SHORT COMMUNICATION

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Bonding of CAD/CAM lithium disilicate restorations with regular and flowable composite resin with and without wetting resin

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Abstract

Objectives: Assess the effect of applying a low viscosity resin before placing ceramic restoration with two different luting agent.

Materials and methods: Twenty slices from IPS e.max CAD (Ivoclar Vivadent) were fired and assigned to 5 treatment surface groups; E: hydrofluoric acid (HF); E/S: HF-etching + silane (20 s); E/S+: HF-etching + silane (60 s); S: silane (20 s); S+: silane (60 s). Before building the cylinder with regular or flowable composite resin for the SBS test, half of each treatment surface group received adhesive (Optibond FL bottle #2), and the other half no adhesive. The SBS test was performed after 24 h. Statistical analysis was performed using multifactorial analyses of variance Two-way ANOVA. The Tukey HSD test was used to test the average values of all groups. The significance level adopted in all analyses was ≤ 0.05 ($p < 0.05$).

Results: Groups E/S and E/S+, with flowable + adhesive and E/S with regular composite resin + adhesive, showed the highest SBS values (22.05, 18.91 and 21.06 MPa respectively), followed by group E with or without adhesive, for both luting agent. The lowest bond strength was observed when the silane was applied alone, independent of the adhesive coat and luting agent (0.65–5.55 MPa). When the adhesive was omitted, flowable presented high bond strength compared to composite resin in groups ES and ES+.

Conclusions: The low viscosity resin in the etched and silanized ceramic surface is important to obtain high SBS values.

Keywords: Adhesion, Silane, Lithium disilicate

Introduction

A reliable bonding to ceramic restoration can be obtained by the association of factors as well as ceramic topography and chemical adhesion [1–3]. Regarding the surface characteristic, the silica-based ceramic can be easily modified by the hydrofluoric-acid etching (HF) which is able to create micro-mechanical retention by the selective dissolution of the glassy phase. 1. The further step is the silanization that takes care of the chemical adhesion between two different components (ceramic and resin) that need to be linked [2–5]. When silane agent coupling is applied, multiple layers are forming in the ceramic surface, among these layers, the physisorbed and chemisorbed regions consists in loosely bound oligomers thus that can be easily hydrolyzed and affect negatively the

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restoration durability. Therefore, it was suggested to remove these outermost layers by hot air drying and rinsing with hot water [6–10]. The luting step is followed by the direct application of the resin cement in the inner etched and silanated ceramic surface. Some clinicians may omit using the adhesive resin to wet the surfaces. However, depending on the viscosity, the luting agent may not be able to fill in all the micrometric spaces (micro-retentions) created by the HF-etching. The aim of applying a low-viscosity material is to create an interlocking that affects the resin-ceramic bond [11, 12], but these effects still require investigation. Another parameter is the choice of the luting agent, which may influence not only the bond strength but also vary in the degradation process [13, 14]. Regular restorative composite resin used as a luting agent can present some advantages over resin cement as well as degradation resistance and color stability [15–24] and can also compensate for little cracks or imperfections in the margins [24]. However, the viscosity of the composite resin is higher compared to resin cement, because of the higher filler content, which in turn might influence the ability to penetrated the microretentions of the etched ceramic. The purpose of this study was to assess the effect of wetting with adhesive resin before placing ceramic restorations with different luting agent, regular versus flowable composite resins. The first null hypothesis tested was that various treatments (etching, silane, wetting resin) do not affect the bond strength. The second null hypothesis was that the luting agent (restorative versus flowable) does not make a difference.

Materials and methods

Specimen preparation

Twenty slices (14 × 16 × 2 mm) of IPS e.max CAD (EMAX; Ivoclar Vivadent, AG, Schann, Liechtenstein) were obtained by sectioning CAD/CAM blocks with a slow-speed diamond water-cooled saw (Isomet, Buehler, Lake Bluff, IL, USA). The slices were crystalized in a ceramic furnace (Austromat 624; DEKEMA Dental-Keramiköfen GmgH, Freilassing, Germany) following the manufacturer's instructions. All the slices were embedded in an acrylic resin positioning base (Palapress; Heraeus Kulzer, Hanau, Germany) and polished with 400- and 600-grit silicon carbide paper under water cooling. The surfaces of all slices were then cleaned in an ultrasonic bath in distilled water during 5 min.

