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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 (μSBS) of different adhesive protocols containing dual-cured resin cements bonded to two glass ceramics: fluorapatite leucite (FLC) and lithium dissilicate 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 μSBS 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 μSBS values compared to the remaining protocols. For the LDC, the combination of the conventional resin cement and an adhesive resin containing photoactivators produced higher μSBS 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.

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 dissilicate 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 dissilicate 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 dissilicate reinforced ceramic disks for 20 s [8]. Ceramic blocks were ultrasonic cleaned in distillied 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_2O_3, K_2O, Na_2O, CaO, P_2O_5, F, Li_2O, ZrO_2 and pigments (*fluorapatite leucite glass-ceramic*) Lot: K33292	Ivoclar Vivadent, Schaan, Liechtenstein
IPS e.Max Press	SiO_2, Li_2O, K_2O, MgO, ZnO, Al_2O_3, P_2O_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-*p*-tol, T-sulfinate, colloidal silica, barium glass, titanium dioxide Lot: 26D	Kuraray, Osaka, Japan
Adper Scothbond 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-*p*-tol, colloidal silica Lot: 07706-A	Kuraray, Osaka, Japan
ED primer	*Primer A:* HEMA, 10-MDP, N-methacryloyl-5-aminosalicylic acid, diethanol-*p*-toluidine, water Lot: 00226A *Primer B:* N-methacryloyl-5-aminosalicylic acid, T-sulfinate, diethanol-*p*-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-isopropylic benzenic sodium sulfinate.

cement (RelyX ARC, 3M ESPE) and the self-adhesive (U100, 3M ESPE), a pre-hydrolyzed silane agent (Ceramic Primer, 3M ESPE, St Paul, USA) was applied; for the self-etching cement (Panavia F, Kuraray), a silane agent containing 10-MDP monomers (Clearfil Porcelain Bond Activator, Kuraray, Japan) was mixed with a self-etching primer (Clearfil SE Bond, Primer, Kuraray CO, Osaka, Japan) for silane hydrolization prior its use. Silane agents were applied on the ceramic disks, left undisturbed for 60 s and gently blown-dried for 5 s.

2.2. Adhesive protocols

Application of the adhesive systems on the ceramic surfaces were performed according to the experimental groups: a thin layer of the different bonding resins were actively applied with disposable microbrushes for 5 s performing circular rubbing movements on the etched ceramic surface, excess material was removed. When the bonding resins were combined, they were applied separately. When ED Primer was used, Liquid A and Liquid B were previously mixed following manufacturer's instructions and then applied actively and blow-dried after 30 s for solvent evaporation. The ceramic blocks were fixed in a device specially developed to allow light curing through the ceramic in order to simulate a more realistic clinical situation. A silicon mold containing four cylindrical orifices with internal diameter of 2 mm, 1 mm in height, 4 mm apart from each other was securely placed on the ceramic surface. Resin cements were mixed following manufactures' instructions and carefully inserted inside the cylindrical orifices using a precision-applicator syringe (Sistema Centrix, Nova DFL, RJ, Brazil). After 3 min, photo-curing was performed for 60 s using a quartz–tungsten halogen unit (LC Demetron, Kerr Orange, CA, USA) with a curved light guide (11 mm tip). The irradiance was constantly monitored (minimum of 600 mW/cm^2) throughout the whole experiment. Four resin cement composite cylinders were produced 4 mm apart from each other on each ceramic block. Afterwards, the mold was carefully removed to expose the resin cement cylinders.

2.3. Micro-shear bond strength (μSBS) test and fracture pattern analysis

The specimens were submitted to μSBS test on a mechanical testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil). A stainless steel wire (0.35 mm diameter) was wound around the resin cement cylinder at the ceramic–resin cement interface. A shear load, using a stainless steel wire measuring 0.35 mm in diameter, was applied to the base of each cylinder at a crosshead speed of 0.5 mm/min until failure and converted to microshear bond strength (MPa) by dividing the load by the surface area of the each cylinder. The average value of the four bonded cylinders for each ceramic block was considered as the corresponding value for each specimen. The fractured specimens were mounted on aluminum stubs, coated with gold (SCD 050, Baltec, Vaduz, Liechtenstein) and evaluated by scanning electron microscopy (JSM-5600LV, JEOL, Tokyo, Japan). The failure modes were classified as: type I-adhesive failure between resin cement and ceramic; type II-cohesive failure within the ceramic or resin cement; and type III-mixed failures involving type I and type II.

