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Effect of resin cement selection on the microtensile bond strength of adhesively veneered 3Y-TZP

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Abstract

Objective. The aim of this study was to investigate the effect of resin cement selection on the microtensile bond strength (μTBS) of adhesively veneered 3Y-TZP. ***Materials and methods.*** 3Y-TZP discs were fabricated from commercial powders and treated by sandblasting and zirconia primer. Porcelain discs were sectioned from a feldspathic block and conditioned with 5% HF and silane agent. Pre-treated surfaces of zirconia and porcelain discs were bonded together using one of the three following resin cements: Multilink N (MN), Panavia F (PA) or RelyX Unicem (RU), respectively. After light-curing the joined discs were cut into microbars where 15 microbars per group were randomly chosen for μTBS test until failure occurred (24 h storage in water in advance, crosshead speed of 0.5 mm/min). The data were analysed by one-way ANOVA and Tukey's test ($p < 0.05$). Fractured zirconia surfaces were examined using both a stereomicroscope and scanning electron microscope to identify the failure mode. ***Results.*** Significant differences in the μTBS values among three groups were found ($p < 0.001$) and the descending order was PA, RU and MN. No zirconia or feldspathic failure occurred, but the zirconia/cement interfaces suffered from fracture for all samples. Cement cohesive failure and/or feldspathic/cement interfacial failure sometimes were involved. Failures were mainly adhesive for RU, while they were mixed for MN and PA. ***Conclusion.*** When using the adhesive veneering method, Panavia F offers better bond strength than Multilink N or RelyX Unicem, which is probably due to the content of the 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) monomer.

Key Words: *adhesion, bond strength, microtensile, resin cement, zirconium oxide*

Introduction

Computer-aided design and manufacturing (CAD/CAM) was introduced to the dental profession by Duret in 1971 [1]. CAD/CAM has become an important technique in modern dentistry and new dental materials have been developed for this use, in particular new ceramics. Machinable ceramics have high quality and reliability which provide the basic guarantee of the restoration's success [2,3]. These machinable ceramics can be categorized into two major groups: chairside system and laboratory-based system [3]. Chairside materials, such as feldspathic, leucite or lithium disilicate porcelain, all possess good aesthetic properties and full-contour restorations can be fabricated in one visit [4]. The use of these chairside materials is generally limited to single units, although a favourable 10-year result on lithium disilicate 3-unit FDPs was reported [5]. Laboratory materials, on the other hand, such as alumina and zirconia ceramics, usually have better mechanical properties. In particular, 3 mol% yttria stabilized zirconia (3Y-TZP) is the strongest available dental ceramic due to the transformation toughening mechanism (from tetragonal to monoclinic crystal symmetry) and can be used to produce multiple-unit restorations [2,6,7]. However, with its limited colour choices and low translucency, the resulting aesthetic effect of zirconia ceramics is not satisfactory [8–10].

Bi-layered all-ceramic restorations are recommended for obtaining the combination of strength and aesthetics. In these restorations, a high-strength framework is obtained by milling porous 3Y-TZP blocks which subsequently are sintered to full density

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and thereafter veneered by fusing porcelain slurry on top. However, the layer-by-layer porcelain fusion procedure is time- and labour-consuming and, most important, chipping of veneering porcelain is a frequent failure of zirconia-based restorations [11–14]. This chipping problem may be caused by several factors, where one is the structural flaws introduced into the porcelain layers during handmade veneering. Such structural flaws can result in premature failure, even at low functional stresses [15]. Some failures may be caused by the excessive residual stresses generated during veneering fusion due to thermal expansion coefficient (TEC) mismatch between veneer porcelain and zirconia [15] and poor thermal conductivity of zirconia cores [16]. There are several other possible factors that might jeopardize the reliability of this kind of restorations [15].

