



Influence of different adhesive protocols on ceramic bond strength and degree of conversion of resin cements



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ABSTRACT

Since light-curing through ceramic dental restorations can be attenuated by the material crystalline structure, the use of specific adhesive protocols might enhance bonding effectiveness of dual-cure resin cements. This study evaluated the micro-shear bond strength (μ SBS) of different adhesive protocols containing dual-cured resin cements bonded to two glass ceramics: fluorapatite leucite (FLC) and lithium disilicate reinforced ceramic (LDC), and their effect on the degree of conversion (DC) of resin cements. For each ceramic, eight adhesive protocols were tested using combinations of three different resin cements and four adhesive resins. Following the adhesive resin application on ceramic disk surface, resin cement cylinders were produced. After 24 h, the μ SBS test was performed ($n=8$), a shear load was applied at a crosshead speed of 0.5 mm/min until failure and fracture patterns were determined. Resin cement DC analysis was performed by Fourier Transform Infrared Spectroscopy ($n=5$). Data were statistically analyzed using two-way ANOVA, followed by Tukey test ($\alpha=0.05$). The interaction of adhesive protocol and ceramic type significantly affected the micro-shear bond strength and resin cement DC ($p < 0.0001$). For the FLC, adhesive protocols containing the conventional resin cement produced higher μ SBS values compared to the remaining protocols. For the LDC, the combination of the conventional resin cement and an adhesive resin containing photoactivators produced higher μ SBS compared to the other tested adhesive protocols. The conventional resin cement and the self-etch cement produced higher conversion values when luted to the LDC. Selection of specific adhesive protocols should be carefully considered to improve bonding to glass ceramics.

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1. Introduction

In recent years, the use of indirect metal-free ceramic restorations has grown considerably due to the increased demand for esthetic restorative procedures in dentistry. Ceramic restorations have physical–mechanical properties that comply with current clinical demands [1–4], including favorable optical characteristics, chemical stability, biocompatibility and adequate strength, providing highly esthetic-functional treatment options [5]. In order to obtain acceptable clinical results, it is imperative that a strong and a stable link between the ceramic restoration and the tooth structure be created [6]. As a consequence, resin cements are the material of choice for adhesive luting of all ceramic restorations

[6–8]. Ceramics used for dental restorations are brittle materials with high elastic modulus [9] that rely on the retention and support derived from micro-mechanical and/or chemical bonding of the luting agent to the tooth substrate [6]. In this sense, the cementation protocol can be essential for the success of all-ceramic restorations [1,2].

There is no consensus in the scientific literature about the most favorable adhesive protocol for the various ceramic systems currently available. Although the use of adhesive resins may be criticized [10], it is recommended that the bonding surface be initially etched using hydrofluoric acid, followed by the application of a silane agent to ceramics containing silica [11–13] and a low viscosity adhesive resin [14,15] to achieve adequate bonding between resin-based cements and glass ceramics restorations. The combination of resin cements and less viscous adhesive resins to lute dental ceramics depend on the ceramic microstructure [6] and the surface treatment previously performed [6,16]. As consequence, wetting of the ceramic bonding surface by adhesive

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resins is critical to establish optimal bonding between ceramic and resin materials [17]. Moreover, adhesive resins present variable compositions to improve conversion, including, photo-initiators, tertiary amines, sulfinate compounds in order to optimize bonding. After curing, the adhesive resin bonds with the underlying resin cement and becomes micromechanically interlocked within the etched ceramic creating a link between restoration and tooth structure.

Besides resin cement selection [18], proper polymerization of the luting resin is crucial to improve the reliability of the ceramic restorations [19]. Inadequate monomer polymerization can be associated with lower mechanical properties of resin materials [20,21]. The ability of light to reach the adhesive interface is strongly attenuated by either the distance from the light source or by the absorbing characteristics of the indirect restorative materials [19], reducing the total energy reaching the luting agent. This attenuation is dependent on the crystal structure, thickness and shape of the indirect ceramic restoration [22–24]. Even though dual-cured resin cements have been developed to overcome the inability of light to completely reach the bonding resin underneath indirect restorations, [19] the reduction of transmitted irradiance when light curing is performed through the ceramic restorations can influence bond strength and degree of conversion of dual-cure adhesives systems [19,25].

In face of to the great variety of bonding materials currently available presenting different monomer compositions and chemical properties, questions arise about the best choice of resin cements and the most favorable adhesive protocol to be used for luting different ceramic systems. Therefore, the aim of this study was to evaluate the influence of different ceramic bonding protocols on the degree of conversion and bond strength of one conventional, one self-etch and one self-adhesive resin cement bonded to fluorapatite leucite and lithium disilicate reinforced ceramics. The null hypothesis to be tested was that different adhesive protocols do not influence the degree of conversion and micro-shear bond strength of resin cements bonded to glass ceramics.

