Effect of Air–Particle Abrasion of Dentin Surface on Shear Bond Strength of Lithium Disilicate to Dentin Using Three Different Cements

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Abstract

This study aims to evaluate the influence of the air abrasion of dentin on the shear bond strength of lithium disilicate using three different types of luting cements.

Sixty cylindrical specimens were milled from lithium disilicate CAD/CAM blocks (IPSe.max CAD). Sixty sound human maxillary premolar teeth were decoronated to the level of peripheral dentin, then randomly divided into three groups according to the type of luting cement used for the cementation of the lithium disilicate specimens (n = 20); Group A: Glass ionomer cement (Riva Self-Cure); Group B: Adhesive resin cement (Rely X Ultimate); Group C: Self-adhesive resin cement (Rely X U200). Each group was then further subdivided into two subgroups (n=10); Subgroups AI, BI, and CI, in which lithium disilicate specimens were cemented directly to dentin; Subgroups AII, BII, and CII, in which dentin surface was air abraded prior to cementation of lithium disilicate specimens.

A computerized universal testing machine was used to measure the shear bond strength. A digital microscope was used to study the failure mode. SEM was used to analyze the cement-dentin interface of the de-bonded samples. The data were analyzed statistically using One-way ANOVA test and independent sample t-test at the level of significance of (0.05).

Air abrasion of dentin improved the shear bond strength of lithium disilicate to dentin with all three types of cement.

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Introduction

The high demand for aesthetic dentistry has increased the popularity of all-ceramic restorations. In this field, lithium disilicate restorations have been widely used owing to their superior esthetic and adhesive properties [1,2]. These restorations can be cemented using conventional cements, or by adhesive cementation using adhesive or self-adhesive resin cements [3]. Currently, the most commonly used luting cements are glass ionomer and resin cements. Glass ionomer cements (GIC) have the ability of adhesion to metal and tooth structure with the advantages of fluoride release and recharging capability, significant for caries prevention. On the other hand, resin cements have become a more reliable choice for luting indirect restorations owing to their optimal adhesive bonding to enamel, dentin, and ceramic restorations [4].

The bond strength of the luting cement to the restoration and tooth substrate is crucial for the longevity of indirect restorations [5,6]. Generally, adhesion to dentin is more challenging than bonding to enamel and ceramics. The rationale behind this is the intrinsic features of dentin including the organic content, tubular structure, fluid flow, and smear layer. The micromechanical bond between dentin and resin cement is mainly achieved through the penetration and polymerization of resin monomers into the collagen fiber network, commonly known as the 'hybrid layer' [7].

Over the years, the 'etch-and-rinse' adhesive systems have been considered the gold standard protocol for dentin bonding due to the formation of resin tags and a true hybrid layer. Despite the high bond strength of adhesive

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cements, their implementation in clinical practice is technique-sensitive and time-consuming[8].

Alternatively, 'self-adhesive' resin cements were introduced to simplify the multi-step adhesive systems and minimize postoperative sensitivity. The acidic monomers in self-adhesive cements could demineralize and penetrate the tooth surface providing both chemical and micromechanical retention. However, it has been reported that the bonding performance of these cements with dentin is still inferior to that of the adhesive counterparts [9].

To overcome the abovementioned issues, researchers have explored alternative materials, adhesion protocols, and substrate pretreatment procedures that may optimize the level of dentin adhesion [10-12]. In this context, different chemical and mechanical pretreatments have been developed to enhance the surface roughness of dentin, increasing the contact area of bonding and optimizing the adhesion values [7]. One of these methods is the concept of tooth sandblasting with aluminum oxide particles, the so-called 'air abrasion', first introduced by Black in 1945 [13].