In order to overcome these limitations of the conventional veneering method, great efforts have been made to develop novel veneering methods. As an alternative technique adhesive joining of veneers onto ceramic cores was put forward by Lee et al. [17] in 2007. Both the veneer structure and the zirconia core are fabricated separately using high quality machinable blocks by CAD/CAM and then they are precisely bonded together by adhesives. The effectiveness of this veneering method has been confirmed by comparing the bond strength of adhesively veneered structures with conventional veneered structures [18]. In summary, adhesively veneering could simplify the fabrication procedure, improve reliability of veneers and cores due to less handmade fabrication/adjustments and avoid the formation of thermal mismatch residual stresses [18–20]. However, it shall be noted that individual aesthetics can not be easily achieved by use of prefabricated porcelain blocks. It is expected though that the aesthetics of adhesively-veneered zirconia restorations will be further improved in the future with the development of colour gradient porcelain blocks [21].

Reliable adhesion is the key factor for the success of this new method and resin cement selection is the prerequisite for establishing an effective bond, especially when luting zirconia ceramics [22,23]. Until now few studies have focused on this issue for the adhesively veneered 3Y-TZP [17–20]. Thus, it is of utmost importance to study the influence of resin cement selection on the bond strength of adhesively veneered 3Y-TZP and give some suggestions for the clinical application.

Numerous methods can be applied to measure the bond strength, but the microtensile bond strength (μTBS) test seems to have several advantages [22]. The failure that occurs at the adhesive interface is similar to the real clinical situation [23]. Further, the bonded interface of the specimen is ~ 1 mm^2, resulting in a uniform stress distribution, and a pure tensile force during loading can be ensured due to the free interface of microbar away from the attachment [24]. A potential drawback of this method is that the values of μTBS might be affected by poor quality of the ceramics, but this is not the case when using high quality machinable ceramics, as clinically documented for Vitablocs Mark II and 3Y-TZP ceramic blocks [25,26]. In other words, the μTBS test is a suitable method with high accuracy and sensitivity to evaluate the bond strength of this novel structure based on adhesion.

The aim of this study was to evaluate the effect of resin cement selection on the microtensile bond strength of adhesively veneered 3Y-TZP. The null hypothesis was that there is no difference in μTBS among the adhesively veneered 3Y-TZP structures bonded with three different monomer-containing resin cements.

Materials and methods

Sample preparation

Three cylindrical zirconia discs, 12 mm in diameter and 2 mm in thickness, were made from commercial 3Y-TZP powders (Tosoh Corporation, Tokyo, Japan) through uni-axial dry pressing in a stainless steel die (2 MPa, 1 min; 769YP-24B, Tianjin Keqi High-tech companies, Tianjin, China) followed by cold isostatic pressing (200 MPa, 1 min; LDJ-100/320–300, Chuan-xi machinery industry Co. Ltd., Sichuan, China). Sintering was done at 1450°C in an air environment for a holding time of 2 h (SJG-16, Luoyang Shenjia Kiln Co. Ltd., Henan, China). The accuracy of thickness and parallellism of the disc surfaces were ensured by grinding with a precision milling instrument (ACC52DX, Okamoto Machine Tool Works Ltd., Okamoto, Japan). One surface of each zirconia disc was airborne particle abraded with 50 μm aluminium oxide particles (Renfert GmbH, Hilzingen, Germany) at 0.25 MPa pressure at a distance of 10 mm for 15 s followed by ultrasonic cleaning in distilled water for 5 min (KQ218, Kunshan Ultrasonic Instruments Co. Ltd., Jiangsu, China). Three zirconia discs were subjected to one more firing cycle (1000°C, 15 min) to reverse any phase transformation caused by grinding and abrasion [27] and ultrasonically cleaned under the same condition. The abraded surfaces were then treated by zirconia primer (Metal/Zirconia primer, Ivoclar-Vivadent) for 180 s and dispersed with a strong stream of oil-free air.

