

Bonding of Glass Ceramic and Indirect Composite to Non-aged and Aged Resin Composite

Marco Gresnigt^a/Mutlu Özcan^b/Maarten Muis^c /Warner Kalk^d

Purpose: Since adhesion of the restorative materials to pre-polymerized or aged resin composites presents a challenge to the clinicians, existing restorations are often removed and remade prior to cementation of fixed dental prostheses (FDPs). This study evaluated bond strength of non-aged and aged resin composite to an indirect resin composite and pressed glass ceramic using two resin cements.

Materials and Methods: Disk-shaped specimens (diameter: 3.5, thickness: 3 mm) (N = 160) produced from a microhybrid resin composite (Quadrant Anterior Shine) were randomly divided into eight groups. While half of the specimens were kept dry at 37°C for 24 h, the other half was aged by means of thermocycling (6000 times, 5°C to 55°C). The non-aged and aged resin composites were bonded to a highly filled indirect composite (Estenia) and a pressed glass ceramic (IPS Empress II) using either a photopolymerizing (Variolink Veneer) or a dual-polymerizing (Panavia F2.0) resin cement. While cementation surfaces of both the direct and indirect composite materials were silica coated (30 µm SiO₂, CoJet-Sand) and silanized (ESPE-Sil), ceramic surfaces were conditioned with hydrofluoric acid (20 s), neutralized, and silanized prior to cementation. All specimens were cemented under a load of 750 g. Shear force was applied to the adhesive interface in a universal testing machine (1 mm/min). Failure types of the specimens were identified after debonding.

Results: Significant effects of aging ($p < 0.05$), restorative material ($p < 0.05$), and cement type ($p < 0.05$) were observed on the bond strength (3-way ANOVA). Interaction terms were also significant ($p < 0.05$) (Tukey's test). After aging, in terms of bond strength, indirect composite and pressed glass ceramic in combination with both cements showed no significant difference ($p > 0.05$). Both indirect composite (24.3±5.1 MPa) and glass ceramic in combination with Variolink (22±9 MPa) showed the highest results on non-aged composites, but were not significantly different from one another ($p > 0.05$). On the aged composites, indirect composite and glass ceramic showed no significant difference in bond strength within each material group ($p > 0.05$), with both Panavia (17.2±6 and 15±5.5 MPa, respectively) and Variolink (19±8, 12.8±5.3 MPa, respectively), but in all groups, glass ceramic-Variolink on aged composite revealed the lowest results (12.8±5.3 MPa). Among all groups, predominantly cohesive failures were observed in the indirect resin composite substrate (79 out of 80) as opposed to the ceramic (18 out of 80) ($p < 0.05$) (Chi square).

Conclusion: Regardless of the resin cement type, considering the bond values and the failure types, the adhesion quality of indirect composite cemented to non-aged and aged resin composite was superior with both cements compared to that of pressed glass ceramic.

Keywords: aging, cementation, glass ceramic, microhybrid composite, silica coating, surface conditioning.

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^a PhD Student, University Medical Center Groningen, Center for Dentistry and Oral Hygiene, Department of Oral Function, Implantology and Clinical Dental Biomaterials, Groningen, The Netherlands. Hypothesis, experimental design, performed experiment, wrote manuscript.

^b Professor, University of Zürich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zürich, Switzerland. Performed part of experiment, contributed to writing manuscript.

^c Dentistry Student, University Medical Center Groningen, Center for Dentistry and Oral Hygiene, Groningen, The Netherlands. Performed part of experiment.

^d Professor, University Medical Center Groningen, Center for Dentistry and Oral Hygiene, Department of Oral Function, Implantology and Clinical Dental Biomaterials, Groningen, The Netherlands. Proofread manuscript.

Correspondence: Prof. Dr. med. dent. Mutlu Özcan, University of Zürich, Dental Materials Unit, Center for Dental and Oral Medicine Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Plattenstrasse 11, CH-8032, Zürich, Switzerland. Tel: +41-44-63 45600, Fax: +41-44-63 44305. e-mail: mutluozcan@hotmail.com

Adhesively bonded restorations offer the advantage of sealing the margins of the restorations, through which the solubility of cements could be minimized. In addition, the adhesive technique not only enables minimally invasive restorations, but also reinforces glassy matrix ceramics.¹ Since retention of the laminate restorations do not rely on mechanical principles, the cementation of such restorations is crucial for long-term success.^{1,14} Successful cementation increases the retention and the fracture resistance of the tooth and the restoration, while also reducing the incidence of microleakage.^{14,15} In addition to form, contour corrections, and color improvement, laminates are commonly indicated especially for situations where resin composite (hereafter: composite) restorations are aged (ie, discoloured, abraded). Replacing the existing resin composite

Table 1 Brand name, manufacturer, composition, and batch number of the materials used in this study

