

Comparative analysis of shear bond strength of lithium disilicate samples cemented using different resin cement systems: An *in vitro* study

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Abstract

Aim: This study aims to evaluate and compare the shear bond strength (SBS) of three different resin cements - total etch and rinse, self-etch and self-adhesive resin cements, used to bond the lithium disilicate restorations to human dentin.

Settings and Design: Comparative *-Invitro* study design.

Materials and Methods: Forty-five lithium disilicate (IPS E.max) discs (4 mm in diameter and 3 mm thick) were fabricated and randomly divided into three groups ($n = 15$). The occlusal surfaces of 45 extracted human maxillary premolars were ground flat. Fifteen specimens were luted, under a constant load, with each of the following resin cement: Variolink N (Group VN), Multilink N (Group MN), and Multilink Speed (Group MS). All cemented specimens were stored in distilled water for 1-week following which, they were tested under shear loading at a constant crosshead speed of 1 mm/min until fracture on a universal testing machine; the load at fracture was reported in megapascals (MPa) as the bond strength. Fractured specimens were also inspected by the scanning electron microscopy. Statistical analysis of the collected data was performed using one-way ANOVA test, *post hoc* Bonferroni test, and Chi-square test ($\alpha = 0.05$).

Statistical Analysis Used: Oneway ANOVA test and *post hoc* Bonferroni test.

Results: Mean SBS data of the groups in MPa were: Variolink N (Group VN): 14.19 ± 0.76 ; Multilink N (Group MN): 10.702 ± 0.75 ; and Multilink Speed (Group MS): 5.462 ± 0.66 . Significant differences in SBS ($P < 0.001$) of the three resin cement were found. Intergroup comparison revealed statistically significant differences in SBS between Groups VN and MN ($P < 0.001$), Groups B and C ($P < 0.001$), and Groups VN and MS ($P < 0.001$). Chi-square test used to compare the distribution of mode of bond failure among the three groups delineated that the cohesive failure was significantly more among Group VN, whereas adhesive failure was significantly more among Group MN and MS.

Conclusion: Total etch and rinse resin cement, i.e., Variolink N (Group VN) produced significantly higher bond strength of all-ceramics to dentin surfaces than did the self-etch and self-adhesive resin cements, i.e., Multilink N and Multilink Speed, respectively.

Keywords: Self-adhesive resin cement, self-etch resin cement, total-etch resin cement

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INTRODUCTION

Lithium disilicate, a well-known glass ceramic, is highly esthetic, thermal shock resistant material. It has large volume (70% approximately) of the long crystals which increase their flexural strength, fracture resistance, and bond strength, thus, making it possible to utilize this material for the fabrication of prosthesis in the anterior region of the mouth and the restoration of premolars.^[1] Restorations using this glass ceramic can be fabricated either by lost-wax hot pressing techniques or modern computer-aided design (CAD)/CAD milling procedures^[1] which further helps in improving the mechanical and optical properties of the ceramic.^[2] Pressable lithium disilicate is indicated for inlays, onlays, thin veneers, veneers, partial crowns, anterior and posterior crown, 3-unit anterior or premolar bridges, telescope primary crowns, and implant restorations. It can be pressed as thin as 0.3 mm while still ensuring the strength of 400 megapascals (MPa).^[1] Although the flexural strength of zirconia (1000 MPa) is twice the flexural strength of lithium disilicate,^[3] lithium disilicate is used more commonly where esthetics and strength are required simultaneously.