Surface treatments

Five surface conditioning methods were applied: E—5% HF-etching (IPS Ceramic Etching Gel, Ivoclar Vivadent, AG, Schann, Liechtenstein) for 20 s, rinsing with air/water spray for 60 s followed by post-etching cleaning in ultrasonic bath in distilled water for 2 min; E/S—5% HF-etching as above, silane (Silane, Ultradent, South Jordan, Utah, USA) applied for 20 s and air dried for 20 s, hot air dried at 60 °C for 20 s (with hair dryer at 10 cm-distance); E/S+—5% HF-etching as above, silane applied for 60 s, air dried for 20 s and hot air dried 60 °C for 20 s rinsed with boiling water for 15 s and hot air dried for another 20 s; S—no etching, silane for 20 s followed by air dried for 20 s, hot air dried at 60 °C for 20 s or S+—no etching, silane applied for 60 s, air dried for 20 s and hot air dried 60 °C for 20 s, rinsed with boiling water for 15 s and another hot air dried for 20 s.

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SBS test

Specimens were then placed in a positioning device and a Teflon mold (Ultradent Jig, South Jordan, Utah, USA) containing a bonding area 0.04448 cm^2 was used. Half of each group received adhesive resin coating (Optibond FL bottle #2, Kerr Corp. Orange, CA, USA) and for the other half no adhesive was applied. The cylinders for SBS test were built either with regular composite resin (Z100, 3M ESPE, Saint Paul, MN, USA) or flowable composite resin Filtek Supreme Ultra, 3M ESPE, Saint Paul, MN, USA) (Tables 1, 2) inserted into the Teflon mold in two increments of approximately 1.8 mm each, using a small condensing spatula and light polymerized for 20 s (Valo, Ultradent, South Jordan, Utah, USA) at 1000 mW/cm^2. Six resin cylinders (diameter: 2.4 mm) were built onto each ceramic slice (length: 15 mm, width: 4 mm, thickness: 2 mm), 3.5 mm apart, resulting in 12 specimens per group. The test was performed after 24 h of storage in distilled water. All samples were submitted to SBS testing (Shear Bond Tester; Bisco Inc., Schaumburg, IL, USA) with a ramp load of 43.8-kg-force/min. The failure mode was assessed by macro-photography (Nikon D60 and Sigma Lens 105 mm and a teleconverter 2×, Vivitar).

Statistical analysis

The mean bond strength and standard deviation were calculated for all groups. Statistical analysis was performed using multifactorial analyses of variance Two-way ANOVA. The Tukey HSD test was used to test the average values of all groups. The significance level adopted in all analyses was ≤ 0.05 ($p < 0.05$) (Table 3).

Results

All SBS means and standard deviation are presented in Table 4. The comparison between groups where restorative composite resin was used as a luting agent showed higher SBS values for ES with adhesive, followed by E = ES+ with and without adhesive. No statistical differences were found between E, ES and ES+ when the adhesive was omitted. However not wetting with adhesive showed a significant decrease in bond strength for ES and ES+, while for E the values were not statistically different. When flowable composite resin was used as a luting agent the highest SBS value were obtained with ES

Table 1 Materials tested, brand name, manufacturer and composition

Materials	Brand name	Manufacturer	Composition
HF	IPS ceramic etching gel	Ivoclar Vivadent	Hydrofluoric acid 2.5 to < 7%
Silane	Silane	Ultradent	Methacryloxy propyl trimethoxy silane; isopropyl alcohol
Ceramic	IPS e.max CAD	Ivoclar Vivadent	SiO_2; Li_2O; K_2O; P_2O_5; ZrO_2; ZnO; Al_2O_3; MgO; Colouring oxides
Adhesive	Optibond FL (bottle #2)	Kerr Corp.	2-Hydroxyethyl methacrylate; 3-trimethoxysilylpropyl methacrylate; 2-hydroxy-1,3-propanediyl bismethacrylate; alkali fluorosilicates(Na)
Regular composite resin	Z100	3M ESPE	Bis-GMA and TEG-DMA; inorganic matrix; zirconia/silica
Flowable composite resin	Filtek supreme ultra	3M ESPE	Bis-GMA, UDMA, TEGDMA, and bis-EMA; inorganic matrix: zirconia/silica

