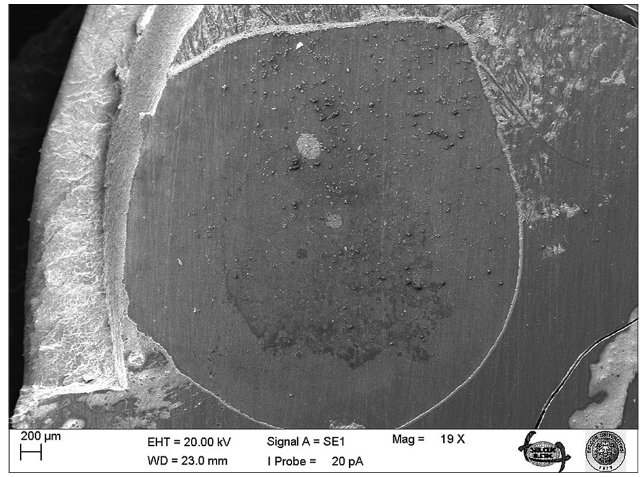

Figure 5: SEM photograph of a sample from the Vertise flow group. The failure mode was completely adhesive

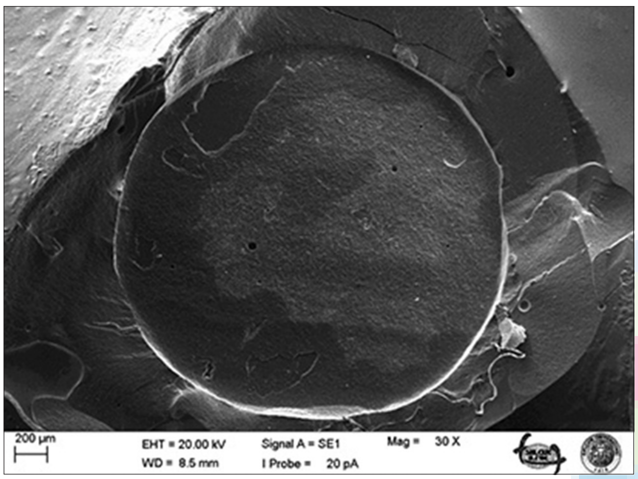

Figure 6: SEM photograph of a sample from the Variolink group. The failure mode was completely cohesive

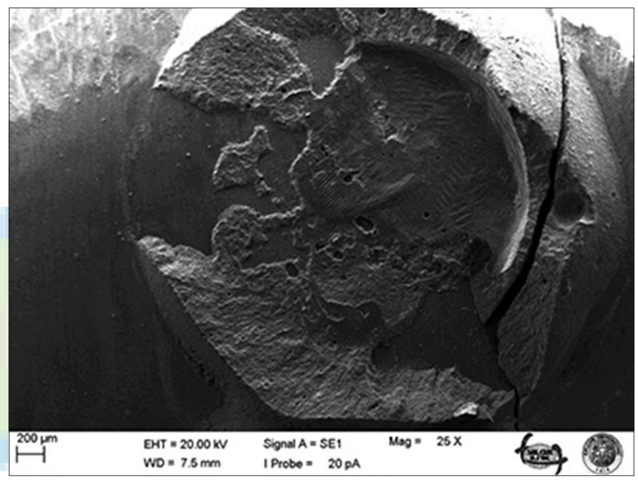

Figure 7: SEM photograph of a sample from the Panavia Fgroup. The failure mode was mixed

