



## Comparison of Shear Bond Strengths of Conventional Resin Cement and Self-adhesive Resin Cement bonded to Lithium Disilicate: An *in vitro* Study

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### ABSTRACT

**Aim:** The aim of this study is to compare the shear bond strengths of conventional resin cement and self-adhesive resin cement bonded to lithium disilicate.

**Materials and methods:** A total of 40 extracted human molar teeth were mounted in self-cure acrylic resin. Teeth were prepared to obtain flat occlusal surface. About 40 lithium disilicate specimens of dimension—10 mm in diameter and thickness of 2 mm—were fabricated using lost wax technique. The samples were divided into four groups: Groups I, II, III, and IV (n = 10). The specimens were surface treated with Monobond S silane coupling agent. Self-etching primer and bonding agent were applied on the bonding surface of the teeth in groups I and III. The specimens were bonded to the primed teeth with the Multilink N resin cement and subjected to the universal testing machine. The specimens were light-cured. Specimens in groups II and IV were luted to teeth using self-adhesive cement RelyX U100. The same force was applied over the specimen as mentioned above. Excess cement was removed, and light curing was done. The specimens in groups III and IV were subjected to thermocycling for 10,000 cycles at temperatures altering between 5°C and 55°C.

**Results:** The shear bond strengths of conventional resin cement and self-adhesive resin cement with lithium disilicate were tested before and after thermocycling. Results indicated that

thermocycling has no significant effect on the bond strengths of conventional or self-adhesive resin cement. However, from the study, it is seen that conventional resin cement had a higher shear bond strength value than the self-adhesive resin cement.

**Conclusion:** There was a significant difference between the average shear bond strength values of conventional resin cement (Multilink N) and self-adhesive resin cement (RelyX U100) when bonded to lithium disilicate disks, and thermocycling had no significant effect on the bond strength of conventional or self-adhesive resin cements.

**Clinical significance:** Among all-ceramic systems available, lithium disilicate materials have emerged as an excellent esthetic material for fabrication of anterior and posterior crowns and three-unit anterior fixed partial dentures because of their high translucency and improved optical properties. For successful clinical outcomes, the luting agent should have high bond strength not only to the ceramic surface, but also to the tooth surface.

**Keywords:** Conventional resin cement, Dual-cure resin cement, Lithium disilicate, Pressable ceramics, Self-adhesive resin cement, Thermocycling.

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### INTRODUCTION

The demand for all-ceramic restorations has grown considerably during the past decade, and esthetics is the only reason for patients to seek ceramic restorations.<sup>1</sup> The popularity of these restorations is attributed to their esthetic quality, fracture resistance, tissue acceptance, and utmost satisfaction.<sup>2</sup> Ceramics in crown and bridge prosthodontics were fused to metal copings to increase

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their resistance to fracture. The metal substructure, however, has esthetic limitations, such as reduced light transmission and discoloration. These disadvantages have promoted the development of all-ceramic system that does not require metal.<sup>3</sup>

The increasing demand for all-ceramic restorations with a substructure of high strength led to the popularity of pressable ceramics. Among the pressable ceramics, lithium disilicate offers excellent esthetics and optimum strength for anterior crown and short span bridges. The most widely used pressable ceramics are IPS Empress 2 or IPS e.max. The IPS e.max system is an innovative all-ceramic system that comprises excellent esthetics, high-strength materials for both the press and computer-aided design and computer-aided manufacturing technology.

Fracture and debonding are the two critical properties of all-ceramic restorations.<sup>4</sup> When bonding ceramic to tooth structure, two different interfaces need to be considered: The dentin/adhesive interface and the ceramic/cement interface. The bond strength at both of these interfaces should be optimized because the lower one will determine the final bond strength of the cemented restoration.<sup>5</sup>

Adhesive cements play a major role in achieving greater bond strength. Composition of the resin luting agents and their polymerization forms may influence their properties. The polymerization method of the composite resin influences the quality of bonding to the dentin substrate. Bond strength is directly related to the polymerization degree of conversion. The bond strength between resin luting agent and dentin structures is an important feature that must be investigated. Resin luting agent should provide bond strength sufficient to resist stresses generated by its polymerization shrinkage.<sup>6</sup>

The purpose of this *in vitro* study is to evaluate the shear bond strengths of two resin cement, namely, Multilink N and RelyX U100 cemented to IPS e.max Press lithium disilicate ceramic using Monobond S as silane coupling agent.