2.4. Degree of conversion analysis

Fifty ceramic blocks (12 mm diameter, 2 mm height, shade A2) from each ceramic type were prepared for DC. Resin cements were mixed and applied into a disk-shaped Teflon mold (0.5 mm height and 5 mm in diameter). The different adhesive protocols were applied on a Mylar strip and positioned on top of the uncured resin cements to allow interaction between bonding resins and resin cement. Ceramic blocks were placed on top of the Mylar strip and after 3 min, light curing was performed for 60 s (LC Demetron, Kerr Orange, CA, USA) through the ceramic, similarly to the bond procedures used in the μSBS test. The Mylar strip was used to inhibit micromechanical retention between the different bonding resins and the ceramic, allowing the ceramic block removal after resin cement setting. Five specimens were made for each group ($n=5$) and stored dry at 37 °C in absence of light for 24 h after photo-activation. After light curing, the ceramic blocks and the Mylar strips were removed and discarded and the cured resin cement was carefully removed from the mold. The bonding surface was polished with 1200-grit SiC papers (Buehler-Met II, Buheler, Germany) and a stereoscope (SMZ1000, Nikon, Japan) at 40× magnification was used to verify the complete removal of the adhesive layer in order to analyze the resin cement degree of conversion. DC was performed by Fourier Transform Infrared Spectroscopy (Spectrum 100 Optica; PerkinElmer, MA, USA) according to the baseline method (BLM) [25]. Briefly, BLM measures the vinyl groups (C=C) intensity ratio of aliphatic and aromatic groups by drawing linear baselines from points taken in the depressions adjacent to the specific peaks. Degree of conversion is determined by measuring the decrease of the C=C rationed before and after polymerization to an internal aromatic C=C standard. The specimens were placed on the ATR crystal surface and the infrared spectra were collected between 500 and 4000 cm^{-1} at 4 cm^{-1} spectral resolution. Degree of conversion (DC) was calculated by changes in C=C absorption peak ratios of aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) peaks in both uncured and cured states obtained from the infrared spectra according to the following equation:

$$DC\ (\%) = \left(1 - \frac{R^{(Cured)}}{R^{(Uncured)}}\right) \times 100$$

where "*R*" is the ratio of aliphatic and aromatic peak intensities at 1638 cm^{-1} and 1608 cm^{-1} in cured and uncured adhesives. One spectrum was collected form each sample ($n=5$). The average of three readings was considered to obtain the ratio of aliphatic/aromatic peaks for uncured resins cements. For control groups, DC of each resin cement was measured without the adhesive protocols.

2.5. Statistical analysis

Data from μSBST and DC were analyzed by two-way analysis of variance (two-way ANOVA) with main variables as "adhesive protocol" and "ceramic type". For DC analysis, adhesive protocols containing different resin cements were not compared due to their different mechanisms of polymerization. Post-hoc multiple comparisons were performed using Tukey test ($\alpha=0.05$).

3. Results

3.1. Micro-shear bond strength

Two-way ANOVA showed that the interaction of "adhesive protocol" and "ceramic" significantly affected the micro-shear bond strength ($p<0.0001$). Overall μSBST values and standard deviations for all adhesive protocols and ceramic system are summarized in Fig. 1.

For the lithium dissilicate reinforced ceramic (IPS e.MAX) no differences were observed between protocols containing the resin cement RelyX ARC when SB Adhesive and SB Adhesive/SB Catalyst were used ($p=0.6081$). The combination of RelyX ARC and SB

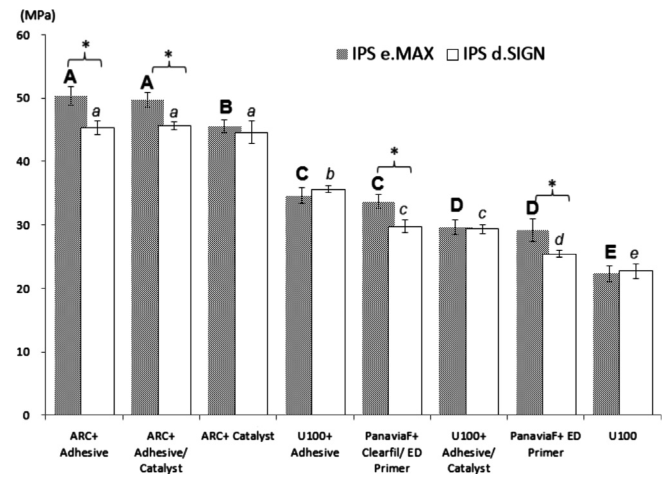

Fig. 1. Micro-shear bond strength values (MPa) and standard deviations for all groups. Values with different capital letters indicate significant differences according to Tukey test ($p < 0.05$) when analyzing different adhesive protocols for lithium reinforced ceramic (IPS e.MAX). Different lowercase letters indicate significant differences according to Tukey test ($p < 0.05$) when analyzing different adhesive protocols for fluorapatite leucite ceramic (IPS d.SIGN). * Indicates significant differences according to Tukey test ($p < 0.05$) when analyzing the specific adhesive protocol for both ceramic systems.