Currently, airborne particle abrasion has many applications in clinical practice including cavity preparation, pretreatment of ceramic restorations, and pretreatment of teeth before direct and indirect restorations [14]. Despite the limited data of clinical research, several *in vitro* studies have reported the positive impact of air abrasion on bond strength of adhesive bonding agents, whether self-etch or etch-and-rinse [7,15]. However, evidence regarding the effect of air abrasion of dentin surface on the bonding performance of dental cements is still deficient. Therefore, this study was conducted to find out the effect of air abrasion of dentin surface on the shear bond strength (SBS) of lithium disilicate using different types of luting cements. The null hypothesis has two folds, the first is that the type of luting cement doesn't affect the shear bond strength of lithium disilicate to dentin. The second is that air abrasion of dentin does not affect the shear bond strength of lithium disilicate.

Materials and methods

The chemical composition along with the manufacturing companies of the different materials used in this study are listed in Table 1.

Sample preparation

Preparation of lithium disilicate specimens

Sixty specimens (cylindrical-shaped, 5 mm diameter x 3 mm height) were milled from lithium disilicate CAD/CAM blocks (IPS e.max CAD All LT Cl4, Ivoclar, Vivadent Germany) using a custom-made milling and sectioning machine. The lithium disilicate block was fixed to the milling machine via its fitting pin. A diamond cutting disc mounted in a straight hand piece, that was attached to the movable part of the machine, was used to mill the block into a cylinder with a diameter of 5 mm under copious water cooling. The same disc was then used to cut the cylinder into discs with a 3 mm thickness.
Each specimen was then treated on its bonding surface with silicon carbide abrasive paper #grit 80 under water cooling using a grinding and polishing machine to provide standardized roughness. The cylinders were then cleaned in an ultrasonic unit with distilled water for 5 minutes to remove contaminants.

Preparation of teeth specimens

This work has been approved by the research ethics committee in May 2023 (no.808223). Sixty sound human maxillary first premolar teeth extracted for orthodontic purposes were collected. A digital microscope (Dino-Lite, Taiwan) was used to examine the selected teeth to be free from caries, cracks, and restorations. The teeth were washed with distilled water and then stored in normal saline at room temperature. For each tooth, to determine the area to be decoronated, a line was drawn 1.5 mm below the central groove. Subsequently, each tooth was fixed to the dental surveyor and immersed in cold cure acrylic along its longitudinal axis till the indicated line using a custom-made silicon mold with dimensions (1.5× 1.5× 2 cm). Each tooth was then decoronated to the level of the demarcated line using a diamond disc (0.3mm) fixed to a straight handpiece under copious water cooling. The cut dentin surface was then examined under a digital microscope for any remnants of enamel. To provide a standardized surface roughness of all specimens, a silicon carbide abrasive paper grit #220, and then grit # 500 were used to finish the cut surface of the tooth under water cooling using the same grinding and polishing machine that was used for finishing the lithium disilicate specimens.

Sample grouping

Teeth specimens were divided randomly into three main groups (n=20) according to the type of luting cement used for cementation of lithium disilicate specimens to dentin as follows:

Group A: Conventional glass ionomer cement (Riva Self-Cure, SDI, Victoria, Australia).

Group B: Adhesive resin cement (Rely X Ultimate, 3M ESPE, Germany).

Group C: Self-adhesive resin cement (Rely X U200, 3M ESPE, Germany).

Each group was subdivided into two subgroups of ten teeth each according to the use of sandblasting of teeth or not. Subgroups AI, BI, and CI: the dentin surface was sandblasted with 25 µm aluminum oxide prior to cementation of lithium disilicate specimens; Subgroups AII, BII, and CII: the lithium disilicate specimens were cemented directly on the dentin surface. The chemical composition along with the manufacturing companies of the different materials used in this study are listed in Table 1.

Cementation procedure

Surface treatment of Lithium disilicate specimens

For all groups, the bonded surface of each specimen was etched with hydrofluoric acid ≤ 5% using IPS Ceramic Etching Gel (Ivoclar, Vivadent) for 20 seconds according to the manufacturer's instructions, that was then rinsed off with water and air-dried. At that point, the specimens were thoroughly cleaned in an ultrasonic cleaner for 5 minutes. For subgroups BI, BII, CI, and CII, Single Bond Universal Adhesive (3M ESPE) was applied to the bonding surface of lithium disilicate specimens with a disposable micro brush for 20 seconds, then air-dried for 5 seconds and left without light curing.