Product name	Manufacturer	Chemical composition	Batch number
PMMA	AutoPlast, Candulor; Altstätten, Switzerland	Polymethylmethacrylate	F42Q28
Quadrant Anterior Shine	Cavex; Haarlem, The Netherlands	Methacrylate-based monomers 23.7 wt%, silica, silicate glass and fluoride containing fillers 75.6 wt%, polymerization catalysts 0.6 wt%, inorganic pigments 0.1 wt%	010100C
IPS Empress II	Ivoclar Vivadent; Schaan, Liechtenstein	Silica, aluminum oxide, lithium oxide, magnesium oxide, zinc oxide, potassium oxide, inorganic pigments	H2941
Estenia	Kuraray; Tokyo, Japan	Urethane tetramethacrylate (UTMA), lanthanum oxide	00002A 00003A 00004G 00005E
CoJet-Sand	3M ESPE; Seefeld, Germany	Aluminum trioxide particles coated with silica, particles size: 30 µm	165092
ESPE-Sil	3M ESPE	Ethyl alcohol, 3-methacryloxypropyl-trimethoxy silane, ethanol	189599 198310
Monobond S	Ivoclar Vivadent	1% 3-methacryloxypropyl-trimethoxy silane, 50% to 52% ethanol	11340
Visio-Bond	3M ESPE	Dicyclopentylidimethylene diacrylate, 2-propenoic acid, 2-methyl,2-(2-hydroxyethyl) (3-methoxypropyl) (aminoP ethyl ester)	260950
IPS Empress ceramic etching gel	Ivoclar Vivadent	5% hydrofluoric acid	J04659 J03578
Variolink Veneer	Ivoclar Vivadent	Urethanedimethacrylate, inorganic fillers, ytterberiumtrifluoride, initiators, stabilizers, pigments	H26575
Panavia F2.0	Kuraray	Silanated barium glass, silanated silica, surface treated sodium fluoride, bis-phenol A polyethoxy dimethacrylate, MDP, hydrophobic dimethacrylate, hydrophilic dimethacrylate, benzoyl peroxide, sodium aromatic sulfinate, N,N-diethanol p-toluidine, photoinitiator	00168A

restorations with new ones may lead to removal of sound dental tissues. In several clinical studies, it was concluded that marginal defects were the main reasons for failures of laminates, and such defects were noticed particularly at the locations where existing restorations were present.^{23,24,32}

Indirect laminates can be fabricated either from composites made of particulate filler composites or ceramics.^{9,18} However, Wakiaga et al⁴⁰ in a Cochrane review recently stated that there is no evidence base for selecting which material should be used for laminate restorations. Particulate filler composites for indirect composite restorations are characterized by a filler/matrix ratio of a given volume or weight of the resin composite and

contain a significantly higher amount of fillers than direct resin composites. One recent indirect composite, Estenia, contains up to 92 wt% compared to the preceding generations, which contain 50 to 80 wt%.^{16,36}

Ceramics and polymeric laminate materials are subjected to different adhesion protocols for durable cementation. For conditioning glassy matrix ceramics, hydrofluoric acid (HF) etching followed by application of a silane coupling agent is recommended as a well established method.^{3,4,7,10,17,21} HF selectively dissolves the glass or crystalline components of the ceramic and produces a porous, irregular surface. These porous surfaces increase the surface area and penetration of resin into the microretentions of the etched surfaces, by which

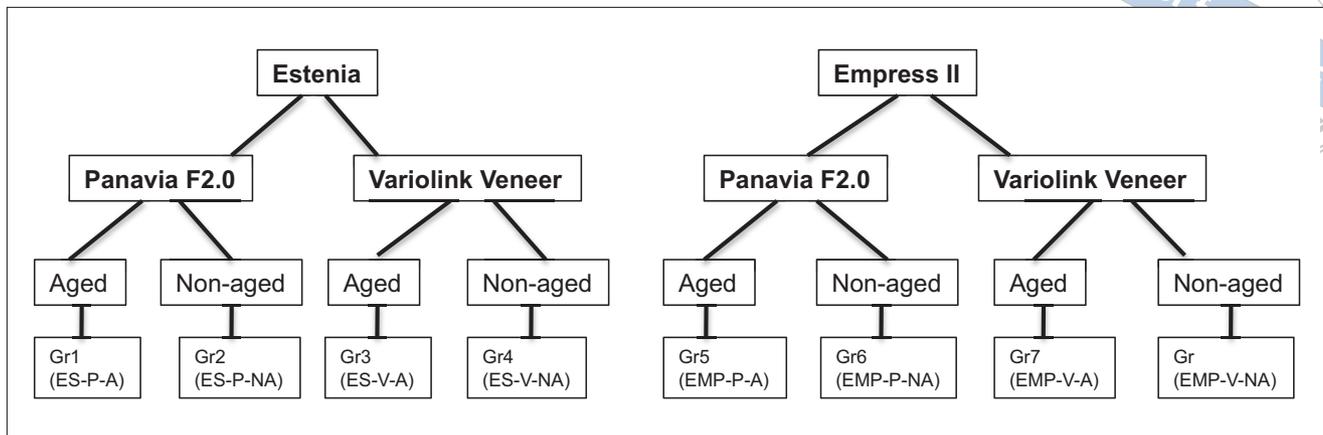


Fig 1 Schematic representation of the experimental groups depending on the restorative material, cement type, and aging conditions of the resin composite. ES-P-A: Estenia, Panavia, Aged; ES-P-NA: Estenia, Panavia, Non-aged; ES-V-A: Estenia, Variolink, Aged; ES-V-NA, Estenia, Variolink, Non-aged; EMP-P-A: Empress, Panavia, Aged; EMP-P-NA: Empress, Panavia, Non-aged; EMP-V-A: Empress, Variolink, Aged; EMP-V-NA: Empress, Variolink, Non-aged.