The integrity and the longevity of the tooth-cement-ceramic interface depend on the luting or bonding procedure, adhesive capacity, and the stiffness of the luting agent being used for the cementation of the restoration to the tooth substrate; therefore, the luting agent used for their cementation can be the “Achilles heel.”^[4] The desired features of a luting agent are its optical characteristics, improved mechanical properties, low solubility, decreased microleakage, low incidence of marginal staining, and ability to bond to multiple substrates.^[5,6] Moreover, due to the high strength^[7] and glass properties of the lithium disilicate adhesive cementation^[8] is recommended to lute such restorations to the tooth substrate.^[5]

The resin cements were first developed in 1950s and by Dr. Rafael Brown in 1963^[9] and according to the conditioning of tooth before cementation, resin cements are divided into three groups, i.e., total etch and rinse resin cements, self-etch resin cement system, and self-adhesive resin cement system/all-in-one resin cements.^[8,10] The conventional technique for cementation, i.e., total-etch adhesive system, is technique sensitive and involves various steps before cementation.^[11] In this technique, the intaglio surface of the restoration is etched with hydrofluoric (HF) acid followed by the application of the silane coupling agent which is responsible for the chemical union between the restoration and the resin cement.^[5,8] Consecutively, the prepared tooth surface is conditioned with phosphoric acid;

to increase the surface energy by removing the smear layer; and then, the bonding agent is applied following which final cementation is performed.^[8] To reduce the number of operative steps and simplify the clinical procedures self-etch resin cement systems were introduced, which includes the application of self-etching acidic primer followed by the application of resin cement.^[8] In 2002, self-adhesive resin cements were introduced^[10] those incorporate etchant, primer, and bonding resin in a single solution. Therefore, no treatment of the prepared tooth before cementation is required.^[8] They possess no postoperative sensitivity, reduce chairside time, are moisture tolerant, dimensionally stable, and easy to apply, release fluoride ions, offer good esthetics, have optimal mechanical properties, and adhere micromechanically. The adhesion between the cement and tooth is obtained by the chemical interaction between the multifunctional monomer with phosphoric acid groups and hydroxyapatite.^[10] Moreover, they act as a permeable membrane after polymerization resulting in mechanical disruption of the coupling between the adhesive and composite resin that can be minimized by selecting a conventional 3-step and 2-step resin cement system.^[12] Many studies have compared shear bond strength (SBS) of total-etch resin cements with self-etch resin cements or self-adhesive resin cements^[13-21] but few studies compared all the three resin cement systems. However, scarce information is available with regard to the bond strength directly between dentin and indirect substrates. Hence, this study aimed at comparing the SBS of lithium disilicate samples cemented on tooth substrate using total-etch, self-etch, and self-adhesive resin cement systems from the same manufacturer. The null hypothesis stated that there is no significance difference in the SBS of lithium disilicate samples bonded using total etch and rinse, self-etch and self-adhesive resin cements.

MATERIALS AND METHODS

For this study, 45 disk-shaped lithium disilicate specimens were fabricated and luted to the tooth substrate following which they were divided into three groups based on the type resin cements used for cementation: Group VN (Variolink N); Group MN (Multilink N); and Group MS (Multilink Speed). All the bonded specimens were stored and tested for their SBS. Details of the resin cements used in the study are listed in Table 1.

Preparation of tooth surface

Forty-five freshly extracted human maxillary premolars were stored in distilled water at room temperature from the day of extraction until testing. The teeth were mounted up to 1 mm below the cemento-enamel junction in autopolymerizing acrylic resin (DPI RR Cold Cure, The Bombay Burmah

Trading Corporation Ltd., Mumbai, India) in a custom made metallic mold (2 cm × 1.5 cm × 2 cm) [Figure 1] with the help of the Ney's surveyor (DENTSPLY India Pvt. Ltd., India). The mounted samples were randomly divided into three groups: Group VN, Group MN, and Group MS ($n = 15$) [Table 1]. The occlusal surface of the mounted samples was ground flat and parallel to the base of the mold with the help of a diamond disk, mounted to a dental surveyor milling machine (Bredent BF2, Bredent GmbH and Co. KG, Senden, Germany) [Figure 2].

Fabrication of lithium disilicate discs

Forty-five lithium disilicate (IPS E.max Press, Ivoclar Vivadent AG, Schaan Liechtenstein) discs (4 mm diameter and 3 mm width) were fabricated using a retractable custom made metallic die [Figure 3]; that was used to fabricate the wax patterns for the discs; and IPS Emax Press ingots (Ivoclar Vivadent) employing the lost-wax hot pressing technique.