Three cylindrical porcelain discs, 12 mm in diameter and 3 mm in thickness, were cut from a feldspathic block (Vitablocs Mark II; Vita Zahnfabrik, Bad Säckingen, Germany) by a precision cutting instrument (J5060; Shanghai radio special machinery factory, Shanghai, China). The location of the cuts was controlled automatically. The porcelain discs

were ultrasonically cleaned in distilled water for 5 min and dried with oil-free air. One surface of each disc was etched with 5% HF acid (IPS Ceramic Etching Gel, Ivoclar-Vivadent) for 2 min and then removed by spraying for 60 s and dried for 20 s with oil-free air. The silane bonding agent (Monobond-S, Ivoclar-Vivadent) was then applied onto the etched surface for 60 s and was allowed to evaporate completely.

Three resin cements, Multilink N (MN, Ivoclar-Vivadent, Schaan, Liechtenstein), Panavia F (PA, Kuraray Co. Ltd., Tokyo, Japan) and RelyX Unicem (RU, 3M ESPE, St. Paul, MN), were applied onto the treated surfaces of randomly chosen zirconia and porcelain discs to bond them together. The thickness of resin cement was ~ 100 μm and this was achieved by pressing the bonded sample to the same height as two 5.10 mm zirconia reference cylinders aided by a flat and heavy glass plate for 15 min. Large excesses were removed carefully using a scraper without destroying the bonded structure. Small cement excesses were left and anti-oxidant (Liquid Strip, Ivoclar-Vivadent) was also used around the bonding interface to ensure complete curing. Three specimens were light polymerized with the same unit at four different locations for 60 s each using a halogen light-curing unit with an output of 700 mW/cm^2 [23]. Materials used in this study are listed in Table I.

Twenty-four hours later, each joined disc was sectioned into several microbars (5.10 mm × 1 mm × 1 mm). The obtained microbars were ultrasonically cleaned in acetone solution for 5 min and in distilled water for 5 min. Thereafter the microbars were stored in demineralized water at 37°C for 24 h (HH-W21-Cr600, Beijing Changan scientific instrument factory, Beijing, China). The microbars were examined using a stereomicroscope (KH-1000; Hirox Co. Ltd., Tokyo, Japan) and only intact microbars were selected for further testing (Figure 1). Fifteen microbars per group (n = 15) were chosen randomly for the μTBS test and their interface areas were measured with a digital caliper (SH6L03722, Wenzhou Sanhe Yiqi Co. Ltd., Zhejiang, China).

Microtensile bond strength test

Each microbar was bonded to the attachment unit using a light-polymerized adhesive resin (Clearfil SE; Kuraray Co. Ltd., Osaka, Japan). Special care was taken to centre the two bonding interfaces at the free space between the two parts of the attachment unit and to align the samples parallel to the loading direction [28,29]. The microbars were tested under tensile load at a cross-head speed of 0.5 mm/min until failure occurred (EZ-L; Shimadzu Corp, Kyoto, Japan).

Microscope examination and fracture mode analysis

The fractured zirconia surfaces were evaluated by using both a stereomicroscope (×100) and a scanning electron microscope (SEM, SSX-550, Shimadzu Corp, Japan). Energy Dispersive Spectroscopy (EDS) was sometimes used as a complementary method to clearly distinguish the materials on the fracture surfaces. The failure modes were classified into six types: cohesive failure in zirconia, veneer or resin cement, adhesive failure at the bonded interface of zirconia/cement or feldspathic/cement and, finally, mixed failure.

Table I. Summary of the materials applied in the study with their main compositions.

Materials	Name (Batch)	Main composition
Resin cement	Panavia F[a] (A:00252A; B:00029F)	A:10-MDP, DMA, silanated silica filler, silanated colloidal silica, dl-CQ, catalysts, initiator B: DMA; silanated Ba-glass filler, catalysts, accelerator, pigment
	Multilink N[b] (N01598)	DMA, HEMA. Ba-glass filler and silica filler, ytterbium trifluoride, catalyst, stabilizer
	RelyX Unicem Aplicap[c] (400559)	DMA, methacrylated phosphoric ester, acetate, glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, pigment, initiator, stabilizer
Zirconia primer	Metal/Zirconia Primer[b] (N01589)	phophonic acid acrylete, solvent, ethoxylated Bis-EMA, initiator, stabilizer
Etching acid	IPS ceramic etching gel[b] (P48565)	5% Hydrofluoric acid
Silane bonding agent	Monobond-S[b] (P57807)	silane methacrylate
Oxyguide	Liquid Strip[b] (N00867)	glycerine gel
Adhesive	Clearfil repair bond-adhesive[a] (01475A)	BisGMA, 2-HEMA, 10-MDP, DMA, colloidal silica, dl-CQ, initiator, accelerator