## MATERIALS AND METHODS

The present *in vitro* study was conducted in the ceramic laboratory, Department of Prosthodontics at The Oxford Dental College Hospital and Research Center, Bengaluru, Karnataka, India.

### Materials Required

A total of 40 extracted human 2nd molar teeth, autopolymerizing acrylic resin (DPI-RR cold cure™/DPI, Mumbai, India), blue inlay wax (Hindustan Dental Products, Mumbai, India), lithium disilicate ingots (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein),

Multilink N (Ivoclar Vivadent, Liechtenstein): Multistep resin cement, RelyX U100 resin cement (3M ESPE dental products; Seefeld, Germany), single-step resin cement, distilled water, rubber bowl, porcelain jar, lecron carver, straight plaster spatula, calibrated jar, polyvinyl pipe of 3 cm diameter and 5 cm length, mixing pad, cement spatula (plastic spatula or agate spatula), Moon's curved explorer, Vernier caliper (GDC™, Germany), metallic scale of 15 cm length, slow-speed airtor handpiece (NSK EC JAPAN), Universal testing machine (Hounsfield), mounting assembly, Programat EP300 furnace (Ivoclar Vivadent, Schaan, Liechtenstein), light curing unit (Lediton, Ivoclar Vivadent, Schaan, Liechtenstein), and Thermocycling unit (Uvigene, England) were used.

### Methodology

A total of 40 extracted sound human 2nd molar teeth were collected for the study. The teeth were cleaned and stored in distilled water at room temperature. A cylindrical polyvinyl pipe mold of 3 cm diameter and 5 cm length was cut. Autopolymerizing acrylic resin was mixed in a ratio of 3:1 by volume and poured in the cylindrical polyvinyl pipe mold. The teeth were embedded in the center of the mold containing autopolymerizing acrylic resin until the cemento-enamel junction. Once the acrylic resin sets, the resin was retrieved from the cylindrical polyvinyl pipe mold. Occlusal surfaces of the specimens were trimmed until pulp canals were seen, with a slow speed handpiece using diamond disk under copious water irrigation to obtain a flat occlusal surface perpendicular to the long axis of the tooth.

Lithium disilicate disks were then fabricated in the following steps involving preparation of mold, impression, wax pattern, investment burn out, and pressing. Ingots of IPS e.max of lithium disilicate were then placed in the mold space and pressed in a pressable machine (Programat EP300 furnace, Ivoclar Vivadent, Schaan, Liechtenstein) of temperature 920°C for 20 minutes followed by cooling it in air for half an hour. This was followed by the retrieval of the samples from the investment mold and cleaning of the samples using glass beads. The samples were separated from the attached sprued using a metal cutting disk. All the 40 samples were then evaluated for their shape and size.

The samples were randomly divided into four groups (n = 10), namely, I, II, III, and IV. Group I consists of 10 samples of lithium disilicate disks treated with Monobond S and cemented using Multilink N resin cement without thermocycling. Group II consists of 10 samples of lithium disilicate disks treated with Monobond S and cemented using RelyX U100 resin cement without thermocycling. Group III consists of 10 samples of lithium disilicate disks

treated with Monobond S and cemented using Multilink N resin cement subjected to thermocycling. Group IV consists of 10 samples of lithium disilicate disks treated with Monobond S and cemented using RelyX U100 resin cement subjected to thermocycling.