durable adhesion is achieved. Application of adhesive resin after HF etching further promotes the wettability of the luting cement on the cementation surface of the ceramic.²² On the other hand, for surface conditioning of polymeric materials, recent studies demonstrated favorable results using air-borne particle abrasion with silica-coated alumina particles followed by silanization, as opposed to acid etching and silanization or using alumina air abrasion and silanization.^{2,6,20,21} In the silica-coating technique, due to the blasting pressure, both alumina and silica particles that are attached to the surface further react with the silane. Silane molecules react with water to form three silanol groups ($-\text{Si}-\text{OH}$) from the corresponding methoxy groups ($-\text{Si}-\text{O}-\text{CH}_3$).²⁰ The silanol groups then form a siloxane ($-\text{Si}-\text{O}-\text{Si}-\text{O}-$) network with the silica surface and make covalent bridges with the surface hydroxyl groups: $-\text{Al}-\text{O}-\text{Si}-$. Monomeric ends of the silane molecules react with the methacrylate groups of the adhesive resins by a free radical polymerization process. Resin cements vary depending on their composition. In previous studies, favorable results have been obtained with 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-, or methacrylate-based cements in combination with¹⁹ or without silica coating^{15,25} on glass-based ceramics.

Although many studies concentrated on the adhesion of resins to newly polymerized fresh composites,^{12,27,31,38,39} limited information is available on the adhesion of composites or cements on aged composites.^{6,22} It can, however, be anticipated that fewer surface free radicals are available on aged composites, which may yield inferior adhesion of ceramics and indirect composites on such surfaces compared to non-aged ones. Karlsson et al¹³ reported that 60% of the laminate veneers crossed an existing composite restoration. Hence, more information is required on the durability of laminates on existing restorations. The objectives of this study were therefore to evaluate the bond strength of an indirect composite and pressed glass ceramic, indicated for laminate

restorations, to non-aged and aged microhybrid composite using two different resin cements and to analyze the failure types.

MATERIALS AND METHODS

The brand name, manufacturer, chemical composition, and batch numbers of the materials used in this study are listed in Table 1. Figure 1 schematically represents experimental groups and number of specimens depending on the substrate-adherend combinations and the aging conditions.

Specimen Preparation

The microhybrid composite (Quadrant Anterior Shine, Cavex; Haarlem, The Netherlands), representing the existing restorations under the laminates ($N = 160$, $n = 20$ per group), were packed into polyethylene molds (diameter: 3.5 mm, height: 3 mm) with a hand instrument and photopolymerized incrementally in layers of not more than 2 mm. Each increment was polymerized with a halogen photopolymerization unit (Demetron LC, SDS Kerr; Orange, CA, USA) for 40 s from a distance of 2 mm from the surface. Light intensity was > 450 mW/cm², verified by a radiometer (Demetron LC, Kerr) after every 10 specimens. The surface layer was covered by a glass plate in order to create a smooth surface and to prevent the formation of an oxygen inhibited layer. After polymerization, the polyethylene molds were gently removed from the test specimens.

Disk-shaped indirect resin composites from particulate filler composite (diameter: 17 mm, height: 2 mm) ($N = 80$) (Estenia) were fabricated using a mold on a glass plate. They were heated (100°C to 110°C for 15 min) and photopolymerized (400 to 515 nm for 270 s) using a special polymerization device (Shining 2000, Tecnomedica; Bareggio, Italy) according to the manufacturer's recom-



Fig 2 Representative specimen mounted in the jig of the universal testing machine with adhesive interface submitted to shear force.

Table 2 The mean shear bond strength values in MPa (\pm SD) for the experimental groups

Groups	Mean (+SD)
1 ES-P-A	17.2 \pm 6.0 ^{a,c,d}
2 ES-P-NA	18.7 \pm 3.5 ^{a,b,c}
3 ES-V-A	19.0 \pm 8.0 ^{a,b,c}
4 ES-V-NA	24.3 \pm 5.1 ^b
5 EMP-P-A	15.0 \pm 5.5 ^{c,d}
6 EMP-P-NA	18.7 \pm 3.5 ^{a,b,c}
7 EMP-V-A	12.8 \pm 5.3 ^d
8 EMP-V-NA	22.0 \pm 9.0 ^{a,b}

The same superscript letters in the same column indicate no significant differences ($\alpha = 0.05$). For group descriptions, see Fig 1.

mendations. Ceramic disks (N = 80) (diameter: 17 mm, height: 2 mm) were made of a pressed glass ceramic (IPS Empress II) using the lost-wax technique according to the manufacturer's recommendations.