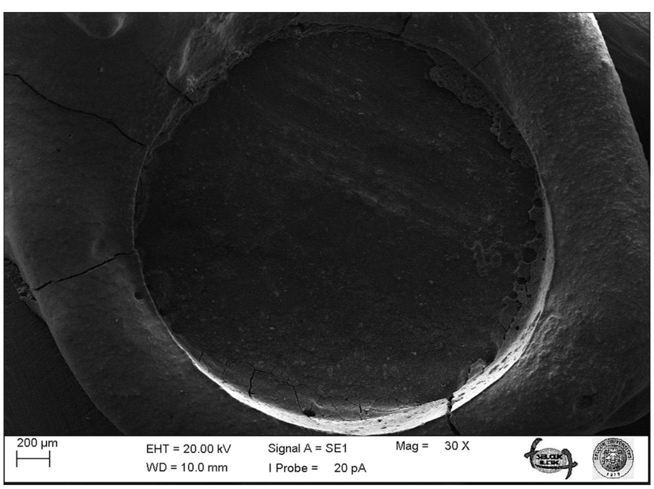

Figure 8: SEM photograph of a sample from the GC EQUIA group. The failure mode was cohesive

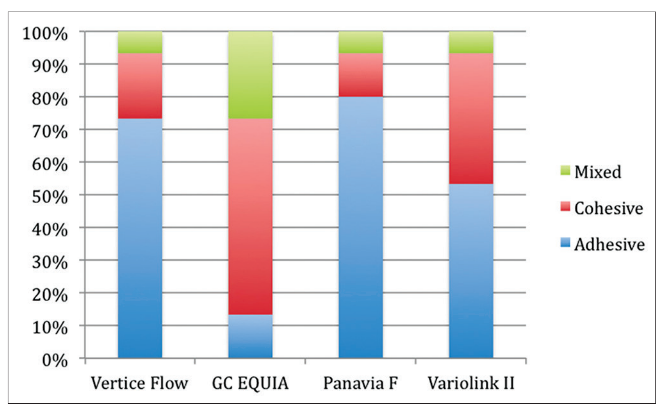

Figure 9: Mean percentages of areas assigned to the failure modes observed in the four adhesive resins

Discussion

During prosthodontic treatments, it is understandably desirable to aim for ease, reduced chair time, and increased patient comfort during the procedures. This *in vitro* study compared the influence of four different adhesive systems on the bond strength between dentin and zirconium. The results show that dentin–zirconia bond strength is dependent on the adhesive systems used. The GC EQUIA

Table 1: Materials used in this study

Material	Manufacturer	Chemical composition
Vertise Flow	Kerr, Orange, CA, USA	Glycerol phosphate dimethacrylate, prepolymerized filler, 1-µ barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride
Panavia F 2.0	Kuraray, Osaka, Japan	Paste A: BPEDMA/MDP/DMA/silica/barium sulfate/dibenzoylperoxide Paste B: N, N-Diethanol-p-toluidine/silica sodiumfluoride
Variolink II	Ivoclar, Ellwangen, Germany	Bis-GMA, UDMA, TEGDMA, barium glass sil. ytterbium fluoride, mixed oxide sil, Ba-Al-fluoro-silicate glass, catalysts, stabilizer, pigment
EQUIA Fill	GC Industrial, Tokyo, Japan	Water, fluoro-alumino-silicate glass, polybasic carboxylic acid, polyacrylic acid

Table 2: The µTBS values in N (SD)

Panavia F	11,17	3,89	c
Vertise flow	8,1	2,75	c
Variolink II	18,16	5,56	b
GC EQUIA	25,19	6,12	a

Same lowercase letters indicate an insignificant difference ($P>0.05$). SD=Standard deviation

group exhibited the highest bond strength compared with the Panavia F, Vertise Flow, and Variolink II groups. Thus, the results partly support the hypothesis that self-adhering adhesive systems exhibit higher bond strength than Panavia F and Variolink II. This result is in agreement with the results of Braga *et al*.,[21,22] who concluded that the composition of adhesive and polymerization forms might influence their properties and bond strengths.