Cementation of lithium disilicate discs to the tooth substrate

The surface of all the discs was etched with 4.5% HF acid (IPS Ceramic Etching gel, Ivoclar Vivadent) for 20 s and then washed thoroughly and dried. Following this, a

silane coupling agent (Monobond N, Ivoclar Vivadent) was applied for 60 s. The remaining excess was dispersed. In Group VN, the prepared tooth was etched with 37% phosphoric acid gel (N-etch, Ivoclar Vivadent) for 15 s and washed. Consecutively, dentin-enamel adhesive system (Syntac[®] Primer and Syntac[®] Adhesive, Ivoclar Vivadent) and Heliobond (Ivoclar Vivadent) was applied according to the manufacturer's instructions. The base and catalyst of Variolink N (Ivoclar Vivadent) were mixed and applied on the intaglio surface of the pretreated discs and discs were seated on the pretreated tooth surface under constant load [Figure 4]. The resin cement was cured according to the manufacturer's instructions. In Group MN, Multilink N Primer A and Primer B (Ivoclar Vivadent) mixed in 1:1 ratio was applied and scrubbed on the prepared tooth surface according to the manufacturer's instructions. Subsequently, the desired amount of Multilink N (Ivoclar Vivadent) was mixed and dispensed on the intaglio surface of the pretreated disc, and the discs were seated, on the pretreated tooth surface under constant load, and the resin cement was cured according to the manufacturer's instructions. In Group MS, the desired amount of Multilink Speed (Ivoclar Vivadent) was mixed



Figure 1: Mounted specimen in custom made metallic mold

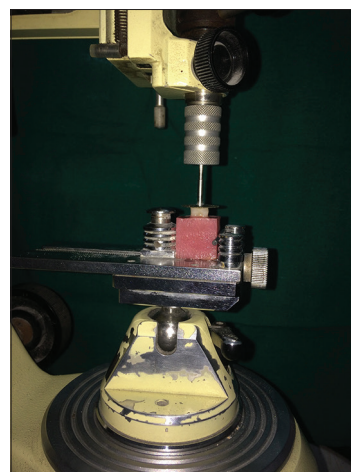


Figure 2: Preparation of occlusal surface of maxillary premolars

Table 1: Resin cements used in the study with grouping

Resin cements	Composition	Manufacturer
VN (Group VN: Total etch and rinse)	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, Ba-Al-fluorosilicate glass, barium glass, ytterbium trifluoride, spheroid mixed oxide, initiators, stabilizers, pigments	Ivoclar Vivadent
Syntac primer	Triethyleneglycol methacrylate, polyethyleneglycol dimethacrylate, maleic acid and ketone in aqueous solution	Ivoclar Vivadent
Syntac adhesive	Polyethylene dimethacrylate and glutaraldehyde in aqueous solution	Ivoclar Vivadent
Heliobond	Bis-GMA, triethyleneglycol dimethacrylate, stabilizers and initiators	Ivoclar Vivadent
MN (Group MN: Self-etch and rinse)	Dimethacrylates, HEMA, barium glass, ytterbium trifluoride, spheroid mixed oxide	Ivoclar Vivadent
Primer A	Aqueous solution of initiators	Ivoclar Vivadent
Primer B	HEMA, phosphonic acid, and methacrylate monomers	Ivoclar Vivadent
MS (Group MS: Self-adhesive)	Dimethacrylates, acidic monomers, barium glass, ytterbium trifluoride, co-polymer, silicon dioxide, initiators, stabilizers and color pigments	Ivoclar Vivadent

VN: Variolink N, MN: Multilink N, MS: Multilink speed, HEMA: Hydroxyethyl methacrylate, Bis-GMA: Bisphenol A-glycidyl methacrylate