After fabricating the indirect composite and ceramic specimens, they were all embedded in polyethylene rings (diameter: 20 mm, height: 10 mm) using auto-polymerizing polymethylmethacrylate (PMMA) (AutoPlast, Candulor; Altstätten, Switzerland) ensuring that one surface of the disk remained uncovered for bonding procedures. Cementation surfaces were then finished with 1200-grit silicone carbide abrasive papers under water cooling (Struers; Rodovre, Denmark). This surface area allowed bonding 4 microhybrid composites at a time to the substrate disks.

The microhybrid composites (N = 160) were randomly divided into two groups. While half of the specimens were only stored dry for 24 h at 37°C (non-aged control group), the other half was aged by thermocycling (Willytec; Gräfelfing, Germany) 6000 times (5°C to 55°C, dwell time: 30 s, transfer time from one bath to the other: 5 s).

Surface Conditioning Methods

After aging, the bonding surfaces of all microhybrid composites and the indirect resin composite specimens were conditioned using silica coating and silanization. This was performed using an intraoral air-abrasion device (Dento-Prep, Rønvig; Daugaard, Denmark) filled with 30- μ m alumina particles coated with silica (CoJet-Sand, 3M ESPE; Seefeld, Germany), from a distance of approximately 10 mm at a pressure of 2.5 bars for 4 s. Following surface conditioning, the remnants of sand particles were gently air blown away. The conditioned substrates

were then coated with a 3-methacryloxypropyl trimethoxysilane coupling agent, γ -MPS (ESPE-Sil, 3M ESPE), and left to react for 5 min. Finally, the bonding agent (Visio-Bond, 3M ESPE) was applied with a microbrush, air thinned, and photopolymerized for 20 s.

The ceramic disks were etched with 5% HF acid (IPS Empress Ceramic Etching Gel, Ivoclar Vivadent; Schaan, Liechtenstein) for 20 s, washed and rinsed in a polyethylene cup. They were then neutralized in a diluted solution of neutralizing powder (CaCO₃ and Na₂CO₃) for 5 min, washed thoroughly for 20 s using water, and air dried. Then a silane coupling agent (Monobond S, Ivoclar Vivadent) was applied, allowed to react for 60 s, and air dried according to the manufacturer's instructions.

Cementation

The conditioned substrates (indirect composite and ceramic) and adherends (microhybrid composite) were bonded to each other using either Variolink Veneer (Ivoclar Vivadent) or Panavia F2.0 (Kuraray; Tokyo, Japan) resin cements. Both cements were mixed and polymerized according to each manufacturer's instructions.

The microhybrid resin composites received a thin layer of the luting cement and were then placed on the substrate using an alignment apparatus under a load of 750 g to ensure an even film thickness of the cement. The cement surplus was removed first using the tip of a probe, followed by a microbrush. The bonded area was photopolymerized for 40 s each from two directions. The irradiation distance between the exit window and the bonded interface was maintained at 2 mm to obtain adequate polymerization. An oxygen inhibiting gel (Oxy-

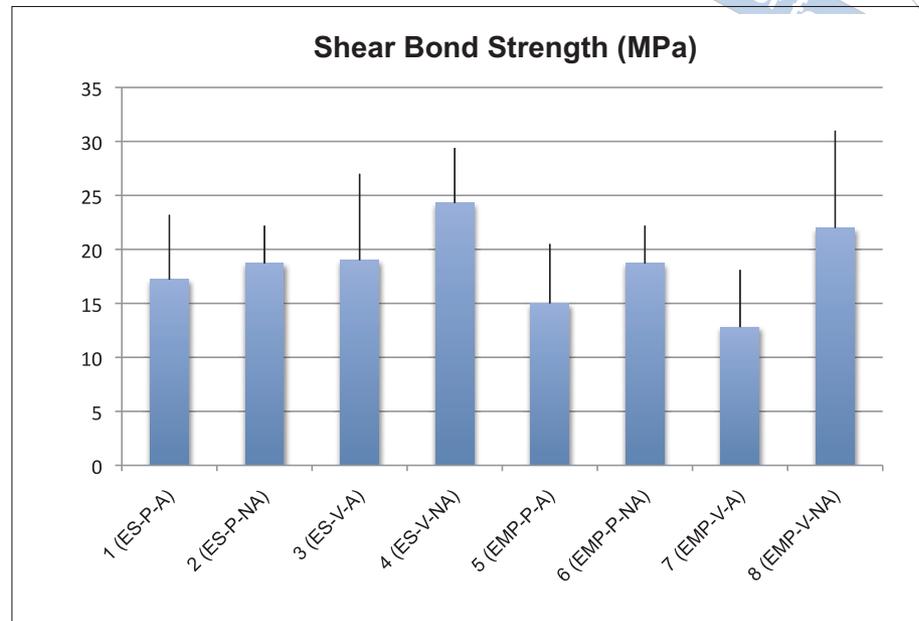


Fig 3 Mean bond strength values (MPa) for substrate-adherend combinations with and without aging. For group descriptions, see Fig 1.

guard II, Kuraray; Okayama, Japan; Batch #00482A) was applied on the free surfaces. After waiting for 5 min, it was washed away, rinsed, and dried. After bonding, the specimens were stored 24 h in distilled water prior to shear bond testing.