Air abrasion of teeth

The dentin surface of teeth specimens of subgroups AII, BII &CII was sandblasted with 25 µm aluminum oxide powder using RONDOFLEX™ Plus 360 intra-oral sandblaster (Kavo Dental Excellence, Germany) with the aid of a modified dental surveyor. The tip of the device was directed perpendicular to the dentin surface at a distance of 2mm whereby a continuous stream of particles was directed to the treatment site for 10 seconds under 3.2 bar air pressure.

Surface treatment of the teeth

-Subgroups AI & AII (GIC cement was used): The dentin surface was conditioned with 37% phosphoric acid for 15 seconds then washed thoroughly with water and air-dried.

-Subgroups BI & BII (Rely X Ultimate adhesive resin cement was used): The dentin surface was etched with 37% phosphoric acid for 15 seconds, then universal adhesive (Single Bond Universal Adhesive, 3M ESPE) was applied with a micro brush and rubbed for 20 seconds, then air-dried for 5 seconds and light cured for 10 seconds using light curing pen (Eighteeth, China) with light intensity of 1000 Mw/cm2.

-Subgroups CI &CII (Rely X U200 self-adhesive resin cement was used): Neither etching nor bonding were used according to the manufacturer's instructions.

Cementation procedure

Cementation with GIC: The cement capsule was mixed using an amalgamator for 10 seconds according to the manufacturer's directions, the cement was then extruded out of the capsule on the bonding surface of lithium disilicate specimen using a capsule applicator. The cement-loaded lithium disilicate specimen was then seated on the dentin surface under a constant load of 5 Kg with the aid of a dental surveyor, the excess cement was removed with a dental explorer.

Cementation with adhesive and self-adhesive resin cement: The resin cement was loaded on the bonding surface of each lithium disilicate specimen, which was then seated on the tooth surface under a constant load of 5 Kg with the aid of a dental surveyor. A micro brush was used to remove the excess cement, then light cured for 20 seconds from four directions (buccal, lingual, mesial, and distal) using light curing pen. After cementation, all specimens were stored in distilled water for 24 hours.

Shear bond strength test

The shear bond strength was tested using a computer-controlled testing machine (LARYEE, China). A knife-edge chisel rod at a crosshead speed of 1 mm/min was used to apply the shear force at the bonding interface of each specimen. When the fracture occurred, the maximum failure load was recorded in Newton (N). The failure load (N) was divided by the bonding area (mm^2) to calculate the shear bond strength values in MPa.

$$Shear\ bond\ strength\ (SBS)\ value\ =\ failure\ load\ value\ (N)\ /\ area\ (mm2)$$

Statistical analysis

Statistical Package for Social Science

(SPSS version 16) was used to analyze the collected data. The normality of distribution of the data was assessed using the Shapiro–Wilk test. One-way ANOVA test was used for comparison among the different groups. Post hoc Tukey test was used to look for the differences between each two groups. Independent sample t-test was used to compare between the sandblasted and non-sandblasted subgroups with each cement type.

Failure mode analysis

The fractured specimens were examined under a digital microscope at a magnification of 50x. The Failure modes were classified as follows: Type I: Adhesive failure between the dentin and cement. Type II: Cohesive failure within the cement or tooth structure. Type III: Mixed failure.