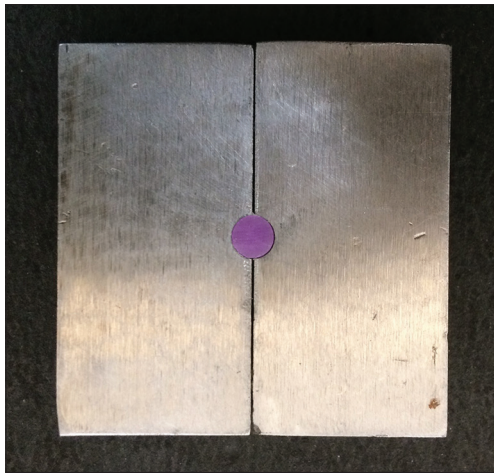


Figure 3: Retractable custom made metallic mold for wax pattern fabrication

and dispensed on the intaglio surface of the pretreated discs, and the discs were then seated on the prepared tooth surface under constant load, and the resin cement was cured, according to the manufacturer's instructions. No pretreatment of the prepared tooth surface was required as per the manufacturer's instructions.

All the bonded specimens were stored in the distilled water for 1 week at a constant temperature of 37°C. SBS was measured for each sample with a universal testing machine (UTM) (Autograph, AG-IS, Shimadzu). The sample was loaded at 90° to the long axis of the tooth at the ceramic tooth interface at the crosshead speed of 1 mm/min. The samples were loaded until ceramic disc debonded from the tooth surface, and the maximum load was measured. The SBS was calculated in MPa by dividing this value by the area of the discs for each specimen. Following this, the mode of failure was checked for all the samples using scanning electron microscope (SEM) (LEO 435 VP, SEMTech Solutions). The data obtained [Table 2] were statistically analyzed using appropriate tests.

RESULTS

One-way ANOVA test [Table 3] was used to compare the mean SBS of three groups and revealed that there was a significant difference ($P < 0.001$) in the SBS of three resin cements. The *post hoc* Bonferroni test [Table 4] evaluated the most clinically recommendable resin cement among the three groups and showed that the mean SBS value (MPa) was highest for Group VN (Variolink N; 14.19 ± 0.76 MPa) followed by Group MN (Multilink N; 10.7 ± 0.75 MPa) and was least for Group MS (Multilink Speed; 5.46 ± 0.66 MPa). In the current study, SEM study was done at $\times 500$ on fractured specimens, and mode

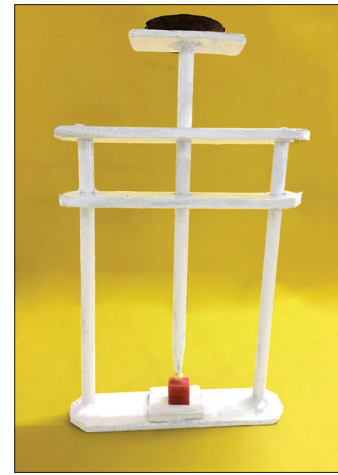


Figure 4: Cementation of discs under constant load

Table 2: Shear bond strength (megapascals) of different resin cement systems

Number of specimens	Group VN	Group MN	Group MS
1	14.41	9.57	4.47
2	14.94	9.58	5.648
3	14.59	10.19	4.312
4	14.94	11.55	6.23
5	14.86	9.82	5.419
6	15.08	10.55	4.96
7	12.96	11.11	5.16
8	13.87	11.97	5.41
9	13.44	11.79	5.07
10	13.34	11.07	5.96
11	15.29	10.83	5.67
12	13.21	10.41	5.162
13	14.25	10.27	6.45
14	13.52	11.09	6.59
15	14.16	10.73	5.42

VN: Variolink N, MN: Multilink N, MS: Multilink speed

Table 3: One-way ANOVA test to compare the mean shear bond strength values (megapascals) of three groups

Groups	Mean \pm SD	P
Group VN: VN	14.19 \pm 0.76	<0.001*
Group MN: MN	10.70 \pm 0.75	
Group MS: MS	5.46 \pm 0.66	