Catalyst produced lower bond strength compared to RelyX ARC/SB Adhesive ($p = 0.0286$). Nevertheless, the adhesive protocols containing RelyX ARC presented the highest µSBST values for IPS e.MAX. When Panavia F was associated with ED Primer and Clearfil higher bond strengths were obtained compared to ED Primer alone ($p = 0.0311$). Bonding protocols containing Panavia F produced intermediate bond strength values when bonded to IPS e.MAX. RelyX U100/SB Adhesive/SB Catalyst ($p = 0.0007$) and RelyX U100/SB Catalyst ($p = 0.0034$) produced higher µSBST values compared to RelyX U100. RelyX U100/SB Adhesive produced higher bond strengths compared to and RelyX U100/SB Catalyst/SB Adhesive ($p = 0.0389$).

For the fluorapatite leucite glass-ceramic (IPS d.SIGN), the different adhesive protocols containing RelyX ARC presented once again the highest values; no differences in bond strength were observed between protocols containing RelyX ARC. RelyX U100 used according to the manufacturer's instructions produced the lowest values; however bond strength was significantly improved when RelyX U100 was associated with SB Adhesive ($p = 0.0011$) and SB Adhesive/SB Catalyst ($p = 0.0072$). RelyX U100/SB Adhesive/SB Catalyst produced lower bond strengths compared to RelyX U100/SB Adhesive ($p = 0.0345$). Bond strengths of RelyX U100/SB Adhesive and Panavia F/Clearfil/ED Primer were not statically different ($p = 0.0871$). Adhesive protocols containing Panavia F produced higher bond strengths when associated with Clearfil SE Bond compared to ED Primer alone ($p = 0.0425$), resulting in intermediate results. RelyX U100/SB Adhesive/SB Catalyst produced higher bond strength values compared to Panavia F/ED primer ($p = 0.0145$).

Bond strengths were greater for the lithium reinforced ceramic (IPS e.MAX) compared to the fluorapatite leucite ceramic (IPS d.SIGN) when Panavia F/ED primer ($p = 0.0376$), Panavia F/ED primer/Clearfil ($p = 0.0299$), RelyX ARC/SB Adhesive ($p = 0.0287$) and RelyX ARC/SB Adhesive/SB Catalyst ($p = 0.0416$) were used. The remaining adhesive protocols did not present statistically different results between IPS d.SIGN and IPS e.MAX ceramic systems.

3.2. *Fracture patterns*

Overall fracture patterns are summarized as percentages in Fig. 2. For all groups, the most predominant type of failure was

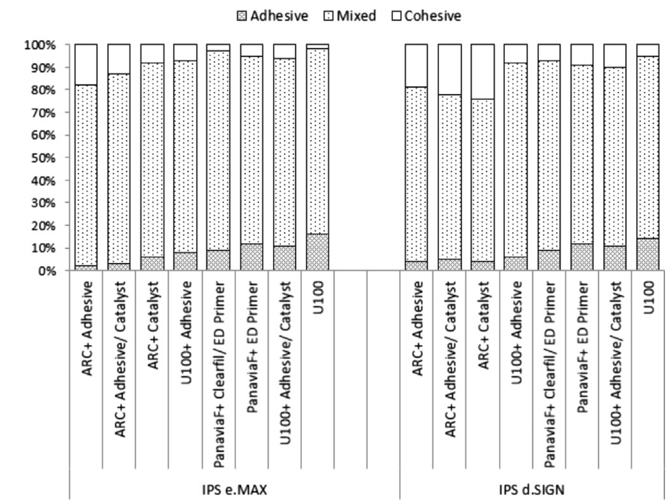

Fig. 2. Failure mode distribution for all groups.

mixed for both ceramics. All groups presented cohesive failures along the ceramic interface, which occurred more frequently on RelyX ARC adhesive protocols especially for IPS d.SIGN. Adhesive failures were observed in all groups for both ceramics. Reduced specimens with adhesive failures were observed when RelyX ARC adhesive protocols were used, however RelyX U100 bonded-specimens presented a higher occurrence of adhesive failures.