Manufacturers: [a] Kuraray Medical, Osaka, Japan; [b] Ivoclar vivadent, Schaan, Liechtenstein; [c] 3M Espe, Seefeld, Germany.
10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate; DMA, dimethacrylate; CQ, Camphorquinone; TMSPMA, 3-trimethoxysilylpropyl methacrylate; BisGMA, bisphenol A diglycidylmethacrylate; 2-HEMA, 2-hydroxyethyl methacrylate.

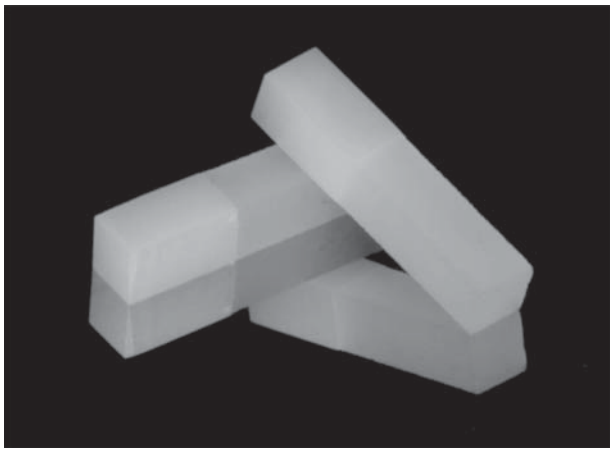

Figure 1. The experimental microbars used for μTBS tests.

Statistical analysis

The values of μTBS were calculated based on the fracture load measured during test. The data were statistically analysed by one-way ANOVA and Tukey's multiple comparison test to determine whether significant differences existed or not. SPSS 13.0 (SPSS Inc., Chicago, IL) was used to analyse the data and $p < 0.05$ was considered significant.

Results

In the present study, the null hypothesis that there is no difference in μTBS among three groups bonded with different monomer-containing resin cements has been rejected. The values of μTBS and distribution of failures of three groups are summarized in Table II. The μTBS was significantly influenced by the resin cement type ($F = 51.357$; $p < 0.001$). Statistically significant differences were observed among the three groups ($p < 0.05$) and the descending order was PA, RU and MN according to the observed μTBS values.

Three characteristic morphologies of fractured zirconia surfaces are shown in Figure 2. Observe that no fracture occurred within feldspathic porcelain or zirconia ceramics during the test. All failures occurred at the zirconia/cement interface, either completely (adhesive failure) or partly (mixed failure). Adhesive failure was the main mode in the RU group, while failures were mainly mixed for MN and PA, especially in the PA group (Table II).

Table II. Mean (SD) of μTBS (MPa) and failures' distribution (amount and percentage) in three groups ($n = 15$).

Group	Mean (SD)	A	M
PA	37.94 (4.54)[a]	6 (40%)	9 (60%)
RU	25.70 (4.92)[b]	14 (93%)	1 (7%)
MN	20.85 (4.81)[c]	9 (60%)	6 (40%)

Different alphabetical letters indicate groups that were statistically different ($p < 0.05$).
A, adhesive failure at zirconia/cement interface; M, mixed failure.