*Significant difference. VN: Variolink N, MN: Multilink N, MS: Multilink speed, SD: Standard deviation

Table 4: Post hoc Bonferroni test for intergroup comparison of mean difference of shear bond strength values

Groups	Shear bond strength (MPa) (N/mm ²)	
	Mean difference	P
Group VN: VN Group MN: MN	3.49	<0.001*
Group VN: VN Group MS: MS	8.73	<0.001*
Group MN: MN Group MS: MS	5.24	<0.001*

*Significant difference. MPa: Megapascals, N/mm²: Newton/millimeters², VN: Variolink N, MN: Multilink N, MS: Multilink speed

of failure for all the specimens from all the groups was analyzed. The intergroup comparison of mode of failure was depicted by Chi-square test [Table 5], and a significant

difference was determined in the distribution of the mode of failure among the three groups. The cohesive failure was significantly ($P < 0.001$) more among Group VN, whereas adhesive failure was significantly more among Group MN followed by Group MS.

DISCUSSION

All-ceramic restorations are currently available highly esthetic restorative material that can simulate the appearance of natural dentition.^[22] Their evolution has been a battle for ideal strength-esthetic combinations.^[23] One of the most popular all-ceramic systems is pressable ceramics due to their excellent mechanical and esthetic properties.^[18] Even though they have many advantages, dental ceramics are fragile under tensile strain^[22] and are prone to fracture under chewing loads.^[5] Thereby making the cementation process very important for the clinical success of these restorations.^[22] A strong resin bond between a ceramic restoration and the tooth structure provides good support for the restoration and transmits functional loads through the bonded interface.^[24] The purpose of this study was to evaluate and compare the SBS of three different resin cements to both ceramic and dentin and to evaluate the mode of bond failure by SEM due to the widespread popularity and usage, IPS E.max Press (a lithium disilicate, heat pressed all ceramic material) has been used in this study.^[22]

At the ceramic tooth interface, stresses are complex and can be identified as tensile or shear types of stresses created by forces working either perpendicular or parallel to the tooth surface.^[25] Moreover, Holderegger *et al.*^[14] stated that the forces of displacement of crown tend to be closer to shear than to tensile stresses. In 1993, Oilo discussed the accuracy and clinical relevance of different testing methods and concluded that SBS was the simple^[26] and most common testing method.^[18,27] In addition, the appropriateness of this method to test the occlusal and approximal surfaces was certified by Lührs *et al.*^[28] Thus, in this study SBS test has been used to measure the bond strength of the resin cements.

Table 5: Chi-square test to compare the distribution of mode of bond failure among the three groups

Mode of failure	Group VN (n=15), n (%)	Group MN (n=15), n (%)	Group MS (n=15), n (%)	Total, n (%)
Adhesive	0 (0.0)	9 (60.0)	12 (80.0)	21 (46.7)
Cohesive	15* (100.0)	6 (40.0)	3 (20.0)	24 (53.3)
Total	15 (100.0)	15 (100.0)	15 (100.0)	45 (100.0)

$\chi^2=20.893$, * $P<0.001$. VN: Variolink N, MN: Multilink N, MS: Multilink speed

In the present study, lithium disilicate discs were luted to human maxillary premolars using three different resin cement systems. In Group VN, the tooth substrate was etched with 37% phosphoric acid followed by the application of dentin bonding agent and resin cement whereas Group MN includes only primer application followed by resin cement. Moreover, in Group MS, no pretreatment of the tooth was required. This difference in the luting procedure can be imputed to the difference in the composition of resin cements.