2. Material and methods

Sixty-four ceramic blocks (12 mm diameter, 2 mm height, shade A2) were prepared using one fluorapatite leucite glass-ceramic (IPS d.SIGN, Ivoclar Vivadent, Schaan, Liechtenstein) and one lithium disilicate ceramic (IPS e.Max Press, Ivoclar Vivadent, Schaan, Liechtenstein) (Table 1) totaling 128 blocks. Ceramic blocks were randomly assigned to eight adhesive protocols (16 groups/ $n=8$). The ceramic bonding surfaces were standardized by wet-polishing (Aropol 2V, Arotec, Cotia, SP, Brazil) with increasingly fine silicon carbide paper 1000, 1200 and 2000-grit (Buehler-Met II, Buheler, Germany) and ultrasonically cleaned for five minutes. The combination of three dual-cure resin cements: (i) one conventional (RelyX ARC, 3M ESPE, St Paul, USA), (ii) a self-etching (Panavia F, Kuraray CO, Osaka, Japan) and (iii) a self-adhesive (U100, 3M ESPE, St Paul, USA); and four adhesive systems: (i) one BisGMA/HEMA/10-MDP hydrophobic component from a self-etching system (Clearfil SE Bond, Bond, Kuraray CO, Osaka, Japan), (ii) a self-etching hydrophilic bond resin with activators (Ed primer, Kuraray CO, Osaka, Japan), (iii) a hydrophobic BisGMA/HEMA bond resin from a conventional adhesive system (Scotchbond Bond Multi-Purpose Plus, Adhesive, 3M ESPE, St Paul, USA) and (iv) a hydrophobic BisGMA/HEMA bond resin that incorporates the peroxide component of a self-cure resin system (Scotchbond Bond Multi-Purpose Plus, Catalyst, 3M ESPE, St Paul, USA) were performed on the ceramic blocks according to the established experimental groups.

2.1. Ceramic surface etching and silanization

The polished surfaces were acid etched with 10% hydrofluoric acid (Dentsply, Petropolis, Brazil): the fluorapatite leucite ceramic blocks were etched for 60 s [10] and the lithium disilicate reinforced ceramic disks for 20 s [8]. Ceramic blocks were ultrasonic cleaned in distilled water for 4 min and completely air-dried for 60 s with oil-free compressed air. Two silane agents were applied according to the resin cement used: for the conventional resin

Table 1
Materials, compositions and manufactures.

Material	Composition	Manufacturer
IPS d.SIGN	SiO ₂ : 50–65 wt%, Al ₂ O ₃ , K ₂ O, Na ₂ O, CaO, P ₂ O ₅ , F, Li ₂ O, ZrO ₂ and pigments (fluorapatite leucite glass-ceramic) Lot: K33292	Ivoclar Vivadent, Schaan, Liechtenstein
IPS e.Max Press	SiO ₂ , Li ₂ O, K ₂ O, MgO, ZnO, Al ₂ O ₃ , P ₂ O ₅ and other oxides (lithium disilicate glass-ceramic) Lot: M72418	Ivoclar Vivadent, Schaan, Liechtenstein
RelyX ARC	TEGDMA, bis-GMA, zirconia/silica filler (67.5 wt%) initiators Lot: FY8HX	3M ESPE, St Paul, MN, USA
RelyX U100	Phosphoric acid methacrylates, dimethacrylates, inorganic fillers (72 wt%), fumed silica, initiators Lot: CA3RW	3M ESPE, St Paul, MN, USA
Panavia F	Paste A: 10-MDP, hydrophilic and hydrophobic dimetacrilates, benzoyl peroxide, camphorquinone, colloidal silica Lot: 249D Paste B: Sodium Fluoride, hydrophilic and hydrophobic dimetacrilates, d- <i>p</i> -tol, T-sulfinate, colloidal silica, barium glass, titanium dioxide Lot: 26D	Kuraray, Osaka, Japan
Adper Scotchbond multi-purpose plus	Adhesive: bis-GMA, HEMA, photo-initiators, amines Lot: 9CC Catalyst: bis-GMA, HEMA, peroxides Lot: 9RL	3M ESPE, St Paul, MN, USA
Clearfil SE bond	Primer: HEMA,10-MDP, Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, Water, Accelerators Lot: 01714-A Bond: 10-MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate dl-Camphorquinone,d- <i>p</i> -tol, colloidal silica Lot: 07706-A	Kuraray, Osaka, Japan
ED primer	Primer A: HEMA, 10-MDP, N-methacryloyl-5-aminosalicylic acid, diethanol- <i>p</i> -toluidine, water Lot: 00226A Primer B: N-methacryloyl-5-aminosalicylic acid, T-sulfinate, diethanol- <i>p</i> -toluidine, water Lot: 00105A	Kuraray, Osaka, Japan
Ceramic primer	Ethyl alcohol, water, Methacryloxypropyltrimethoxysilane Lot: 8YH	3M ESPE, St Paul, MN, USA
Clearfil porcelain Bond activator	3-Trimethoxysilylpropyl methacrylate, hydrophobic aromatic dimethacrylate Lot: 00208B	Kuraray, Osaka, Japan

Abbreviations: Bis-GMA=bisphenol A-glycidyl methylmethacrylate; HEMA=hydroxyethyl methacrylate; UDMA=urethane dimethacrylate; TEGDMA=triethylene glycol dimethacrylate; 10-MDP=10-methacryloyloxydecyl dihydrogen phosphate; d-*p*-tol=diethanol-*p*-toluidine; T-sulfinate=T-isopropyl benzenic sodium sulfinate.

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