

Role of adhesive systems on the luting interface's thickness of ceramic laminate veneers

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Abstract: This *in vitro* study aimed to evaluate the effects of different luting protocols on the thickness of luting interface of ceramic laminate veneers. Thirty-six lithium disilicate blocks (7 × 8 × 0.6 mm) were cemented onto bovine enamel. They were divided into 6 groups based on the luting protocol (no previous photoactivation of the dental adhesive; previous activation of the dental adhesive only on enamel surface; and previous photoactivation of the dental adhesive on both the enamel surface and inner surface of ceramic laminate) and the luting materials used (Single Bond Universal/RelyX Veneer and Tetric N Bond/Variolink Veneer). The luting interface thickness of ceramic laminate veneers was evaluated using a laser scanning confocal microscope (n = 6). The luting interface measurements were analyzed using 2-way ANOVA and the Tukey least significant difference test ($\alpha = 0.05$). Prior activation of the adhesive on the dental enamel and inside the ceramic laminate exhibited higher luting interface thickness than that with no prior photoactivation of both luting materials ($p < 0.05$). Specimens cemented with Tetric N Bond/Variolink Veneer, submitted for prior photoactivation of the adhesive on the dental enamel and on both dental enamel and inner surface of ceramic, exhibited lower luting interface thickness than those luted with Single Bond Universal/RelyX Veneer ($p < 0.05$). The prior photoactivation of dental adhesives influenced the thickness of luting interface in laminate restorations. Tetric N Bond/Variolink Veneer yielded more satisfactory results than Single Bond Universal/RelyX Veneer when the adhesive was light activated.

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Introduction

To avoid unnecessary dental damage, the current restorative dentistry concept recommends a conservative approach in all restorative procedures.¹ Therefore, ceramic laminate veneers associated with adhesive luting procedures are considered a satisfactory alternative since it allows minimum preparation during the treatment.^{2,3,4} Ceramic veneer restorations reproduce polychromatism, translucency, and texture, resulting in a natural and harmonious smile.^{5,6}

The use of dental adhesives and light-cured resin cements is a satisfactory choice for luting ceramic veneers and promoting retention and resistance

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to fracture,^{2,3,7,8,9,10,11} besides the esthetic quality of the restorations.^{12,13} The bonding effectiveness of luting materials is influenced by several factors related to the material itself, such as monomer composition, filler content,^{14,15} and thickness and/or opacity of the ceramic restoration.^{2,3} These could interfere with the efficiency of light-activated luting resin materials and directly affect the polymerization process.^{2,3,13,16}

Adhesive system formulations have been constantly improved to simplify the clinical performance reducing application time. Universal adhesive systems are characterized as a multi-mode adhesive system containing silane and 10-MDP, which are compounds that can improve the bond strength between the ceramic and dental substrate.¹⁷ Silane is a bifunctional molecule that connects the inorganic ceramic substrate with the organic resin matrix. However, materials with greater technical simplicity or fewer operative steps do not always exhibit better performance.^{17,18,19} Moro et al.¹⁸ and Romanini-Junior et al.¹⁹ affirm that the silane incorporated in a universal adhesive does not produce the same adhesive strength as silane applied separately, recommending to apply additional silane in the lithium disilicate restorations.^{18,19}

During the luting procedure, the requirement of prior photoactivation of dental adhesive on the enamel substrate and inside the ceramic laminate is unclear.^{2,3,20} In some situations, it could be performed concomitantly with the activation of the resin cement.²¹ The appropriate photoactivation of the materials

used in the luting procedure is an essential step for esthetic and mechanical longevity of ceramic laminate restorations.^{2,3}

Unfavorable discrepancy on the luting interface of the restorations could produce damage to the tooth and periodontal tissues and contribute to the longevity of oral rehabilitation.^{22,23,24} Higher discrepancies could result in modification of the chemical, physical, and mechanical properties of the luting agents,²⁵ contributing to bacterial microleakage and their subproducts.²⁶ Moreover, the increased cement thickness of restorations can reduce fracture strength.^{25,27,28} Thus, the tooth becomes more susceptible to post-operative sensitivity,²³ secondary caries, and marginal discoloration.^{29,30}

Therefore, the purpose of this in vitro study was to evaluate the effect of different luting protocols on the luting interface thickness of lithium disilicate ceramic laminate veneers, using 2 luting material systems. The null hypotheses tested were: a) that the luting protocol would not promote any difference in the luting interface thickness of the ceramic veneers; and b) that different luting material systems would not result in a difference in the thickness of the adhesive interface.