Testing Procedure and Failure Analysis

Specimens were then mounted in the jig of a universal testing machine (Zwick ROELL Z2.5 MA 18-1-3/7; Ulm, Germany) and the shear force was applied to the adhesive interface until failure occurred (Fig 2). The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 1 mm/min, and the stress-strain curve was analyzed with a software program (TestXpert, Zwick ROELL). Subsequently, digital photos (Canon Ixus 40, Canon; Tokyo, Japan) were taken of the substrate surfaces.

After debonding, the failure sites were examined by two calibrated operators (M.G., M.Ö.) both visually and from digital photographs using a software program (CoreIDRAW 9.0, Corel; Ottawa, Canada). Failure types were scored using a 5-point scale ranging from 1 to 5, where the substrate was either the indirect composite or the pressed glass ceramic: 1: cohesive failure in the substrate covering >1/3 of the surface; 2: cohesive failure in the substrate covering <1/3 of the surface; 3: cohesive failure of the cement adhered on the substrate covering >1/3 of the surface; 4: cohesive failure of the cement adhered on the substrate covering <1/3 of the surface; 5: adhesive failure with no resin left on the substrate. Representative specimens were sputter coated with 5 to 7 nm of Au/Pd (BAL-TEC sputter coater; type 07 120B; Balzers, Liechtenstein) and evaluated in a scanning electron microscope (JEOL FE-SEM 6301F; Tokyo, Japan) at 20X and 25X magnification.

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS; Chicago, IL, USA). Bond strength data (MPa) were submitted to three-way ANOVA, with the bond strength as the dependent variable and bonding substrate (indirect composite or ceramic) (2 levels), luting material (2 levels), and aging factor (2 levels) as independent variables. Since all corresponding interactions were statistically significant ($p < 0.05$), Tukey's test was used for multiple comparisons ($p < 0.05$). The chi-square test was used to analyze the differences between failure types. P values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Mean bond strength results and significant differences between the experimental groups are presented in Table 2.

Significant effects of aging ($p < 0.05$), restorative material ($p < 0.05$), and cement type ($p < 0.05$) were observed on the bond strength (3-way ANOVA). Interaction terms were also significant ($p < 0.05$) (Tukey's test).

After aging, in terms of bond strength, indirect composite and pressed glass ceramic in combination with both cements showed no significant difference ($p > 0.05$). Both indirect composite (24.3 ± 5.1 MPa) and glass ceramic in combination with Variolink (22 ± 9 MPa) showed the highest results on non-aged composites, but were not significantly different from one another ($p > 0.05$). On the aged composites, indirect composite and glass ceramic showed no significant difference in bond strength within each material group ($p > 0.05$), with both Panavia (17.2 ± 6 and 15 ± 5.5 MPa, respectively) and Variolink (19 ± 8 , 12.8 ± 5.3 MPa, respectively), but in all groups,

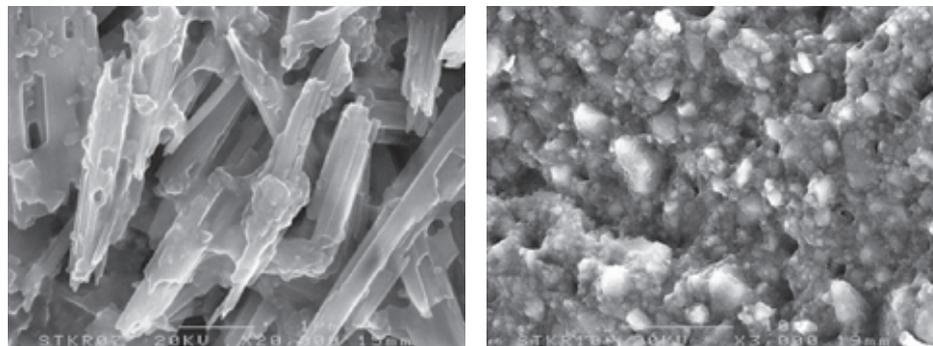


Fig 4a (left) SEM image of typical surface of hydrofluoric acid etched glass ceramic (IPS Empress II) (20,000X magnification). Note the microretentive surface of the ceramic after etching.

Fig 4b (right) SEM image of typical surface of silica coated indirect composite (Estenia) (3000X magnification). Note the presence of the attached silica/alumina particles on the indirect composite after air blasting.