3.3. *Degree of conversion*

Two-way ANOVA showed that the interaction of "adhesive protocol" and "ceramic system" significantly affected resin cement degree of conversion for adhesive protocols containing Panavia F ($p < 0.0001$). Adhesive protocols containing RelyX ARC were significantly affected by "ceramic type" ($p < 0.0001$). Overall values of degree of conversion are summarized in Fig. 3.

Adhesive protocols containing RelyX ARC produced higher degree of conversion values for IPS e.MAX. No differences between RelyX ARC adhesive protocols occurred among the different bonding protocols for each ceramic system. Panavia F/ED primer ($p = 0.0425$) and Panavia F/ED Primer/Clearfil ($p = 0.0389$) produced higher resin cement conversion values when light curing was performed through IPS e.MAX. The adhesive protocol containing Panavia F that combined ED Primer/Clearfil SE Bond compared to ED Primer alone presented no statistical differences ($p = 0.0999$). Higher resin cement conversion occurred when Panavia F/ED primer and Panavia F/ED Primer/Clearfil used compared to Panavia F by itself irrespective of the ceramic type. Adhesive protocols containing RelyX U100 produced degree of conversions values that were not statistically different between IPS e.MAX and IPS d.SIGN; for each ceramic system, adhesive protocols containing RelyX U100 produced conversion values that were not statistically different.

4. Discussion

Since proper adhesive bonding is a determinant factor regarding long-term success of several ceramic restorative procedures in dentistry [2,3,5–7,26–29] different resin cements were evaluated in an attempt to obtain further information about their behavior when distinct adhesive protocols and ceramic types are employed. Two-way ANOVA revealed that the adhesive protocols tested ($p < 0.001$) and the different ceramic systems influenced ceramic bond strength. Therefore, the null hypothesis was rejected

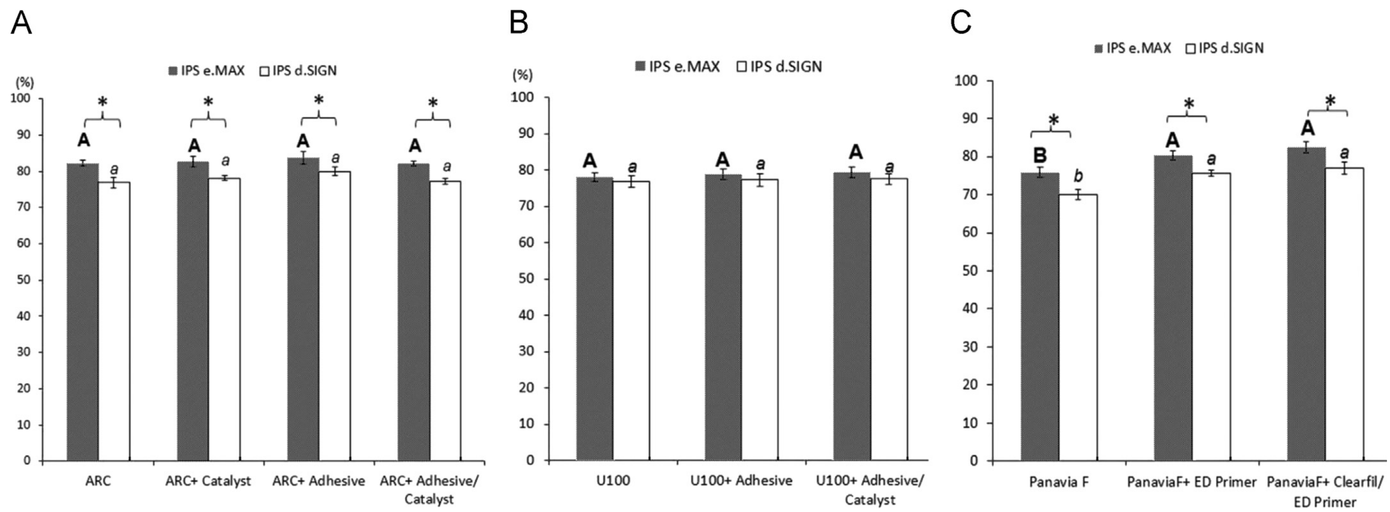

Fig. 3. Values for the degree of conversion (%) and standard deviations of resin cements with different adhesive protocols: (A) resin cement RelyX ARC; (B) resin cement U100; and (C) resin cement Panavia F. Different uppercase letters indicate significant differences according to Tukey test ($p < 0.05$) when analyzing different adhesive protocols for IPS e.MAX for (A) –(C). Different lowercase letters indicate significant differences according to Tukey test ($p < 0.05$) when analyzing different adhesive protocols for IPS d.SIGN for (A)–(C) respectively. * Indicates significant differences according to Tukey test ($p < 0.05$) when analyzing individual adhesive protocols for both ceramic systems for (A)–(C) respectively.

for the tested adhesive protocols significantly affected resin cement effectiveness in bonding to the different glass ceramics.