### Cementation

The samples with the teeth embedded in acrylic resin were cemented with lithium disilicate disks using resin cement. All the 40 lithium disilicate disks were surface treated with Monobond S silane coupling agent for 60 seconds as per manufacturer's recommendations. The tooth samples were later placed over a metal block on the lower half of the universal testing machine, and the lithium disilicate disks were mounted onto a jig in the upper half of the universal testing machine. The 20 disks were cemented using Multilink N, which is a multistep resin cement, and 20 disks cemented using RelyX U100, which is single-step resin cement. Self-etching primer and bonding agent supplied in the Multilink N kit were applied on the bonding surface of the teeth in groups I and III for 15 seconds as per the manufacturer's instructions.

The samples of groups II and IV were luted to lithium disilicate disks using self-adhesive resin cement RelyX U100. The samples of groups II and IV were luted using RelyX U100, and samples in groups I and III were cemented using Multilink N resin cement. The samples were then subjected to a force of 50 gm. Excess cement was removed from the periphery using an explorer. The specimens were then light-cured using light-curing unit (Ledition, Ivoclar Vivadent, Schaan, Liechtenstein) for 40 seconds as per the manufacturer's recommendations.

### Thermocycling

The samples in groups III and IV after cementation were subjected to thermocycling for 10,000 cycles by immersing in water bath between 5°C and 55°C with an immersion time of 1 minute. Transfer time between the two water baths was 15 seconds.

### Mounting of Samples for Shear Testing

After completion of thermocycling, the samples were placed in a sample holder and mounted on the universal testing machine. Load was then applied perpendicular to

**Table 1:** Mean and SD of shear bond strength scores according to the four groups

| Groups | n  | Mean ± SD    | Standard error |
|--------|----|--------------|----------------|
| I      | 10 | 14.64 ± 2.37 | 0.74           |
| II     | 10 | 5.48 ± 1.01  | 0.32           |
| III    | 10 | 15.59 ± 2.89 | 0.91           |
| IV     | 10 | 6.36 ± 1.03  | 0.32           |

SD: Standard deviation

the long axis of the samples, with a chisel-shaped piston at a crosshead speed of 0.5 mm/minutes until failure. The maximum force (N) was recorded, and the shear bond strength (MPa) was calculated by dividing the load (N) by the bonding area (mm<sup>2</sup>).

### Statistical Analysis

One-way analysis of variance (ANOVA) test ( $\alpha = 0.05$ ) for multiple group comparisons and Tukey's *post hoc* pairwise comparison test ( $\alpha = 0.05$ ) were applied for statistical analysis.

### RESULTS

Formula for calculating shear bond strength (MPa) = Force (N)/surface area (A) (mm<sup>2</sup>)

$A = \pi r^2$  (A = surface area,  $\pi$  = constant = 3.14, r = radius of the disk is 5 mm)

From the results of Tables 1 and 2, it can be seen that significant difference was observed between the four groups (I, II, III, and IV) with respect to bond strength scores (F = 70.371,  $p < 0.05$ ) at 5% level of significance.

Comparison of the four groups (I, II, III, and IV) with respect to bond strength scores by applying the Tukey's multiple *post hoc* procedures and the results are presented in Table 3.

### DISCUSSION

The demands for ceramic veneers and all-ceramic crowns have also increased. This led to greater use of composite resin cements to provide strength for all-ceramic restorations and ensure adhesion to the tooth. Among ceramics, lithium disilicate glass ceramic core veneered with sintered glass ceramic offers strength high enough for the fabrication of short-span fixed partial dentures. The clinical success of ceramic restoration is also dependent on the luting cement and the cementation.<sup>7</sup>

**Table 2:** Comparison of the four groups with respect to bond strength scores by one-way ANOVA

| Source of variation | Sum of squares | Degree of freedom | Mean square | f-value | Significance |
|---------------------|----------------|-------------------|-------------|---------|--------------|
| Between groups      | 852.696        | 3                 | 284.232     | 70.371  | 0.000*       |
| Within groups       | 145.405        | 36                | 4.039       |         |              |
| Total               | 998.101        | 39                |             |         |              |