Methodology

The luting agent materials tested are described in Table 1. This study was submitted and approved

Table 1. Materials, classification, composition, and batch number of materials tested.

Material	Classification	Composition	Batch
Single Bond Universal (3M ESPE)	Adhesive System	MDP, Bis-GMA, HEMA, photoinitiators, dimethacrylate, water, ethanol, silane.	1523700430
RelyX Veneer (3M ESPE)	Resin Cement	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica and colloidal silica. Average particle size of 0.6 mm. Filler loading 66% by weight.	1529200384
RelyX Ceramic Primer (3M ESPE)	Ceramic Primer (silane)	MPS, ethanol, water	1400900844
Tetric N Bond (Ivoclar Vivadent)	Adhesive System	Bis-GMA, UDMA, dimethacrylate, HEMA, phosphonic acid acrylate, nanofillers (SiO ₂), ethanol, initiators and stabilizers	U18895
Variolink Veneer (Ivoclar Vivadent)	Resin Cement	Dimethacrylates, inorganic fillers, ytterbium trifluoride, catalysts and stabilizers, pigments. Filler loading 60.1% by weight.	U13581
Monobond Plus (Ivoclar Vivadent)	Ceramic Primer (silane)	Ethanol, 3-trimethoxysilypropyl methacrylate, 10-MDP, disulfide acrylate	U29879

Bis-GMA: bisphenol-A glycidyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; MPS: methacryloyloxypropyltrimethoxysilane (pre-hydrolyzed silane); UDMA: urethane dimethacrylate.

by the Ethics Committee of the Araçatuba School of Dentistry, São Paulo State University (#15-00673).

Thirty-six ceramic slices ($7 \times 8 \times 0.6$ mm) were made from lithium disilicate blocks (HT B1 IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein) using a cutter machine (Isomet 5000; Buehler, Lake Bluff, USA) and low speed diamond saw under water cooling. The ceramic laminate veneers were sintered in a specific oven (Programat EP 5000; Ivoclar Vivadent, Schaan, Liechtenstein) for 1 hour at 780°C according to the recommendations of the manufacture.^{2,3}

Thirty-six bovine incisors were used in this experimental study; teeth exhibiting fractures and/or cracks were excluded. The crowns of all the teeth were separated from the root, 1.0 mm above the cementum-enamel junction, using a low-speed diamond saw under water cooling. The surfaces of the enamel specimens ($7 \times 8 \times 4$ mm) were also planned using #600 grit silicon carbide paper (Extec Corp., Enfield, USA) under water cooling systems. The teeth were divided into 6 experimental groups according to the luting protocols and the luting materials ($n = 6$).³¹

In SBU_{WPA} group, the enamel surface was etched using 37% phosphoric acid (Dentsply, Mallefer, Ballaigues, Switzerland) for 30 seconds. The acid was removed with deionized water and the enamel was dried with air jets. A layer of dental adhesive (Single Bond Universal; 3M ESPE, St Paul, USA) was actively applied for 20 seconds. Solvent evaporation was performed using an air jet, from an oil-free air-water syringe, for 5 seconds without activation of the adhesive system. The air nozzle was positioned at 45° to the enamel surface at a distance of approximately 1.5 cm from the sample.³² The intaglio surface of lithium disilicate ceramic was conditioned using 10% hydrofluoric acid (Dentsply, Mallefer, Ballaigues, Switzerland) for 20 seconds, washed with deionized water, and dried with air jet. The silane (RelyX Ceramic Primer; 3M ESPE, St Paul, USA) was actively applied to the etched surface for 60 seconds and dried with air jet for 5 seconds. The dental adhesive (Single Bond Universal; 3M ESPE, St Paul, USA) was applied for 20 seconds without activation, and the solvent evaporation was performed as described above. Translucent light-activated resin cement (RelyX Veneer; 3M ESPE, St Paul, USA) was applied

on the intaglio ceramic surface and the restoration was positioned on the dental enamel substrate. To standardize the luting procedure, a load of 4.9 N was applied for 5 seconds on the restoration and then removed prior the photoactivation. The excess resin cement was removed with a microbrush and activated with a LED polywave unit (Valo polywave; Ultradent, South Jordan, USA) for 30 seconds inside a black box to prevent external light interference. A radiometer (Ecel RD7, Dabi Atlante, Ribeirao Preto, Brazil) was used to measure the luminous intensity of the polymerization light at 1584 mW/cm^2 .^{2,3}

SBU_{PAE} group specimens were treated as described for the SBU_{WPA} ; however, the dental adhesive on the enamel surface was previously activated for 10 seconds. In SBU_{PAEC} , the dental adhesive on the enamel surface and inner surface of the ceramic laminate veneer was previously activated for 10 seconds.