Shear stresses play an important role on bonding failures of restorative materials in the oral cavity [30]. According to finite element analysis studies (FEA), the most critical zone for failure is located in the cement region [31], corroborating the importance of adequate bonding and high conversion rates of resin cements to bonded to ceramics. The tested resin cements and adhesive resins present distinct chemical compositions with different monomers and curing systems which affect the resin cement mechanical properties [32,33]. Monomer type may also influence bond strength performance due to chemical interaction between specific monomer functional groups and the bonding substrate and chemical initiators may benefit polymerization in the absence of light. Besides Bis-GMA, TEGDMA and UDMA, monomers commonly found in conventional resin cements, the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is found in Panavia F, ED Primer and Clearfill SE, promoting chemical bonding to metal oxides present in some ceramic systems. Nevertheless, the tested adhesive protocols containing RelyX ARC, a MDP-free resin cement, produced higher bond strengths than Panavia F for both ceramics. Such outcome could be directly related to the fact that RelyX ARC presents higher flexural strength compared with Panavia F [32]. Therefore, differences in their mechanical properties most likely affected bond strength, for resin cements with weaker cohesive strengths are expected to fail at lower loads. The most predominant fracture pattern for all groups was mixed, involving a combination of adhesive and cohesive failures of ceramic or resin cement. The occurrence of mixed failures may be explained by the use of a micro-shear test that produces a more heterogeneous stress distribution reducing the number of pure adhesive failures.

In addition to adequate mechanical properties, proper ceramic micromechanical retention associated with proper monomer conversion are important factors to determine bond strength. In this sense, the tested adhesive protocols containing different adhesive resins played an important role on the ceramic bond strengths. For instance, RelyX U100 provided the lowest bond strength to both ceramics when used by itself following manufacturer's instructions; however, when RelyX U100 was associated with SB Adhesive or SB Adhesive/SB Catalyst higher bond strength values were obtained irrespective of ceramic type. In order to establish reliable ceramic bonding, adequate infiltration of monomers into the ceramic microporosities and proper mechanical properties of the luting agents must occur. According to this approach, the wetting ability of the cement is critical to provide high bond strength values.

The application of unfilled adhesive resins that are able to penetrate into the etched ceramic irregularities improves the mechanical properties of glass-ceramics [34], Thereby, application of low viscosity adhesives promotes better wetting and consequently superior interlocking within the ceramic irregularities [35], improving bond strength. Even though the application of low viscosity adhesive resins increased bond strength of RelyX U100, the combination of SB Adhesive/SB Catalyst produced lower bond strength values compared to SB Adhesive application by itself when RelyX U100 was used for both ceramic systems. A possible explanation for SB Catalyst bond strength reduction is the fact that the SB Catalyst contains benzoyl peroxide in it composition, which is responsible for the chemical start-up reaction due to the presence of specific tertiary amines. RelyX U100 requires a distinct initiator system so SB Catalyst application may have hampered de contact between the resin cement and SB Adhesive, reducing monomer interaction and the bond strength values compared to the RelyX ARC/SB Adhesive group.

For both ceramic systems, protocols containing RelyX ARC produced the highest bond strengths. The mechanical properties of the adhesive resin interfused in the ceramic microporosities and the resin cement plays an important role on ceramic bonding. The fact that the light reaching the luting cement and the adhesive resin is attenuated by the absorbing characteristics of the indirect restorative material [19,25,36] might have influenced bond strengths. Even though no differences in bond strengths were observed among RelyX ARC adhesive protocols bonded to the feldsphatic ceramic, for the lithium reinforced ceramic RelyX ARC/SB Catalyst produced lower bond strengths compared to protocols containing SB adhesive. Moreover, when the lithium reinforced ceramic was luted with RelyX ARC protocols containing SB Adhesive, a light curing resin, higher bond strengths were obtained compared to the felpsphatic ceramic. This might be explained by the fact that the degree of light attenuation is primarily dependent on the characteristics of the restorative material, such as crystalline structure, thickness, and opacity [19]. Combination of scattering, reflecting, and absorbing properties at the outer surface of the intervening material reduced the ability of light to reach the adhesive resin and resin cement and underneath the different tested ceramics. Therefore, different light transmittance through the tested ceramic systems might explain higher conversion values for RelyX ARC when light curing was performed through the lithium reinforced ceramic.