SEM analysis

A randomly selected specimen from each subgroup was selected for examination under SEM to analyze the cement-tooth interface after de-bonding. The samples were sectioned bucco-lingually using a diamond disc fixed to a straight handpiece under copious water cooling. The selected specimens were polished to get a smooth surface followed by cleaning with 70% ethanol in an ultrasonic bath for 2 min. The surface of each sample was conditioned using 37% phosphoric acid for 15 seconds to remove cutting derbies and smear layer, followed by dentin decalcification for 30 seconds using 6% Normality hydrochloric acid and rinsed thoroughly with water. The sample was then deproteinized in 2.5% NaOCl for 10 minutes to dissolve the organic dentin matrix to enable examination of the interface. Finally, the samples were washed with 96% ethanol to remove water. After sample preparation, each surface was sputtered with gold nanoparticles, the coated samples were then viewed using 10keV beam voltage on a scanning electron microscope Axia™ ChemiSEM™ (Thermo Scientific Company, Netherlands) at different magnifications.

Results

The descriptive and inferential statistics are summarized in (Table 2). The mean values of SBS in (MPa) of the subgroups are shown in (Figure 1). The highest mean value of SBS was recorded in subgroup CII (Relyx U200 with sandblasting (16.495± 0.633 MPa), while, the lowest mean value (0.957± 0.057MPa) was recorded in subgroup AI (Riva Self-Cure without sandblasting).

Group	Sandblasted	NonSandblasted
GIC A	1.38±0.26 A[b*]	0.96±0.25 B[c]
Ultimate B	16.19±2.4 A[a]	11.27±3.88 B[b]
U200 C	17.98±2.98 [Aa]	14.78±3.34 B[a]

Table 2. Summary of the descriptive and inferential statistics.

*The uppercase letters demonstrate row differences, while lowercase letters demonstrate column differences (p > 0.05).

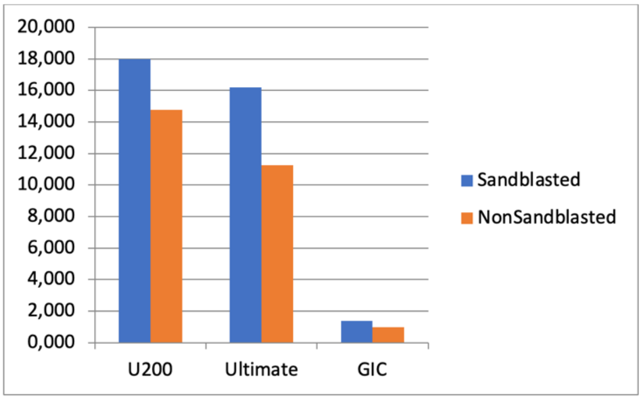

Figure 1. Bar chart showing the mean values of SBS of all subgroups.

One-way ANOVA was used to compare the SBS values among the subgroups with and without sandblasting and revealed a statistically significant difference *P* = (.000). Post Hoc Tukey test was used for multiple comparisons to look for the significance between subgroups. This test showed statistically significant differences in the SBS values between subgroups AI & BI *P* = (.000), AI &CI *P* = (.000), BI &CI *P* = (.000) AII & BII *P* = (.000), and between AII & CII, while no significant difference was found between subgroups BII & CII *P* = (.18). For comparison of the SBS between the subgroups with and without sandblasting, Independent sample t-test was used and showed a statistically significant difference between AI & AII *P* = (.002), BI & BII *P* = (.003), and CI & CII *P* = (.03).

The failure modes are shown in Table 3, the adhesive failure was observed in 40% of the samples in AI and 30% of the samples in AII. The majority of samples in BI and CI showed mixed failure, whereas the predominant failure mode was cohesive in the subgroups BII and CII. Concerning SEM analysis, the cement/dentin interface is observed in Figures 2, 3, and 4. The SEM image of the U200 self-adhesive resin cement/ dentin interface illustrated an irregular interface with no visible resin tags in the dentin. The SEM image of the U200 self-adhesive resin cement bonded to the air-abraded dentin interface showed a barely visible area of resin infiltration into the dentin and the aluminum crystals deposited in the dentin. The SEM image of the Ultimate adhesive resin cement bonded to the air-abraded dentin demonstrated the infiltration of resin into the dentin, the resin tags are visible and long.