\*Statistically significant

**Table 3:** Comparison of the four groups with respect to bond strength scores by applying Tukey's multiple *post hoc* test

|   | Multilink N with lithium disilicate without thermocycling | RelyX U100 with lithium disilicate without thermocycling | Multilink N with lithium disilicate with thermocycling | RelyX U100 with lithium disilicate with thermocycling |
|---|---|--|--|---|
| Shear bond strength                                       |   |  |  |   |
| Mean (MPa)  | 14.6  | 5.4  | 15.5   | 6.3   |
| SD  | 2.37  | 1.01   | 2.89   | 1.03  |
| Multilink N with lithium disilicate without thermocycling |   | p = 0.000*   | p = 0.760  | p = 0.000*  |
| RelyX U100 with lithium disilicate without thermocycling  | p = 0.000*  |  | p = 0.000*   | p = 0.760   |
| Multilink N with lithium disilicate with thermocycling    | p = 0.760   | p = 0.000*   |  | p = 0.000*  |
| RelyX U100 with lithium disilicate with thermocycling     | p = 0.000*  | p = 0.760  | p = 0.000*   |   |

\*Statistically significant

Pressable ceramic used in the present study is IPS e.max Press, which is a lithium disilicate glass ceramic ingot, i.e., heat pressed using the lost wax technique. It is recommended in situations where average to high translucency is needed. Its increased flexural strength makes it suitable in the fabrication of 3-unit fixed partial denture in the anterior region.

The application of dual-polymerizing resin cement for all-ceramic restorations has considerably increased due to the ability of resin cements to polymerize completely and their greater resistance to occlusal loading.

Bond strength of resin cement to ceramic material is influenced by the polymerization mode of the resin luting agents (visible light, dual, or autopolymerizing), thermal cycling and water storage, and the luting agent itself. An adhesive luting agent is desirable when the retentive area is small and the retention may be inadequate. The composition of the material is an important feature in the properties of resin-luting agents. The RelyX U100 is an ethanol-based adhesive, which reacts with hydroxyapatite and also penetrates and modifies the smear layer. Similarly, Multilink N is a phosphoric-based adhesive, which is responsible for the adhesive and self-etching ability. Adhesive luting agents increase the fracture resistance of all-ceramic materials by penetrating into the flaws and irregularities of the restoration's internal surface and inhibiting the crack propagation.

Pekkan and Hekimoglu,<sup>8</sup> in their studies, evaluated the shear and tensile bond strength between dentin and ceramics using dual-polymerizing resin cement. The dual-polymerizing resin cement had higher shear bond strength values than the tensile bond strength values. In this study, thermal cycling was used to simulate the oral environment. Shear stress is considered the ideal representative of typical clinical stress.<sup>9</sup> Clinically, all-ceramic restorations are subjected to masticatory force under dry and wet conditions, and therefore, these conditions were considered in this *in vitro* study.

Samples cemented with Multilink N (group I) had a higher mean shear bond strength value of 14.64 MPa. However, samples when cemented with RelyX (group II) had relatively lower mean shear bond strength values of 5.48 MPa. Similarly, samples when cemented with Multilink N (group III) had a higher mean shear bond strength value of 15.59 MPa and samples cemented with RelyX (group IV) had a relatively lower mean shear bond strength value of 6.36 MPa.

Viotti et al<sup>10</sup> evaluated the bond strength of different self-adhesive cements and compared them with conventional luting cements and found that the bond strength of multistep luting cements was significantly higher than those observed with most of the self-adhesive cements.

The bonding performance of multistep resin cements is mainly dependent on the quality of the hybridization layer, which is established during the pretreatment of dentin. The application mode of the primer is the key factor for receiving high bond strength with Multilink N. It may be interpreted that Multilink N is able to produce high-bond strength, though not reliably.<sup>11</sup> The success in the bonding of conventional resin cement to teeth and ceramic depends on the type of adhesive used (total etch or self-etch) and the quality of the dentin surface pretreatment.

Liu et al,<sup>9</sup> in their studies, justified that self-adhesive cements are not able to completely demineralize/dissolve the smear layer of dentin and that they have significantly lower bond strength than conventional resin cements that require the dentin to be treated with total-etch or self-etch systems.<sup>7</sup> The low bond strength recorded for self-adhesive resin cement is probably related to the cement's limited ability to demineralize and infiltrate dentin substrate.