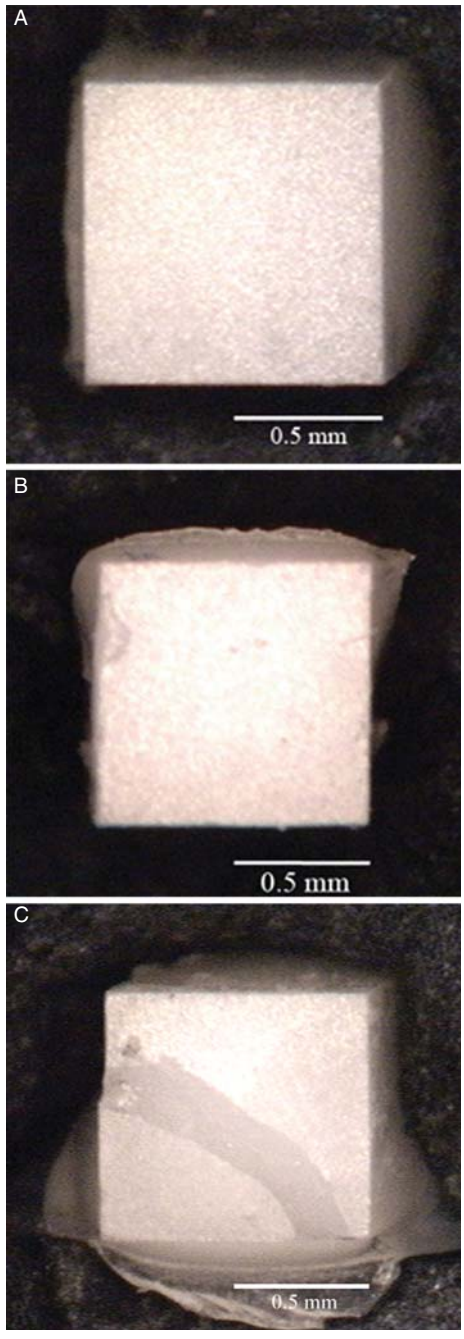

Figure 2. Stereomicroscope images of the fractured zirconia surfaces representing different failure modes. (A) Adhesive failure at the zirconia/resin cement interface; (B) Mixed failure (M-I) and (C) Mixed failure (M-II).

The mixed failures can be further categorized into two sub-types. The commonly occurring type, labelled as M-I, always included partial cohesive failure in resin cement and partial adhesive failure at the zirconia/cement interface (Figure 2B). In the other type of mixed failures, labelled as M-II, adhesive failure at the feldspathic/cement interface co-existed with cement cohesive failure and zirconia/cement interfacial failure (Figure 2C). This was confirmed