The bond strengths were evaluated with a microshear bond test, as this simple test protocol allows for straightforward specimen preparation.[23,24] The microshear bond strength (µSBS) test could have additional advantages over the microtensile bond strength (µTBS) test because it is performed without the need for sectioning procedures, which could induce early micro-cracking, to obtain specimens.[24,25]

While researchers in many *in vitro* bond strength studies have applied adhesive systems to zirconium disks,[16,26] those methods are not representative of restorative procedures in clinical settings. In the current study, adhesive systems were applied between dentin disks and zirconium rods. It has been speculated that most indirect restoration materials, such as zirconia, restrict access to adequate light intensity. The degree of polymerization is still influenced when a light barrier simulating a zirconia indirect restoration is placed between the light source and the cement. Inadequate light intensity adversely affects bond strength,[27,28] which might explain the low microshear bond strength results in the Panavia F, Variolink II, and Vertise groups in this study. Uo *et al*. reported that glass-ionomer cement (Fuji I) exhibited higher bond strength than adhesive resin cement (Panavia F) with zirconia ceramics. This result confirms our results that GC EQUIA exhibited higher bond strength than the other groups.[29]

GC EQUIA is a self-adhering system composed of high-viscosity glass-ionomer cement. It contains an adhesive monomer, methyl methacrylate (MMA), and functional methacrylate.[30] Chemical adhesion of the material to dental tissues can be added. The properties of GC EQUIA include resin-modified glass cement adhesion to moist tooth structures and base metals, anticariogenic properties due to the release of fluoride, thermal compatibility with tooth enamel, biocompatibility, and low toxicity. The chemical adhesion of resin-modified glass cement to the hard tissue of teeth through a combination of polycarboxylic acids and hydroxyapatite has been cited as the most important advantage of resin-modified glass cement.[31] In the present study, the highest bond strength values were observed in the resin-modified glass cement group, which is explained by the glass-ionomer cement's high monomer conversion and chemical adhesion to the hard tissue of teeth.[32]

Vertise Flow is a self-adhering, light-cure flowable material that eliminates the additional etching/priming/bonding steps necessary to bond a resin composite to dentin or enamel. It incorporates the adhesive technology found in OptiBond products to create proven bonds to the tooth structure. Vertise Flow bonds via two methods: Primarily, through the chemical bond between the phosphate functional groups of a glycerol phosphate dimethacrylate monomer and calcium ions of the tooth, and secondarily, through a micromechanical bond resulting from an interpenetrating network that forms between the polymerized monomers of Vertise Flow and the collagen fibers (as well as the smear layer) of dentin.[33] Due to light-cure polymerization, it has the lowest bond strength compared to the other groups in this study.

Altintas *et al*. reported that Variolink II showed the highest shear bond strength of all resin cements tested (Chemlace II, Suber-bond C and B, and Panavia F).[16] They reported that the SBS of zirconium to Panavia F and Variolink II was 4.0 ± 0.8 and 5.4 ± 2.3, respectively.[16] These results were lower than the values obtained in this study (11.17 ± 3.89 and 18.16 ± 5.56 MPa, respectively). 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) is present in Panavia F and the phosphate ester group of this monomer bonds chemically to aluminum and zirconium oxides.[34-36] However, it represented the lowest bond strength.

The present study also addressed the question of failure modes. The failures were predominantly cohesive in the resin cement in the GC EQUIA and Variolink II groups. However, the adhesive failures in the Panavia F and Vertise flow groups occurred between the zirconium core and the resin cement. No cohesive failures in dentin were observed in the Panavia F, Vertise flow, GC EQUIA, or Variolink II groups, probably because the bond strengths obtained with the different materials were generally lower than the cohesive strength of dentin.[37] The bond strength values might account for the modes of failure at the bonded interface.[16]

It must be noted that only one test (shear bond strength test) was used to evaluate the performance of adhesive materials. The shear bond strength tests are a useful tool to assess the bonding properties between different materials used in restorative dentistry, but no direct extrapolations can be made considering the behavior of these materials under clinical conditions. This may be considered one of the limitations of the current study.

Conclusion

Self-adhering resin cements are promising materials for luting indirect restorations because of their simplified application and reduced technique sensitivity. The available data for GC EQUIA shows better performance compared to other systems, while Vertise Flow had the worst performance, probably because it is a light-cure, self-adhering system. However, long-term clinical studies are necessary to evaluate the *in vivo* performance of self-adhering and other systems.

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