TNB_{WPA} group specimens were similarly luted as described for the SBU_{WPA} ; however, the luting materials used were Tetric N Bond dental adhesive, Monobond Plus silane agent, and medium value shade Variolink Veneer resin cement (Ivoclar Vivadent, Schaan, Liechtenstein). In TNB_{PAE} group, the specimens were submitted to a similar protocol realized in SBU_{PAE} group, while TNB_{PAEC} group protocol was similar to SBU_{PAEC} group, but the same materials as TNB_{WPA} group were used. All specimens were stored in distilled water for 24 hours at 37°C in light-protected recipients.

Specimens from each experimental group were submitted to an aging process using the EQUV UV-accelerated aging machine (Equilam, Diadema, SP, Brazil) with eight fluorescent lamps (40 W each) according to the ASTM -G154 test (American Society for Testing and Materials).^{2,3} Alternating periods of exposure of UV-light and condensation with distilled water were established under conditions of 100% relative humidity and heat. The aging process was performed for 252 hours, totaling 21 aged cycles. For 168 hours, UV-light was used at $60 \pm 3^{\circ}\text{C}$, while a condensation phase without light action was performed at $45 \pm 3^{\circ}\text{C}$ for 84 hours.^{2,3,33,34}

All specimens were sectioned perpendicular to the occluso-apical direction of the restoration with a low-speed diamond saw under water cooling using

Isomet 5000 (Buehler, Lake Bluff, IL, USA) to obtain 3 slices of the central portion. The slices were fixed in acrylic resin (Classico, Sao Paulo, Brazil), finished with #320, #600, #800, and #1200 grit silicon carbide paper. The specimens were polished using diamond pastes (#6, #3, #1, and #0.25 μm) for 3 minutes each. They were cleaned in an ultrasonic unit (Cristofoli, Campo Mourao, Brazil) using deionized water for a period of 8 minutes, between the silicon carbide papers, diamond pastes, and at the end of the process. The specimens were stored in Hanks solution (Sigma Chemical Co., St. Louis, USA) to prevent cell degradation.^{2,3,35}

The thickness of the adhesive interfaces were measured using a laser scanning confocal microscope (LEXT OLS4000; Olympus Schweiz AG, Switzerland).³¹ The adhesive interface analysis was performed at low magnification to obtain an overview of the specimen topography and the specific area was localized using a high magnification. Nine measurements were carried out in 3 different regions; 3 measurements were done at each edge of the restoration specimen and other 3 were evaluated in the mid region. Representative images of the experimental groups were obtained using laser scanning confocal microscope to evaluate the adhesive interface subjected to different luting protocols. Adhesive interface thickness was analyzed using 2-way ANOVA and the Tukey least significant difference test ($\alpha = 0.05$).

Results

Table 2 shows the adhesive interface measurements of ceramic veneers analysis, which demonstrated that the restorations undergoing prior activation of dental adhesive on the dental enamel and ceramic exhibited higher luting interface thickness than those not undergoing prior activation for both luting systems evaluated ($p < 0.05$). Comparing the resin

luting systems, no difference was observed between the Single Bond Universal/RelyX Veneer and Tetric N Bond/Variolink Veneer luting systems in the interface thickness of specimens submitted with no prior activation of the dental adhesive ($p = 0.08$). However, Tetric N Bond/Variolink Veneer exhibited lower luting interface thickness than Single Bond Universal/RelyX Veneer luting system ($p < 0.05$), when the dental adhesive was previously activated on both the enamel substrate as well as on the dental enamel and inner ceramic surface (Table 2).

Discussion

The luting protocol influenced the luting interface thickness of ceramic laminate veneers, and so the first null hypothesis was rejected. The use of different luting materials resulted in differences in adhesive interface measurements, leading to rejection of the second null hypothesis.