Table 3 Distribution and frequency of failure types between the restorative material (substrate) and the cement per experimental group analyzed after bond strength test

Groups	Score 1	Score 2	Score 3	Score 4	Score 5
1 ES-P-A	7 ^{a,A}	12 ^{b,A}	0 ^{c,A}	0 ^{c,A}	1 ^{c,A}
2 ES-P-NA	20 ^{a,B}	0 ^{b,B}	0 ^{b,A}	0 ^{b,A}	0 ^{b,A}
3 ES-V-A	14 ^{a,A}	6 ^{b,B}	0 ^{b,A}	0 ^{b,A}	0 ^{b,A}
4 ES-V-NA	20 ^{a,B}	0 ^{b,B}	0 ^{b,A}	0 ^{b,A}	0 ^{b,A}
5 EMP-P-A	1 ^{a,C}	2 ^{a,B}	1 ^{a,A}	4 ^{a,A}	12 ^{b,B}
6 EMP-P-NA	0 ^{a,C}	3 ^{a,B}	2 ^{a,A}	8 ^{b,B}	7 ^{b,C}
7 EMP-V-A	0 ^{a,C}	0 ^{a,B}	0 ^{a,A}	11 ^{b,B}	9 ^{b,B}
8 EMP-V-NA	9 ^{a,A}	3 ^{b,B}	0 ^{b,A}	3 ^{b,A}	5 ^{b,C}

Score 1: cohesive failure in the substrate covering >1/3 of the surface; score 2: cohesive failure in the substrate covering <1/3 of the surface; score 3: cohesive failure of the cement adhered on the substrate covering >1/3 of the surface; score 4: cohesive failure of the cement adhered on the substrate covering <1/3 of the surface; score 5: adhesive failure with no resin left on the substrate. Same lowercase superscripts in each row and capital superscripts in each column indicate no significant differences ($p < 0.05$; chi-square test).

glass ceramic-Variolink on aged composite revealed the lowest results (12.8 ± 5.3 MPa) (Fig 3).

SEM analysis showed different surface topographies on the ceramic surface after HF etching and silica/alumina particle deposition on the indirect composite after air blasting (Fig 4). Different surface topographies were noted on the ceramic and the indirect composite after these roughening methods. While on etched ceramic more micromechanical retention was visible due to the removal of the glass matrix, on the air abraded surface the roughness was only obtained by the effect of the sand, where in the latter chemical interactions through

free monomers and silane could be expected to be of more importance.

In all groups, more cohesive failures (scores 1 and 2) were observed in the indirect composite substrate (79 out of 80) than in ceramic (18 out of 80) (Table 3, $p < 0.05$). Ceramic groups presented more adhesive failures between the substrate and the adherend (score 5: 33 out of 40) compared to the indirect composites (score 5: 1 out of 40) ($p < 0.05$). In general, the incidence of score 1 observed in non-aged groups decreased after aging. Images of representative failures from different groups showing scores 1 to 5 are presented in Fig 5.

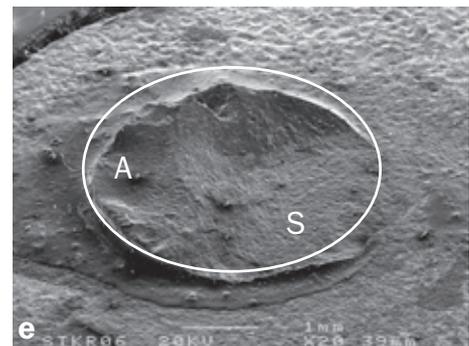
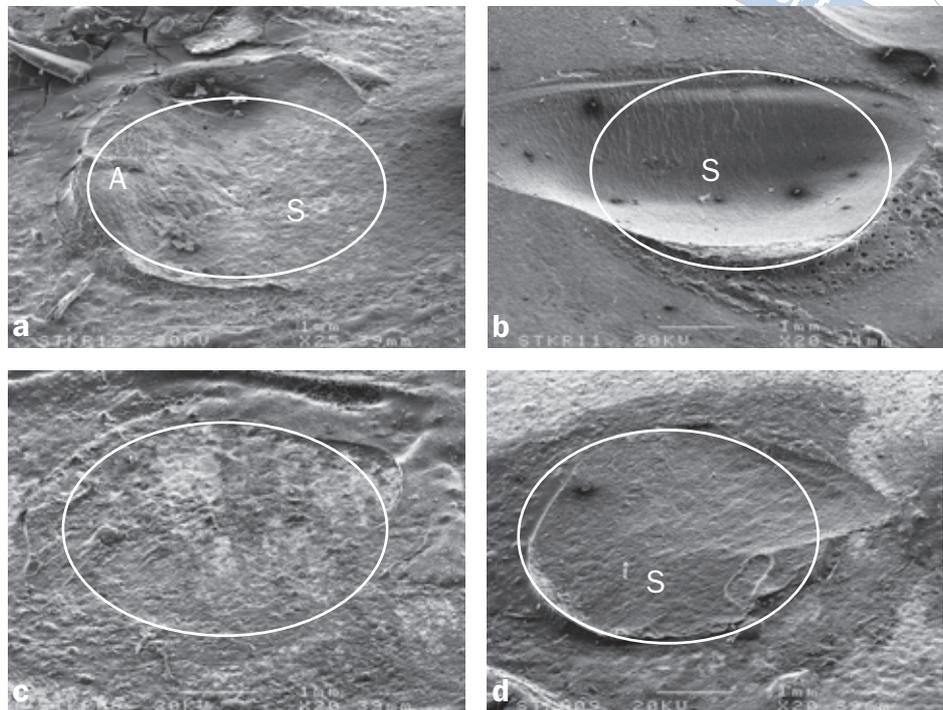