Subgroup	Type I	Type II	Type III
AI	40%	20%	40%
AII	30%	30%	40%
BI	0	20%	80%
BII	0	70%	30%
CI	0	30%	70%
CII	0	80%	20%

Table 3. Failure modes distribution in the subgroups in %.

Discussion

The clinical success of indirect ceramic restoration strongly relies on its durable adhesion to the dentin. Hence, the proper selection of the luting agent and the application of the accurate cementation protocol are of paramount importance. The current study investigated the effect of air abrasion of dentin surface on the SBS of lithium disilicate cemented on dentin using different types of cement. Following the results of this study, there was a statistically significant difference in the SBS values among the three different types of cement. Thus, the first null hypothesis was rejected. The lowest SBS values were recorded with GIC, this could be attributed to the low mechanical properties and lack of the chemical bond of GIC with lithium disilicate as compared to the other resin cements tested in the study [16]. This result is in agreement with previous studies that recorded higher SBS values with the resin cement than with the GIC [17,18].

Regarding the bond strength of the resin cement, the current results revealed that the self-adhesive resin cement provided significantly higher SBS values compared to the adhesive resin cement. This could be attributed to the acidic functional monomers of RelyX U200 self-adhesive resin cement that could enhance the bonding to dentin through two main reactions; the first is a micromechanical bond by the methacrylate monomers which has the ability to demineralize dentin and facilitate the penetration of resin monomer of the cement into the dentin matrix. The second is a chemical bond by the reaction between the acidic group and the calcium of the hydroxyapatite crystals of tooth [19]. In agreement with the present study, Aguiar, et al. [20] have reported a significantly higher bond strength to dentin with RelyX Unicem self-adhesive resin cement compared to RelyX ARC adhesive resin cement. Similarly, D'Arcangelo, et al. [21] have revealed that the self-adhesive resin cement provided the highest bond strength to dentin compared to the self-etch and etch-and-rinse resin cements, the authors indicated that self-adhesive luting system can be suggested for the cementation of glass ceramic restorations.

Bond strength is affected by different parameters that are difficult to standardize. The heterogeneity in tooth structure and the lack of a standard testing protocol could be responsible for the high variability among bond strength values reported in the literature [22]. Many studies have reported that adhesive resin cement has a higher bond strength to the dentin than self-adhesive resin cement [23-25]. The authors have demonstrated that the low pH of the self-adhesive resin cement results in incomplete demineralization of the smear layer leading to an irregular cement–dentin interface with no authentic hybrid layer or resin tags [19]. This is also supported in our SEM analysis, the adhesive resin cement exhibited a regular interface with a homogeneous and continuous hybrid layer, while the self-adhesive resin cement showed a superficial interaction zone with the dentin.

Despite the low acidity of the self-adhesive resin cement, analysis with the photoelectron spectroscopy radiograph has shown that the acidic group could provide micromechanical retention with the dentin surface even without penetration of more than 1 mm [26]. Nevertheless, some authors have indicated that the penetration of the resin into the dentinal tubules has a minor influence on the bonding effectiveness [27].

Acid etching is a predictable clinical procedure that increases wettability and surface roughness allowing the penetration of adhesives and resin cements through the smear layer. However, unlike enamel, the heterogeneous structure and the high organic content of dentin reduced the positive impact of the acid etch on the surface energy and boding of dentin [28]. RelyX ULTIMATE is an adhesive resin cement that is used with either a self-etch or etch-and-rinse bonding protocol. In this study, Single Bond Universal adhesive was used with total etching of dentin. It has been shown that the acid etching of dentin could suppress the bonding effect of the universal adhesive systems, the authors have speculated that the strong acid demineralization of the dentin resulted in a higher dissolution rate of calcium salts. This defeats the potential of establishing a chemical bond between the resin monomers and apatite crystals [29]. The above-mentioned factors could partly explain the results of our study.