The adhesive interface consists of a luting line between the ceramic substrate and the dental surface, and the vertical thickness of this interface becomes a determining factor in establishing favorable characteristics of marginal adaptation.^{36,37} A thicker luting interface exposed to the oral environment may facilitate bacterial adhesion and result in gingival inflammation, periodontal disease, and marginal staining, among other complications.^{36,37}

From Table 2, it can be seen that the groups with no prior dental adhesive photoactivation exhibited lower adhesive interface thickness than those groups subjected to previous adhesive activation on the dental enamel and inside the ceramic laminate (Table 2). It is speculated that this result was due to the mixing of previously non-polymerized monomers of the dental adhesive with fillers and monomers from the light-activated resin cement,^{2,3,38,39} which resulted in

Table 2. Mean \pm standard deviation of luting interface thickness of ceramic laminate veneers (μm) as function of luting protocol and luting agent materials.

Variable	Without previous activation	With previous activation in tooth surface	With previous activation in tooth surface and ceramic
Single Bond Universal/RelyX Veneer	101.80 \pm 51.81 Ac	252.43 \pm 12.93 Ab	311.59 \pm 27.99 Aa
Tetric N Bond/Variolink Veneer	109.89 \pm 48.04 Ac	176.92 \pm 39.46 Bb	263.34 \pm 41.96 Ba

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences ($p < 0.05$).

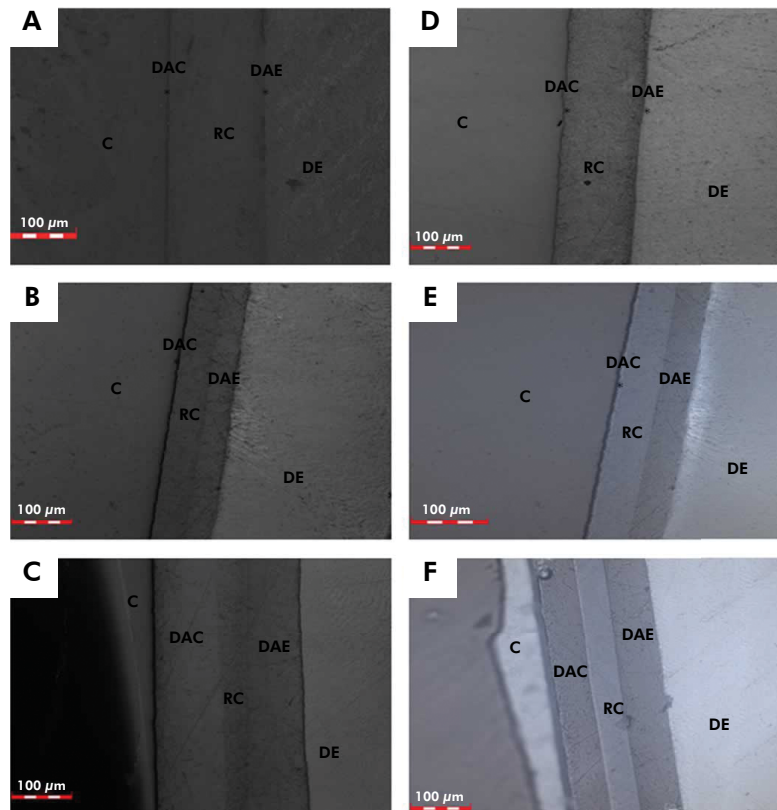
a thin adhesive interface between the ceramic and enamel substrate (Figure A and D).

In Figure C and D, it is possible to verify the increased thickness of the adhesive interface due to the previous adhesive activation. This could damage the chemical, physical, and mechanical properties of the luting agents,²² besides reducing the adhesive interface fracture strength of the ceramic restorations.²⁴ According to Melo Freire et al.,³⁷ the adhesive interface of indirect restorations can be considered acceptable when it presents luting interface thickness values of approximately 120 μm and when the interface quality presents a uniform aspect with no crack or irregularities presence.³⁷ According to these requirements, the groups with no

prior photoactivation of dental adhesives are within clinically acceptable limits.³⁷

The thickness of the adhesive interface could vary according to the specific properties of the resin luting materials.²⁵ Variolink Veneer presents an approximate filler loading of 60% by weight, compared to 66% of RelyX Veneer (Table 1). This characteristic confers low viscosity to resin luting material, allowing higher flow of the light-activated resin cement in a veneer luting procedure. This fact could have contributed to the difference of measurements of the luting adhesive interface of ceramic restorations with different luting material systems (Table 2).