Higher bond strength values were obtained when the lithium reinforced ceramic was treated with SB Adhesive, an adhesive resin containing photo-initiators. Conversely, SB Catalyst is an adhesive resin lacking photo-initiators and containing benzoyl peroxide, which relies on tertiary amines present in RelyX ARC to produce free radicals to induce resin auto-polymerization. We speculate that such increase in bond strength was related to improved polymerization of the adhesive resin, for no significant differences in resin cement degree of conversion of adhesive protocols containing RelyX ARC occurred. It has been well documented that the light-curing mechanism contributes to higher bond strengths compared to chemical-curing most likely due to increased degree of cure [37–39]. As consequence, the application of a chemically cured adhesive on the etched ceramic surfaces might have produced a bonding interface (adhesive resin/ceramic) with lower mechanical properties compared to the light-cured adhesive protocols containing SB Adhesive. Maybe if thicker ceramic specimens were used, the outcome of using adhesive resins with photo-initiators or chemically cured could have been different. More studies should be performed to establish the influence of ceramic thickness on the bond strength of dual-cure resin cements.

In order to reduce the effect of light attenuation caused by indirect restorations on bond effectiveness [36], the tested self-etch resin cement Panavia F presents in its chemical composition sodium benzenesulfinate and the ED Primer solution contains sodium aromatic sulfinate to ensure that the polymerization reaction of the resin cement occurs without light exposure [40]. Sodium benzenesulfinate also has the ability to reduce the adverse effect of low pH on monomer conversion of chemically-cured or dual-cured resins [41]. In the present study, higher self-etching cement conversion values were obtained for both ceramics when ED Primer (pH 3.0) was used. The use of Clearfil SE (pH 2.0) along with Panavia F and ED Primer did not reduce resin cement conversion. Even though resin cement polymerization inhibition can occur in the presence of acidic monomers, the presence of sodium sulfinates in Panavia F and ED primer might have reduced the influence of Clearfil SE lower pH on resin cement conversion [41]. Regarding bond strength values, the use of a filled adhesive system might explain the higher bond strengths obtained for both ceramics systems. The presence of silica colloidal filler particles in Clerafil SE certainly improved the mechanical properties of the adhesive layer resulting in higher bond strength values compared to the unfilled adhesive ED Primer [42]. The lower bond strength values obtained for adhesive protocols containing Panavia F compared to protocols containing RelyX ARC and RelyX U100 associated with SB adhesive can also be related to the different silane agents used [43]. Whereas Ceramic Primer is fully hydrolyzed in the bottle and ready for application, the Porcelain Bond Activator must be activated with Clearfil SE Primer before application, which introduces a higher likelihood of operator variability and incomplete hydrolysis [44]. Therefore, the lower bond strength with Porcelain Bond Activator could in part be due to an incomplete hydrolysis in the activation step. In addition, considering that aging significantly affects bond strengths, future studies could be performed to assess the capacity of different ceramic adhesive protocols to withstand degradation over time, checking if the obtained outcomes would remain stable after aging.

5. Conclusions

Based on the findings of this study, it can be concluded that ceramic type and the selection of adhesive resins to be used with resin cements should be carefully considered in order to improve bonding effectiveness for glass ceramic bonding. While the conversion degree of the self-adhesive is not influenced by the tested ceramics and adhesive protocols, the conventional and self-etch cement present higher conversion values for the lithium disilicate glass-ceramic irrespective of adhesive protocol. Consequently, the use of adhesive resins containing photo-initiators in their composition produces higher bond strengths for the lithium disilicate glass-ceramic compared to the fluorapatite leucite glass-ceramic. Application of low viscosity adhesive resins on etched glass ceramic surface should be performed to improve bonding effectiveness of the tested self-adhesive cement. The use of a filled adhesive resin in conjunction with adhesive resins containing sodium aromatic sulfinates increases bond strength of the tested MDP self-etch resin cement. The conventional resin cement produces higher bond strength values for both ceramics compared to the tested self-etch and self-adhesive cement.

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