The samples were luted to the tooth substrate using “Calibrated finger pressure” which is about 20 gm/mm²,^[28] to ensure a uniform thickness of the resin cement. Therefore, specimens were loaded with a small weight (250 g) adjusted to the specimens surface (12.56 mm²). Consecutively, the bonded samples were stored in distilled water at 37°C for 1 week, which is similar to the study conducted by Pekkan and Hekimoglu^[25] and Peutzfeldt *et al.*^[29] and assuming that the polymerization of the resin cements would have completed and the maximum bond strength would have been attained.^[25]

After aging the samples were exposed to the SBS test using UTM and within the limitations of this study, Variolink N showed highest SBSs to human dentin followed by Multilink N and the SBS values were least for Multilink Speed. These results were in consensus with previous studies reported in the literature.^[17,18,20,30,31] Maximum bond strength of Variolink N can also be attributed to its higher filler content (base: 73.4% wt./46.7% vol and Catalyst: 71.2% wt. and 43.6% vol.) and presence of urethane dimethacrylate in its composition which is more flexible than bisphenol A-glycidyl methacrylate because of urethane linkages and lower viscosity, facilitating the migration of free radicals, increasing the degree of crosslinking which in turn results in superior adhesion and increased bond strength.^[32] It also provides a better degree of conversion, that prevents the leaching of unreacted monomer, thereby inhibiting the hydrolysis when exposed to oral fluids or water storage.^[7]

The lower bond strength of Multilink N when compared to Variolink N can be attributed to its composition and decreased the ability to etch the tooth surface. This self-etch resin cement contains hydroxyethyl methacrylate which is hydrophilic in nature^[7] thus, polymerizes in the presence of water and forms a microporous hydrogel with pore size ranging from 10 to 100 nm.^[26] Furthermore, it absorbs more water that acts as plasticizer within polymer matrix and leads to degradation of the filler-matrix interface resulting in deterioration of mechanical or physical properties of cements. Moreover, the bond strength is

lowered after aging.^[7] Bonding performance of resin cements also depends on the quality of the hybrid layer. The self-etching primer does not lead to the establishment of a dense hybridization layer, which allows the penetration of the water molecules those further leads to the hydrolysis of the cement bond and subsequently decreases the bond strength.^[14]

Contrarily, the higher bond strength of Multilink N as compared to Multilink Speed can be attributed to their capability of penetrating the aqueous channels formed between the smear layer particles, widening these channels and interacting with the top of underlying dentin.^[26] Moreover, the mode of application of the priming mixture is a key factor for receiving high bond strength with Multilink N which is why the mode of application of a priming agent is strongly influenced by the operator, and hence, these systems are judged as technique sensitive.^[14] However, in contrast to Multilink N, Multilink Speed was least influenced by the operator, probably because it uses no priming system, thereby rendering it less technique sensitive.

The minimum bond strength of Multilink Speed is in agreement with the study conducted by Farrokh *et al.*^[33] and can be elucidated with the following reasons: (a) lower degree of cure and higher water solubility as compare to Multilink N; (b) inability to remove the smear layer completely; (c) absence of hybrid layer; (d) insufficient penetration of this cement into the dentinal tubules and collagen fibers because of high viscosity,^[33] and (e) existence of low-molecular-weight oligomers that allows water to penetrate the junction of resin cement and tooth structure.^[34]

To prognosticate the clinical performance of the resin cements, evaluating the bond strength is not sufficient. Thus, the assessment of the mode of failure is also of paramount importance.^[32] In the present study, cohesive and adhesive mode of failures were assessed using SEM ($\times 500$).^[27] The SEM examination revealed a layer of resin cement on the ceramic and tooth surface respectively, representing the cohesive mode of bond failure [Figures 5 and 6] within the Variolink N resin cement that can be allocated to its higher bond strength^[21,28] as compared to other two resin cements. Furthermore, it shows the efficacy of the bond between the interface of the dentin/resin and resin/ceramic interfaces.^[32] The SEM examination of the Multilink N specimens divulged no resin cement on tooth surface which refers to the incomplete removal of the smear layer and deficient demineralization of the dentin whereas resin cement can be seen on the ceramic surface [Figures 7 and 8]. Nevertheless, interfacial gaps [Figure 9] are located in the weak smear layer and can be allocated