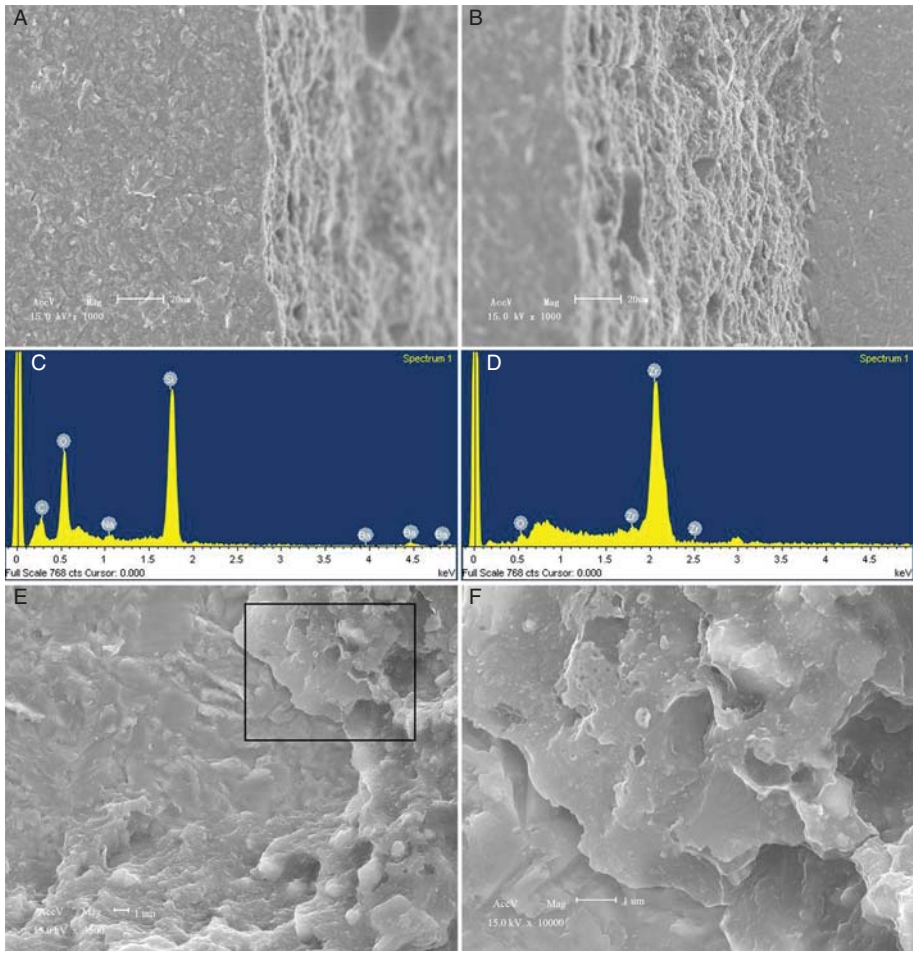

Figure 3. SEM and EDX results of the mixed failure sample showed in Figure 2C bonded with PA. (A) and (B) show the morphology of the upper and the lower exposed flat surface, respectively. Their chemical compositions are shown in (C) and (D), respectively, which confirmed that the material left on the fractured surface was resin cement and the core was pure 3Y-TZP. (E) shows the rough side surface of resin remnants and an unclear boundary between it and the zirconia surface. (F) exposes cracks inside the resin cement.

by simultaneous use of SEM and EDS analysis, as shown in Figures 3A–D.

Further fracture surface analysis of mixed failure samples is shown in Figures 3 and 4. Cement residues were always at the corner or along the edge of the fracture zirconia surfaces. Cement residues of M-I looked like an irregular droplet-shape with a smooth surface and clear boundary (Figure 4). However, in M-II type a large area of cement residues was stuck on the fractured zirconia surface. The fracture side surface of the resin cement was rough and the boundary between it and zirconia surface was not clear, as seen in Figure 3E. At high magnification, further small cracks were revealed to exist inside the resin cement (Figure 3F).

Discussion

Adhesive joining veneers to cores provides an alternative veneering method for the strong 3Y-TZP ceramics. In the present study, the dependence of the μTBS of adhesively veneered 3Y-TZP on the resin cement selection has been verified. The descending order of resin cements according to the μTBS values is consistent with a previous report of zirconia/composite structures bonded with these three resin cements [23]. All failures occurred at the zirconia/cement interface partly or completely. On the contrary, it was noted that failures seldom involved the interface between resin cement and feldspathic porcelain. It indicates that a stronger bonding between resin cement and the glass-rich porcelain is relatively easy to be established. With HF etching and silanization this may occur by micromechanical retention and chemical bonding [30,31]. Therefore the bonding interface of zirconia/cement is the weakest zone in the adhesively veneered 3Y-TZP structure and the bond strength between them would determine the success of this kind of restoration.

Three resin cements were selected in the present study because they have different functional monomers and different physical properties. Panavia F contains the 10-MDP monomer where the terminal phosphate group can react with –OH on the zirconia surface and form chemical bonding between oxygen and zirconium [32]. Especially, with the combination

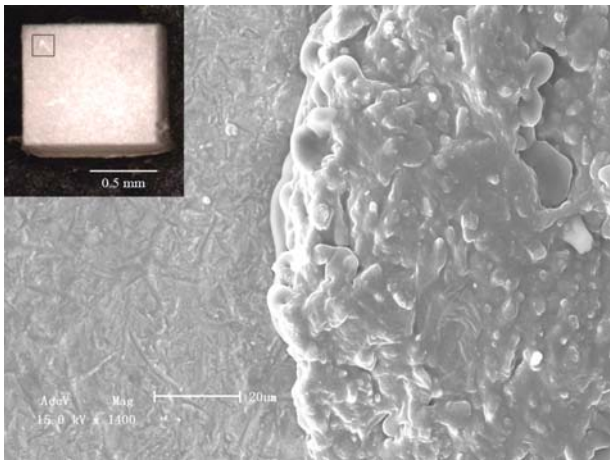

Figure 4. Stereomicroscope and SEM image of one mixed failure sample bonded with RU. A small droplet-like resin remnant is left at the corner of the fractured zirconia surface with smooth surface and clear boundary.