Fig 5a SEM image of representative specimen from group ES-P-A. Cohesive failure in the substrate covering $<1/3$ of the surface (score 2). A= resin cement, S= Estenia.

Fig 5b SEM image of representative specimen from group ES-P-A. Cohesive failure in the substrate covering $>1/3$ of the surface (score 1). S= Estenia.

Fig 5c SEM image of representative specimen from group EMP-P-A. Adhesive failure with no resin left on the substrate in group EMP-P-A (score 5).

Fig 5d SEM image of representative specimen from group EMP-P-A. Cohesive failure of the cement adhered on the substrate covering $<1/3$ of the surface in group EMP-P-A (score 4). S= Estenia.

Fig 5e SEM image of representative specimen from group EMP-P-A. Cohesive failure of the cement adhered on the substrate covering $>1/3$ of the surface in group EMP-P-A (score 3). S= Estenia, A= resin cement.

DISCUSSION

This study was undertaken in order to compare the adhesion of two esthetic restorative materials to non-aged and aged resin composites when bonded with two resin-based luting cements.

Two laminate restorative materials were of interest, namely indirect composite (Estenia) and ceramic (IPS Empress II). Cementation surfaces of both materials were conditioned using different adhesion protocols based on previously reported results.^{3,6,11,20-22} Since lithium-disilicate reinforced IPS Empress II possesses a high crystalline content, the best adhesion results were obtained when they were conditioned with HF acid etching, followed by silane coupling agent compared to other methods, such as alumina deposition or heat treatment and silanization.^{1,17} Thus, the adhesion of composite materials to such ceramics is provided by a combination of mechanical retention that is achieved by HF etching, and chemical

reaction provided by the silane-coupling agent. Similarly, for indirect composite restorations, surface roughening through alumina-silica particle deposition favors micro-mechanical retention, and subsequent application of MPS silanes form the siloxane network ($-\text{Si-O-Si-O}-$).²⁰

In this study, the highest bond strengths were obtained between the Estenia-Variolink Veneer on non-aged composite (24 MPa), but were not significantly different from that of Empress-Variolink Veneer (22 MPa). The indirect composite used was based on urethane tetramethacrylate (UTMA) matrix that contains lanthanum oxide fillers. The high bond strengths could be attributed to durable siloxane bonds between the silica-coated and silanized polymer matrix/filler surface and the bis-GMA-based resin cement. According to previous studies, the degree of conversion of indirect heat and photopolymerized composites range from 47% to 91%.^{16,28,36} In deeper preparations, the polymerization degree has been shown to be less in UTMA-based composites vs those based on bis-GMA,

due to a greater mismatch in the refractive index between monomer and filler.^{28,33} Therefore, some unreacted polymers can be present at the surface which can copolymerize with the methacrylate groups of the silane.²⁸ Even though the degree of conversion of the composite surface was the focus in those studies,^{28,33} in fact, the first layer in contact with the indirect composite consists of the particles attached from the air-abrasion process. In the present study, both dual-polymerizing MDP-containing bis-GMA and photocuring bis-GMA-based cements gave comparable results when non-aged and aged composites were bonded onto the restorative materials tested. When the surface layer contains a less polymerized soft resin layer, this may favor the penetration of the particles that then act as sites for silane reaction in the indirect resin group.²⁶

The results in the indirect resin group bonded to the aged composites tend to give lower bond strengths, but the differences were not significant. The only statistically significant difference was observed when non-aged and aged composites were bonded to the ceramic using Variolink Veneer. Therefore, the hypothesis was only partially accepted. In a study by Kumbuloglu et al,¹⁵ the resin cement Variolink II was bonded to Empress II disks. After 6000 thermocycles between 5°C and 55°C, a mean bond strength of 23.2 MPa was achieved, which was higher than in this study (12.8 MPa). However, the results obtained with Panavia-Empress II in the same study exhibited lower bond strengths (4.3 MPa) than that of this study (15 MPa). The differences between these two studies could be attributed to the aging process employed and the testing procedures. In the study mentioned above,¹⁵ thermocycling was performed after cementation to examine the aging effect on the bonded area. Consequently, the bonded cement/ceramic interface was exposed to aging, whereas in the present study, it was performed prior to cementation on the composite specimens only. The choice of aging methods may have relevance, depending on the objectives of the studies. In previous studies,^{15,20,25} the luting cement was directly attached to the adherend. This cement bulk could be more prone to shrinkage during thermocycling than the thin cement film achieved under 750 g loading. Whether it is the aging effect or the shrinkage occurring at the ceramic/cement interface due to thermocycling needs further investigation. In a study similar to this one, composite disks were adhered with different luting cements to a pressed glass-ceramic (Empress II) with a bis-GMA-based resin cement (Variolink II).²⁵ The ceramic-cement combination exhibited higher shear bond strengths (17.2 MPa) than found in the present study. However, the ceramic-cement complex was thermocycled for only 1000 cycles.^{2,30} Variation in aging conditions in such studies^{2,5,20,30} should be taken into consideration when evaluating the results.