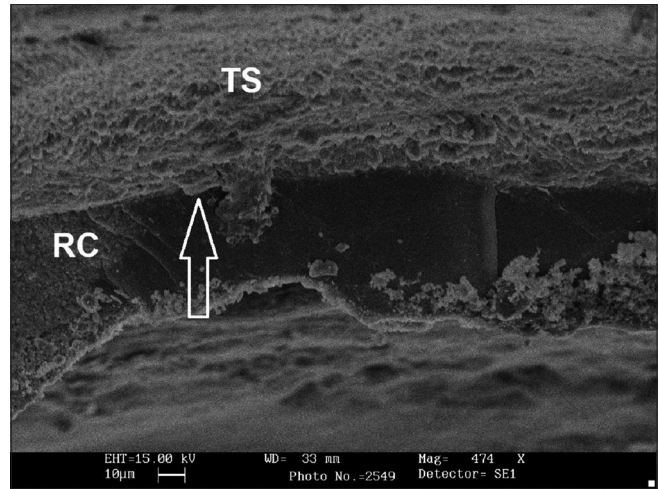


Figure 5: Scanning electron microscopy image of section of tooth after debonding (Group VN). TS: Tooth substrate, RC: Resin cement, Arrowhead: Cement tooth interface, VN: Variolink N

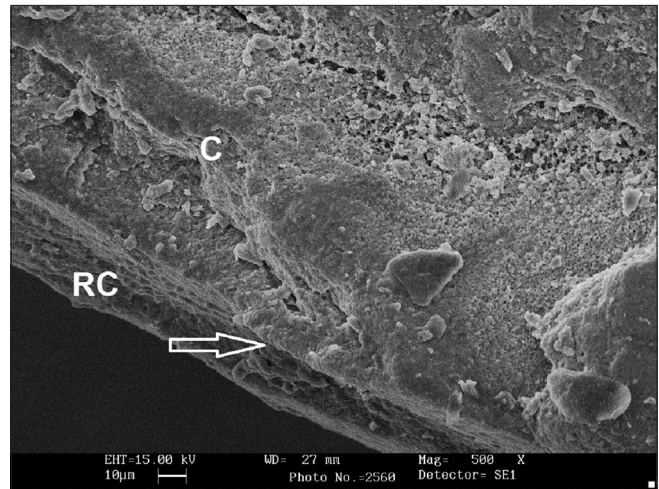


Figure 6: Scanning electron microscopy image of lithium disilicate disc after debonding (Group VN). C: Ceramic surface, RC: Resin cement, Arrowhead: Cement ceramic interface, VN: Variolink N

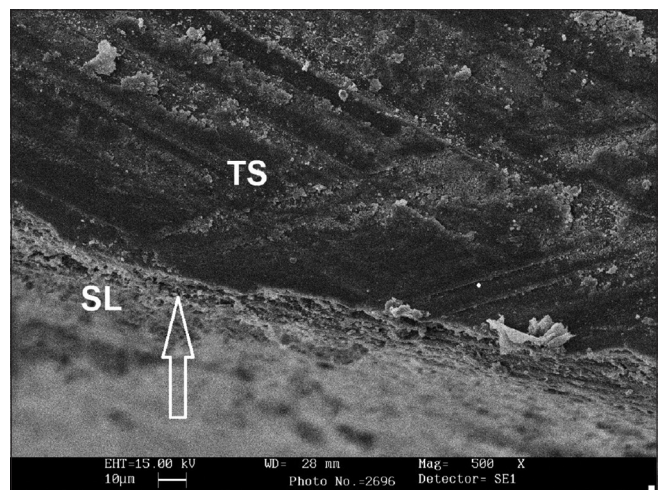


Figure 7: Scanning electron microscopy image of section of tooth after debonding (Group MN). TS: Tooth substrate, SL: Smear layer, Arrowhead: Cement tooth interface, MN: Multilink N