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Table 2 Groups and materials applied

	E		E/S		E/S+		S		S+	
Regular composite resin	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application
Flowable composite resin	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application	Adhesive application before luting	No-adhesive application

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Table 3 Statistical datas

Source	DF	SS	MS	F	PROB > F	Omega squared
Among treatments	9	9877.959	1097.551	94.160	0.000	0.734
Among composite resin and flowable	1	242.225	242.225	20.781	0.000	0.017
Interaction	9	626.287	69.587	5.970	0.000	0.039
Within groups	220	2564.362	11.656			
Total	239	13,310.833	55.694			

Table 4 Different lowercase letter in the same column are statistically different

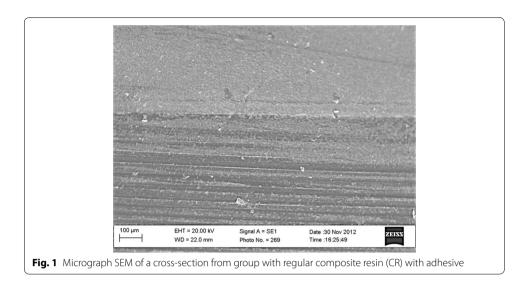| Treatments	Regular composite resin	Flowable composite resin
	Mean (SD) (n = 12)	Mean (SD) (n = 12)
E	15.15 (3.8)bA	13.13 (2.4)bA
E/S	21.06 (4.4)aB	18.91 (3.6)aB
E/S+	15.93 (4.0)bC	22.05 (3.9)aD
S	4.32 (5.1)dE	4.85 (2.9)dE
S+	3.92 (2.5)dF	5.55 (3.3)dF
E (no adhesive)	11.79 (5.2)bcG	11.04 (6.2)bG
E/S (no adhesive)	10.88 (3.2)bcH	17.22 (1.1)bcI
E/S+ (no adhesive)	10.13 (1.0)cJ	17.01 (1.4)bcK
S (no adhesive)	0.65 (2.4)dL	3.31 (1.8)dL
S+ (no adhesive)	1.53 (0.8)dM	2.41 (1.7)dM

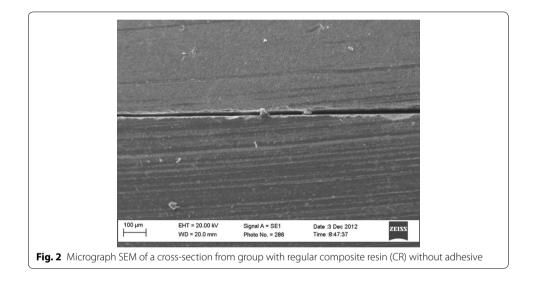Different uppercase letter in the same line are statistically different

E hydrofluoric acid (HF), *E/S* HF-etching + silane (20 s), *E/S+* HF-etching + silane (60 s), *S* silane (20 s), *S+* silane (60 s)

and ES+ with adhesive, followed by E. When the adhesive layer was not applied E, ES and ES+ showed no statistical difference. The lowest SBS values were always obtained with S and S+ regardless of the use of adhesive resin or type of luting agent. The comparison between the luting agents, revealed that for groups ES+ with adhesive, ES and ES+ without adhesive flowable composite resin yielded statistically higher results than restorative composite resin. When the adhesive was used, it was shown a continue adhesive gap, that was not clear in the groups without adhesive (Figs. 1, 2, 3, 4). The failure mode was always adhesive, leaving a clean ceramic surface (Fig. 5).
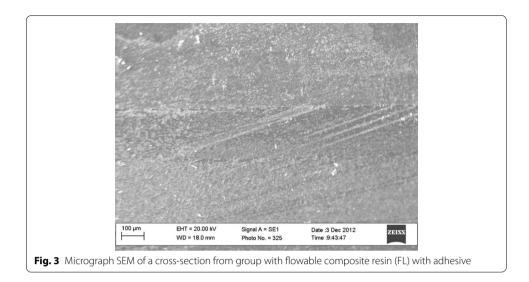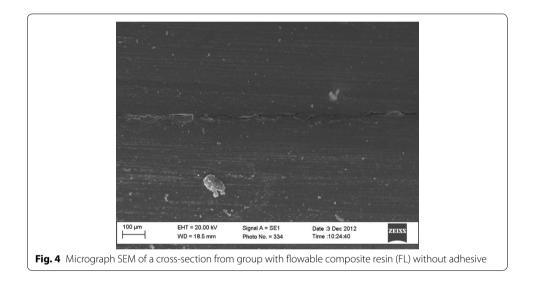
Discussion