Lithium disilicate disks, when cemented with Multilink N without thermocycling, had a mean shear bond strength values of 14.64 MPa, and, with thermocycling, had a mean shear bond strength value of 15.59 MPa.

Kamada et al<sup>12</sup> evaluated the effect of various ceramic surface treatments on the shear bond strength of four

resin luting agents to cerec 2 ceramic materials and concluded that the combined surface treatment with etching and silane coupling agent provides the highest bond strengths after thermocycling. Hitz et al<sup>13</sup> proved that slight increase in bond strength values was seen after thermocycling. However, the effect of thermocycling on bond strength of Multilink N and RelyX U100 prior to and after thermocycling was found to be nonsignificant in the present study.

Lithium disilicate disks, when cemented with RelyX U100 without thermocycling, had a mean shear bond strength value of 5.48 MPa and, with thermocycling, had a mean shear bond strength value of 6.36 MPa. Piwowarczyk et al<sup>14</sup> did a study to compare the shear bond strength of cementing agents, namely, zinc phosphate, glass ionomer, resin-modified glass ionomer, resin cements (RelyX ARC, Panavia F, Variolink), and a self-adhesive universal resin cement (RelyX Unicem) to high-gold content alloy casting and different dental ceramics, namely, high-strength aluminum oxide (Procera Allceram), leucite-reinforced (IPS Empress), and lithium disilicate glass ceramic (IPS Empress 2). They found that after 14 days of water storage followed by thermal cycling, only the RelyX Unicem and 2 of the resin cements (Panavia F and compolite) exhibited strong bond strengths to Procera Allceram, IPS Empress, and IPS Empress 2.

The shear bond strength of Multilink N when compared with RelyX was greater with or without thermocycling. The low bond strength recorded for the self-adhesive resin despite the initial low pH and higher viscosity of the self-adhesive cements may explain as to why no true hybrid layer is formed when applied onto dentin. To promote micromechanical interlocking with dentin collagen fibrils, resin cements should etch the substrate in a relatively short time, requiring optimal wetting properties to ensure a fast interaction with dentin. Luting of porcelain veneers with self-adhesive cements, hence, is not recommended by the manufacturers. The bonding strength of self-adhesive cements can be attributed, in part or primarily, to their inability to chemically interact with dentin hydroxyapatite. The above properties contribute to the poor strength of RelyX U100 adhesive cement in the present study.

However, the shear bond strength in the present study shows a strong significant difference between Multilink N and RelyX U100 before and after thermocycling. This indicates that the bond strength of conventional or multistep resin cement luted to lithium disilicate has higher strength when compared with self-adhesive resin cement. The success in the bonding of conventional resin cement to teeth and ceramic depends on the type of adhesive used and the quality of the dentin surface pretreatment. Stewart GP et al,<sup>5</sup> in their study, concluded that specimens

which were sanded or microetched, but had no silane treatment were associated with low bond strengths. However, in the present study, all samples were treated with Monobond S as silane coupling agent.

To obtain reliable mechanical retention between the composite resin cement and ceramic materials, surface roughening of the ceramic restoration is essential. Various measures used to enhance bond strength of resin cement and silica-based ceramics are etching with hydrofluoric acid/or grit blasting, which provides micromechanical attachment and chemical bonding by a silane coupling agent.<sup>9</sup>

Studies by Holderegger et al<sup>11</sup> have confirmed that 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. However, in contrast to Multilink N, RelyX was least influenced by the operator, probably because it uses no priming system, thereby rendering it less technique sensitive.

However, limited information is available as to how the use of different luting agents and cyclic loading fatigue influence the fracture load of recently developed all-ceramic crowns system.<sup>15</sup>

## CONCLUSION

There is a significant difference between the shear bond strength values of conventional resin cement (Multilink N) and self-adhesive resin cement (RelyX U100) when bonded to lithium disilicate. The application of silane coupling agent increases the bond strength of both conventional and self-adhesive resin cements when luted to lithium disilicate disks. The results of this study are applicable to the all-ceramic resin luting system only.

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