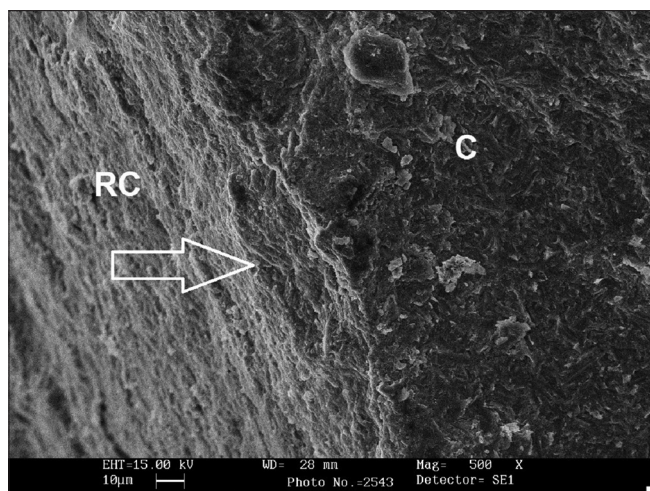


Figure 8: Scanning electron microscopy image of lithium disilicate disc after debonding (Group MN). RC: Resin cement, C: Ceramic surface, Arrowhead: Cement ceramic interface, MN: Multilink N

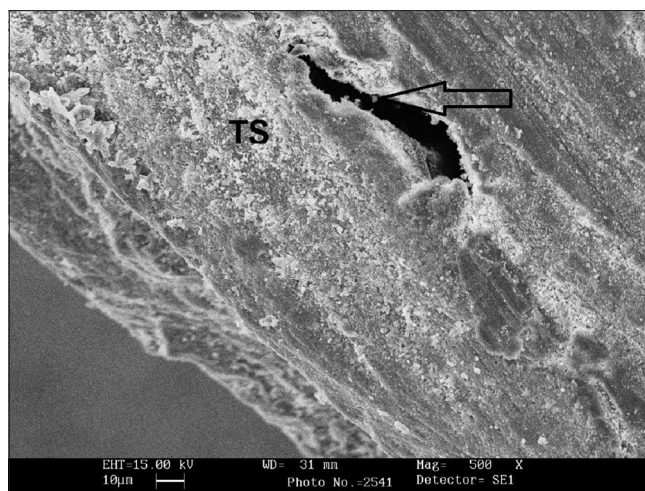


Figure 9: Scanning electron microscopy image of section of tooth after debonding (Group MS). TS: Tooth substrate, Arrowhead: Interfacial gaps, MS: Multilink speed

to the residual dehydration of samples or polymerizations shrinkage of the cement thereby revealing a close bonding of the cements to dentin, but without the formation of the hybrid layer or resin tags.^[30] Thus, little and superficial interaction between resin and dentin without hybrid layer^[28] could be seen. In addition, resin cement could be seen on the surface of the ceramic specimen from Multilink Speed group. This above-mentioned reason justifies the adhesive mode of bond failure for Multilink N and Multilink Speed. The results of the SEM examination for all three groups are in consensus with the findings of the other studies.^[18,28,32] Although this *in vitro* study allowed a prompt evaluation of the bond created between the cement and the all-ceramic material, the adjunct clinical factors such as retentive and resistance form of the preparation were not considered thus, cannot adequately simulate the clinical conditions in every detail.^[35] In addition, the complex nature of the masticatory forces in the oral cavity could not be produced by UTM.^[36] Therefore, the final evaluation of material performance should be determined using long-term clinical studies.^[35]

CONCLUSION

The results obtained from the current investigation corroborate this assertion, and it can be concluded that total etch resin cements are the most reliable luting agents and are clinically recommended to establish a durable bond between lithium disilicate ceramic and dental substrate. However, it can be considered as the gold standard to lute the lithium disilicate or other all-ceramic restorations followed by self-etch resin cement systems and finally self-adhesive resin cement systems. Although the selection of resin cement is based on the clinical situation as well

as clinician's preference, further clinical investigations are requested to decide the reliability and clinical performance of different resin cements.

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Conflicts of interest

There are no conflicts of interest.

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