Adhesive quality should not be assessed considering bond strength values alone. According to the scoring system employed, scores 1 and 2 imply cohesive failures in the restorative materials at varying degrees. In principle, when adhesion at the bonded joint exceeds that of the cohesive strength of the substrate, cohesive failures in the substrate occur. This is usually considered reli-

able adhesion.^{6,7,30} While adhesive failures were more common in the ceramic groups, the indirect composite showed exclusively cohesive failures. The effect of aging on the failure types was evident. While non-aged groups failed exclusively cohesively (covering >1/3 of the substrate surface) in both cement groups, when cementation was performed on the aged composites, the cohesively failed area covered <1/3 of the substrate surface. This indicates some kind of decrease in the adhesion quality. However, the existence of cohesive failures indicates sufficient free radicals forming covalent bonds. When the incidence of cohesive failures in the substrate was less (scores 1 and 2), there was a greater tendency toward adhesive failures (score 5) or cohesive failures (scores 3 and 4) in the resin cement.

Because cohesive failures have been associated with the shear test method,² the microtensile test was suggested by Sano et al.²⁹ Since the latter assesses the bond strength of specimens with reduced areas of adhesive joint, fractures occur basically at the adhesive interface. On the other hand, Shahdad et al³¹ reported that loading the specimens under shear could be considered clinically more relevant than flexural or tensile loading, since it produces elements of shear, tensile and compressive stresses that often occur during chewing. The results of this study need to be verified through microtensile testing. Nevertheless, the failures observed were not cohesive in all groups, meaning that this method could still be used for screening or ranking purposes. In this study, usually when bond strength values exceeded 15 MPa in the indirect composites and 22 MPa in the ceramic groups, the frequency of small cohesive defects in the substrate were more common. However, when the results are coupled with the failure types, impaired adhesion could be expected from the laminate restorations bonded onto existing composite restorations.

Several studies investigated the effect of various aging methods, such as long-term water storage, acidic challenges, and thermocycling, on composite-composite adhesion.^{6,15,22} It was concluded that thermocycling could be considered to represent the worst-case scenario, simulating the thermal changes in oral conditions for aging and stressing the composite.⁶ High or elevated temperatures in the oral environment ranging between 5°C and 55°C weaken the composite.³⁴ Temperature alterations could also decrease the number of unreacted double C=C bonds on the surface or within the composite, which consequently may affect the composite-composite adhesion. Furthermore, in clinical situations, it is also likely that the low pH conditions might decrease the functional groups of free radicals.⁶ Therefore, even lower bond strengths can be expected when laminates are bonded to aged composite restorations in vivo. In this study, only the microhybrid restorative composite was subjected to aging conditions, namely 6000 thermocycles that took approximately 1 week. Water uptake and saturation in the composite was not measured, which can be considered a limitation of this study. The bonded joint was not subjected to aging conditions. On the other hand, it should also be noted that in some bonded joints, thermal aging

may even contribute to further polymerization. This may increase the bond strengths.²⁰ Therefore, depending on the substrate-adherend combination thermocycling does not necessarily decrease the bond strength and age the substrate/adherend interface.

Some clinical studies have described failures of laminate veneers bonded to existing restorations.^{9,13,23,32} Marginal defects or debonding were especially noticed at locations where the veneer ended in an existing composite filling. However, in those studies, the surfaces of the existing composites were not conditioned. Such risks could be diminished when laminates are cemented on preconditioned existing composite restorations. Preconditioning could be also achieved with less cumbersome methods than air abrasion methods, for instance, by using functional adhesion promoters based on MDP monomer. Promising results have been reported using the shear test even after 6000 thermocycles.³⁷ With similar promoters in other studies, more adhesive failures in the shear test⁶ or even lower bond strengths with the microtensile test were reported than when silica coating and silanization were used.²¹ Based on the contradictory results, physicochemical activation²⁶ of the surfaces could currently be recommended. The results of this study, however, need to be verified in situations where the enamel and/or dentin surrounds the existing composite restoration, which may contribute to further adhesion.

CONCLUSIONS

Considering the bond values and the failure types, adhesion quality of indirect composite cemented to non-aged and aged resin composite was superior compared to pressed glass ceramic.

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Clinical relevance: Aging of the substrate composites tends to diminish the adhesion quality. The indirect composite Estenia presented good adhesion on both aged and non-aged composites.