The solubility of resin agents is directly related to the presence and incorporation of water into the



C: ceramic laminate; DAC: dental adhesive in ceramic laminate; RC: resin cement; DAE: dental adhesive on the dental enamel; DE: dental enamel.

Figure. A. Adhesive interface of Single Bond Universal/RelyX Veneer luting materials with no prior dental adhesive photoactivation. B. Adhesive interface of Single Bond Universal/RelyX Veneer luting materials undergoing prior dental adhesive photoactivation on the dental enamel. C. Adhesive interface of Single Bond Universal/RelyX Veneer luting materials undergoing prior dental adhesive photoactivation on the dental enamel and inner surface of ceramic substrate. D. Adhesive interface of Tetric N Bond/Variolink Veneer luting materials with no prior dental adhesive photoactivation. E. Adhesive interface of Tetric N Bond/Variolink Veneer luting materials undergoing prior dental adhesive photoactivation on the dental enamel. F. Adhesive interface of Tetric N Bond/Variolink Veneer luting materials undergoing prior dental adhesive photoactivation on the dental enamel and inner surface of ceramic substrate.

resin matrix of the restorative material,^{2,11} promoting unsatisfactory clinical behavior of these materials.³ This is because significant concentration of water may result in irreversible damage, such as hydrolytic degradation of the chemical agents and hygroscopy of the resin interface.^{1,2} Single Bond Universal contains water and ethanol as solvents in its composition (Table 1), as compared to Tetric N Bond that contains only ethanol as the solvent.^{2,3} This favors volatilization during the luting process of ceramic veneers and avoids water sorption by hydrophilic monomers.^{3,40} The previous activation of Tetric N Bond could have facilitated the solvent volatilization due to gradual increase in temperature from the polymerization light,^{3,11} contributing to a relatively thin adhesive layer compared to the similar usage of Single Bond Universal. However, as Single Bond Universal dental adhesive contains water associated with ethanol,² the volatilization process of the ethanol solvent may have been minimized, thus promoting the formation of a relatively thicker adhesive layer with the residual water/solvent.^{2,41} This could explain the difference between Tetric N Bond/Variolink Veneer and Single Bond Universal/RelyX Veneer luting systems in both luting protocols (Table 2).

Furthermore, RelyX Veneer light-activated resin cement comprises triethylene glycol dimethacrylate (TEGDMA) (Table 1),^{2,39} which promotes water sorption into resinous matrix when combined with bisphenol A-glycidyl methacrylate (Bis-GMA) based resin materials.^{1,2,5} Water sorption is directly proportional to the TEGDMA concentration,^{1,2,5} and this could explain the hygroscopic increase in the adhesive interface and, consequently, the increased luting interface thickness. However, Tetric N Bond dental adhesive is comprised of the urethane dimethacrylate (UDMA) monomer (Table 1),

a hydrophobic compound that allows minimal water sorption into the resinous interface.^{1,3,15} This could explain the lower adhesive interface thickness seen in Tetric N Bond/Variolink Veneer luting system than Single Bond Universal/RelyX Veneer (Table 2).

The present study provides information about the discrepancies of luting interface thickness of lithium disilicate ceramic veneers. The results demonstrated that different luting protocols and systems influenced the adhesive interface thickness of ceramic laminate veneers, suggesting that the absence of prior activation of adhesive with Tetric N Bond/Variolink Veneer luting system is a satisfactory luting technique for ceramic laminates. However, as this study was *in vitro*, the transference of the laboratory results to clinical conditions must be carried out with precaution as *in vitro* evaluation cannot replicate conditions similar to that of the oral cavity.⁴² Further studies are required to evaluate bond strength, permeability, and other physical properties to improve the luting techniques and clinical longevity of ceramic laminates.

Conclusion

Based on the methodology and results obtained in this study, it can be concluded that the prior photoactivation of dental adhesives influenced the thickness of luting interface in laminate restorations, and that the Tetric N Bond/Variolink Veneer yielded more satisfactory results than Single Bond Universal/RelyX Veneer when the adhesive was light activated.

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