of sandblasting, that would both roughen and increase the available reaction surface, a resin cement with 10-MDP monomer can establish strong bonds with zirconia [33,34]. Although some other phosphate monomers, like methacrylated phosphoric ester and phosphoric acid acrylate, also are contained in RelyX Unicem and Multilink N, respectively, the bonds to zirconia are not as strong as 10-MDP [23]. This is one possible reason why mixed failures occurred more frequently in the PA group than the other two groups always associated with high bond strength values [35].

From fracture surface analysis, different failure modes and morphologies of fracture surfaces might also be related to the different wettability of the resin cements. The wettability of RU cement is poorer than the other two resin cements due to its higher percentage of filler, the larger filler size and its higher viscosity [23,28]. It is difficult for RU cement to sufficiently reach and/or adhere to the rough zirconia surface created by sandblasting to generate full micromechanical retention. In addition, a relative smaller contact area limits the chemical reaction between zirconia and the functional monomer. This illustrates that a combination of good wettability is needed so the function of monomer can be maximized. Large cement residues with an unclear boundary between cement/zirconia observed in the PA group again strengthen this hypothesis that strong bonding is the result of the effective functional monomer and good wettability.

The occurrence of M-II failure in the PA group suggests that the bonding strength between zirconia and PA is even higher than the mechanical strength within PA cement itself. Under this condition the interfacial cracks may deviate during its propagation, pass through the relatively weak resin cement and at the end the adhesive layer is pulled off by microtensile load. Because the thickness of resin cement has not been observed to play an important role when luting zirconia [29], the bonding strength measured in this study might be mainly influenced by the weaker strength of resin cement [36]. The strength of any resin cement is determined by its inherent structural defects. These can be gaps between fillers and other components or even critical voids introduced during the bonding process [37]. From this point of view, resin cement with high content of large fillers and/or high viscosity might be harmful for the composites overall mechanical strength. The occurrence of M-II failures in the PA group also indicates that, due to the good mechanical strength of feldspathic porcelain, cracks prefer to propagate along the feldspathic/cement interface under tensile load.

The resultant tensile stress at the veneer undersurface caused by the compliant adhesive resin cement should be considered when dentists select the adhesives. Stiff enough resin cement could minimize veneer flexure in occlusal loading and improve the bond strength between porcelain and ceramics by reducing the resultant tensile stress [19,20]. The elastic modulus of the three resin cements used in this study are 24.4 GPa (PA), 16.5 GPa (RU) and 6 GPa (MN) according to the manufactures. Considering this, PA could be recommended for this adhesively veneering technique. Nevertheless, this comment shall not be vastly overweighed as the higher the modulus is of a material the more brittle it behaves and the higher the stress will become at the bonding edge [37].

A variation of bond strength resulting from selected resin cement is found in the present study, but no zirconia suffered from fracture. It indicates the potential benefits of the adhesively veneering technique to protect the zirconia core if fracture occurs. Serious consideration should be given when this novel technique is applied in the clinic because *in vivo* the microtensile bond strength might be influenced by thermal cycling. Therefore, the long-term reliability of adhesively veneered 3Y-TZP is very important for prolonged success and this will be studied in the future.

Conclusion

The results of this study suggest that resin cement selection plays an important role on μTBS of adhesively veneered 3Y-TZP. Therefore, when using the adhesive veneering method, Panavia F offers better bond strength than Multilink N or RelyX Unicem, which is probably due to the 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) monomer-content.

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Notice of Correction

The version of this article published online ahead of print on 7 Dec 2012 contained errors. Lines were missing in the legends to some of the figures. This have been corrected for this version.