Air abrasion has been widely highlighted as a method of mechanical surface pretreatment in an attempt to enhance dentin bonding. In this context, the literature revealed contradictory results [14,30]. Different types of abrasives like calcium carbonate, sodium bicarbonate, and glycerine have been suggested to be used for sandblasting. Among these materials, aluminum oxide powder prevails in this field as it has the highest hardness compared to the other powders [31].

A systematic review by Lima, et al. 14 has revealed that the APA with Al2O3 has no negative influence on dentin bonding and a positive effect could be obtained only with a particle size greater than 30µm. However, Szerszeń, et al. [32] have reported that the Al_2O_3 abrasion with a different gradation (27 and 50µm) produced heterogeneous surface microgeometry and both could enhance the bond strength. Other parameters including the air pressure, working time, angle, and distance of the nozzle tip from the surface of the tooth are important for the abrasion's effectiveness[14.]

In the present study, air abrasion of the dentin surface with Al2O3 (25µm) significantly increased the SBS of lithium disilicate to dentin with all types of cement. Thus, the second null hypothesis was rejected. This result could be mainly justified by the micromechanical retention obtained with the increase in roughness and surface energy of dentin. Consequently, the air-abraded dentin surface has a greater contact area of adhesion and higher SBS [33]. Moreover, air abrasion with aluminum oxide particles seems to change the wettability of the surface which facilitates the resin penetration in the dentin and improves bonding effectiveness [32]. Along with the surface roughness, the literature has demonstrated other surface characteristics that could be affected by air abrasion including the dentinal tubules, inter-tubular dentin, smear layer, and resin tags [7].

Rafael, et al. [34] have revealed that the effect of APA on dentin surface was comparable to that of phosphoric acid in terms of smear layer removal, their SEM analysis showed that the APA with aluminum oxide particle expanded the contact area of bonding with the preservation of the amount of the intertubular dentin and the diameter of the dentin tubule orifice. Other studies have indicated that air abrasion of dentin does not eliminate the need for acid etching and its effect results in the thinning of the smear layer that could facilitate the penetration of acid in dentin and enhance the resin tags formation [35]. However, previous reports have shown conflicting findings regarding the effect of dentin air abrasion on the formation, removal, or compaction of the smear layer [36].

Regarding the SBS of GIC, the current study showed agreement with de Souza-Zaroni, et al. [37] who have recorded a higher SBS of GIC to the air-abraded dentin. Nevertheless, Chauhan, et al. [38] have reported that the APA of dentin surface has no significant effect on the bond strength of GIC. In line with the present study, previous studies have reported an improvement in the SBS of the self-adhesive resin cement to the sandblasted dentin [39,40]. Likewise, Szerszeń, et al. [32] have analyzed the physiomechanical parameters of dentin subjected to Al_2O_3, the authors have documented that the deposition of aluminum oxide clusters in the smear layer has increased the wettability, surface energy, and surface roughness of dentin leading to a significant increase in the SBS of the self-adhesive resin cement. For the adhesive resin cement, the current findings are consistent with the majority of studies that have demonstrated the positive effect of APA on the bond strength of the total-etch adhesives [7,33]. Besides the in vitro studies, Mavriqi, et al. [15] have documented that the use of water-air particle abrasion on dentin along with a 3-step etch-and-rinse adhesive technique could enhance the bond strength and the clinical performance of indirect glass-ceramic restorations. However, other studies have reported no improvement in the bond strength of the total-etch adhesives to the sandblasted dentin [30,41].

Comparing the SBS among the three types of cement to the sandblasted dentin, the same scenario was shown for the GIC cement which recorded the lowest SBS values. However, there was no significant difference in the SBS between the adhesive and self-adhesive resin cement to the air-abraded dentin. The possible explanation for this could be related to the effect of acid etching used with the adhesive resin cement. It has been shown that the dentinal tubules were more open following sandblasting and acid etching. Additionally, the authors have proposed that the acid etch and water rinse have a positive effect by removing the remnants of Al_2O_3 particles that might interfere with the adhesive penetration in dentin [15,33].