The first null-hypothesis was rejected since the surface treatments affected the SBS values. The second null-hypothesis was rejected in part, as the luting agent had a significant influence on the SBS in groups ES+ with adhesive, ES and ES+ without adhesive. Etching alone and use of adhesive resin promoted similar bond strength, which can be explained by the micromechanical interlocking created by the luting agent or adhesive coat [25]. However, this work reaffirms the importance of associating the mechanical and chemical treatments to reach high bond strength as illustrated by E/S groups and application of the adhesive resin over the silanated surface to provides better infiltration

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Fig. 1 Micrograph SEM of a cross-section from group with regular composite resin (CR) with adhesive

Fig. 2 Micrograph SEM of a cross-section from group with regular composite resin (CR) without adhesive

into the irregularities of the etched surfaces [11]. On the other hand, Passos et al. [12] did not find better results when applying the adhesive coat before the luting agent because they used a dual-cure resin cement which is flowable by nature. When using more viscous luting agents, however, the low viscosity resin seems to be indispensable to wet the etched and silanized ceramic surface, infiltrate in the micrometric spaces and ensure the ceramic bond strength by micromechanical interlocking [11, 25]. The omission of this step probably generated an insufficient penetration of the luting agent in the micro porosity and consequently decreases the ceramic mechanical strength [26]. These empty spaces can be an area of stress concentration since the adhesive layer work like an damper [27]. The lower bond strength obtained by silane alone, regardless of the use of adhesive resin and type of luting agent, can be explained by the absence of micromechanical retention in the ceramic surface, showing that the chemical step alone is not sufficient to obtain high bond strength. Regarding the luting agent, the similarity in bond

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Fig. 3 Micrograph SEM of a cross-section from group with flowable composite resin (FL) with adhesive

Fig. 4 Micrograph SEM of a cross-section from group with flowable composite resin (FL) without adhesive
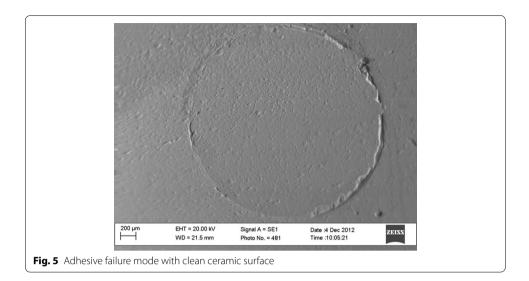
strength between restorative and flowable composite resin could be explained by the fact that in this research the authors did not used pressure during the luting agent application. Seating pressure during luting could promote a better penetration of the resin into the ceramic retention. This study is restricted to some materials and did not evaluated the longevity of the SBS, thus more studies are suggested to compare the luting agent degradation in the course of time.

Conclusion

Wetting with adhesive resin increases the resin-ceramic SBS to the etched and silanated ceramic surface. This is particularly important when using a restorative composite resin as a luting agent. Omission of wetting resin will result in better SBS to the etched and silanated ceramic surface when using a flowable luting agent compared to a restorative composite resin.

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Fig. 5 Adhesive failure mode with clean ceramic surface

Authors' contributions
BG—Contribution to the paper: wrote the paper, performed the experiments in partial fulfillment of requirements for a degree. CAO—Contribution to the paper: Wrote the paper, performed the experiments in partial fulfillment of requirements for a degree. GM—Contribution to the paper: wrote the paper. MHP—Contribution to the paper: wrote the paper. MP—Contribution to the paper: idea, wrote the paper. All authors read and approved the final manuscript.

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Competing interests
The authors declare that they have no competing interests.

Availability of data and materials
Not applicable.

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