In addition to the SBS test, the failure mode analysis is essential to study the clinical performance of dental cement. In the present study, despite the low SBS values obtained with the GIC subgroups (with and without air abrasion), the cohesive and mixed failure patterns were more dominant than the adhesive failure pattern, this could be due to the chemical bond between the GIC cement and dentin. In the experimental groups with resin cements (adhesive & self-adhesive without sandblasting), the predominant failure was mixed and the remaining samples showed cohesive failure. Comparable failure patterns were obtained in a previous study [19]. Conversely, others have shown an adhesive failure with the self-adhesive cement [42]. However, the failure modes in our study comply with the results of the SBS test. For the air-abraded samples with the adhesive and self-adhesive resin cement, the majority of samples showed a cohesive failure in the cement that could imply the effectiveness of the bond between tooth and restoration [43]. These results are in agreement with a previous study that reported a cohesive failure with the air-abraded samples [7].

The major limitation of the present study is related to the immediate testing of the samples, the SBS values obtained after 24 hours of storage in water could be decreased after dynamic loading and thermal stresses in the oral cavity. Thermocycling has been reported in the literature as a widely accepted method allowing more accurate interpretation of the laboratory results. However, the immediate SBS test is still an important baseline, further investigations with aging are suggested to complement our findings. From a clinical point of view, it can be concluded that the air particle abrasion of dentin is a promising technique to enhance bonding of the adhesive restorations to dentin. Additionally, our results indicated that the self-adhesive resin cement can be implemented in the cementation of lithium disilicate crowns, this simplified protocol can be beneficial especially in restorations with subgingival margins when the isolation is critical for adhesive cementation.

Conclusions

Within the limitation of this study, the following conclusions could be drawn:

- Air-particle abrasion of dentin surface with aluminum oxide enhanced the shear bond strength of lithium disilicate significantly regardless of the luting cement type.
- Self-adhesive resin cement provided the highest shear bond strength of lithium disilicate to both sound and air-abraded dentin, while glass ionomer cement recorded the lowest.
- The adhesive mode of failure was noticed only in those specimens cemented with glass ionomer cement, while the predominant failure modes in specimens cemented with adhesive and self-adhesive resin cements were cohesive and mixed, regardless of sandblasting pretreatment or not.

Acknowledgment

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All authors have made substantive contribution to this study and/or manuscript, and all have reviewed the final paper prior to its submission

Declaration of Interest

The authors report no conflict of interest.

Material	Manufacturer	Chemical composition (wt%)
IPS e.max CAD All LT	Ivoclar Vivadent, Liechtenstein	SiO_2, Li_2O, P_2O_5, ZrO_2, ZnO_2, other oxides, coloring oxides
Phosphoric acid conditioner Scotchbond	3M ESPE, Germany	35% phosphoric acid.
IPS Ceramic Etching Gel	Ivoclar, Vivadent, Liechtenstein	≤ 5% hydrofluoric acid.
Single Bond Universal Adhesive	3M ESPE, Germany	10-MDP phosphate monomer, dimethacrylate resin, HEMA, Vitrebond TM copolymer, filler, ethanol, water, silane, initiator
Riva Self Cure glass ionomer cement	SDI, Victoria, Australia	Powder: Fluoroaluminosilicate glass Liquid: Acrylic acid, tartaric acid, water
RelyX Ultimate Adhesive resin cement	3M ESPE, Germany	Base paste; methacrylate monomers, adiopaque, silanated fillers, initiator components, stabilizer, and rheological additives. Catalyst paste; methacrylate monomers, radiopaue alkaline fillers, initiator components, stabilizer, pigments, rheological additives, fluorescence dye.
RelyX U200 Self adhesive resin cement	3M ESPE, Germany	Paste A—amine, bisphenol A glycidyl methacrylate (bis-GMA), triethyleneglycol dimethacrylate (TEGDMA), photoinitiators, inorganic particles of silica and zirconia (68% by weight), and pigments. Paste B—TEGDMA, bis-GMA, inorganic particles of silica and zirconia (67% by weight), benzoyl peroxide.

Table 1. Chemical composition and manufacturer of materials used.

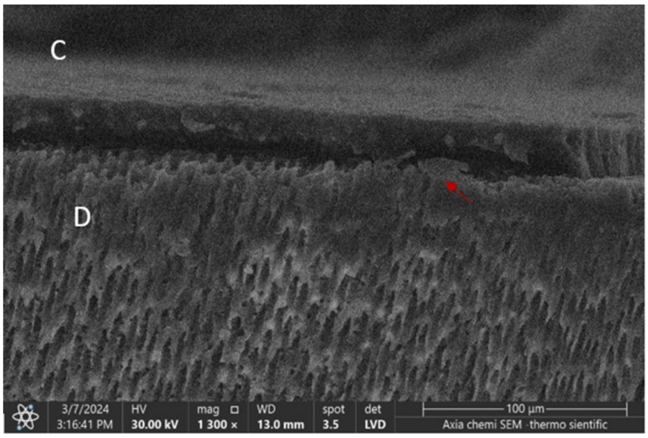

Figure 2. SEM image of RelyX U200 self-adhesive resin cement bonded to dentin (1300×): a representative area of the cement C/dentin D interface showing the smear plugs (red arrow), no visible resin tags are shown in the dentin.

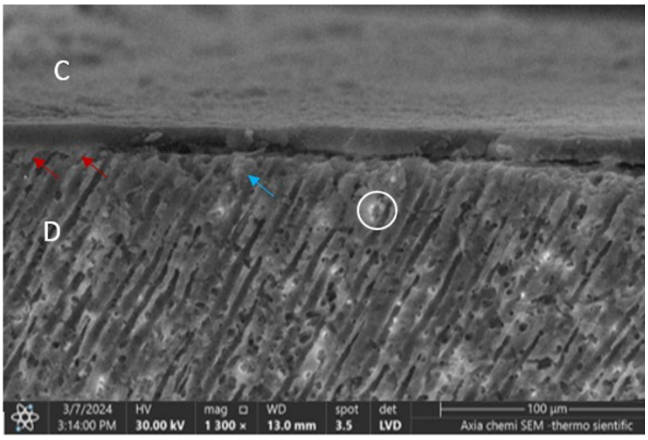

Figure 3. SEM image of RelyX U200 self-adhesive resin cement bonded to air-abraded dentin (1300×). a representative area of the cement C/dentin D interface showing the smear plugs (blue arrow) aluminum crystals deposited in the dentin (hollow circle), and the resin infiltration (red arrow).

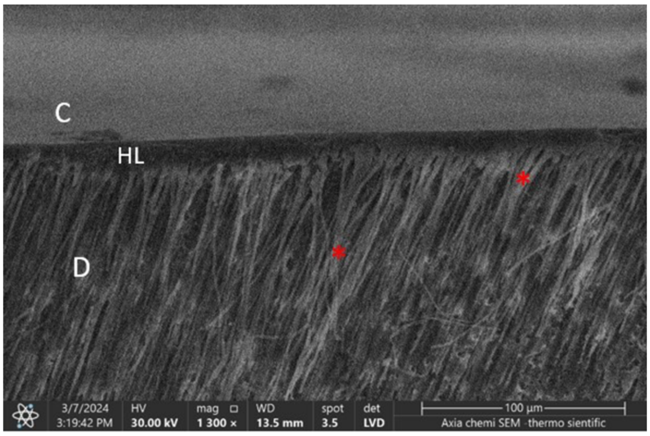

Figure 4. SEM image of RelyX Ultimate adhesive resin cement bonded to air-abraded dentin (1300×) a representative area of the cement C/dentin D interface showing a uniform hybrid layer HL and long resin tags*.

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