SUMMARY

Background/Aim: The aim of this study was to evaluate the bond strength of glass ceramic inlay system using 2 antibacterial adhesive luting protocols with 2 cementation techniques to bur-cut dentin. Material and Methods: Class I inlay cavities with 6-degree occlusal divergence and size of 6-, 3- and 2-mm in length, width and depth, were prepared on extracted human molars, randomly assigned to 2 main groups; each to 1 cementation technique, with or without immediate-dentin-bonding (IDB or NIDB) further divided into 3 subgroups; 2 to 2 antibacterial luting protocols, traditional (T) and experimental (E); and 1 to a control (C) group. In group IDB-T, IDB-E and IDB-C dentin bonding was applied immediately after cavity preparation. In group NIDB-T, NIDB-E and NIDB-C dentin bonding was applied just before cementation of the restorations. The cavities in IDB-T and NIDB-T were treated with 2% chlorhexidine-digluconate (CHX) prior to dentin bonding application. The cavities in IDB-E and NIDB-E were treated only with dentin bonding system containing MDPB (12-methacryloyloxydodecylpyridinium bromide) active monomer featuring antibacterial effect. IDB-C and NIDB-C served as control. Dual-cure adhesive resin cement was used for the cementation of lithium disilicate-based ceramic inlay restorations. Fourteen test specimens per group were prepared for microtensile testing and consecutively subjected to tensile load at a crosshead speed of 1 mm/min. The mode of failure was observed under SEM and evaluated for each group. The Kruskal-Wallis test was used to investigate the statistical difference between groups (α=0.05). Results: The microtensile load was 5.96 MPa (median: 5.99 MPa) for IDB-T, 7.23 MPa (median: 7.55 MPa) for IDB-E, 6.68 MPa (median: 6.56 MPa) for IDB-C, 7.24 MPa (median: 7.20 MPa) for NIDB-T, 6.98 MPa (median: 6.30 MPa) for NIDB-E, and 7.02 MPa (median: 6.99 MPa) for NIDB-C, with no statistical difference between the groups (p>0.05). SEM monitoring for mode of failure revealed either cohesive (within resin cement) or adhesive-cohesive (mostly within resin cement along with partially involved areas between resin cement and ceramic restoration) character. Conclusions: Within the limitations of the current study, none of the tested antibacterial luting protocols with either cementation technique was found to be superior in terms of bond strength.

Key words: Inlays, Glass Ceramics, Immediate-Dentin-Bonding, Microtensile Bond Strength, Antibacterial Cleansing Effect

Effect of Two Antibacterial Luting Protocols with and without Immediate-Dentin-Bonding on Microtensile Bond Strength of Glass Ceramic to Bur-Cut Cavity Floor Dentin

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Introduction

The demand for aesthetic restoration has increased the interest in metal-free tooth-coloured restorations as stronger and tougher materials have been developed along with advanced processing technologies. Direct composites have generally been preferred over indirect esthetic restoratives as their application requires minimal intervention and cavity preparation. However, indirect restorations are usually recommended whenever teeth require larger restorations.

Multiphase leucite-reinforced and lithium disilicate-based glass-ceramics are of the frequent materials of choice for rehabilitation with indirect restorations. The benefits of these all-ceramic materials are pronounced in terms of machinability, polishability, reduced wear of opposing tooth structure, increased biocompatibility, superior natural appearance and excellent aesthetics with the advantages of ease of fabrication techniques.

Aesthetic results and longevity of these all-ceramics critically depend on the cementation procedure as it involves the application of adhesive systems and resin-based luting agents. When bonding ceramic to tooth structure dentin-adhesive interface and cement-ceramic interface need to be considered as two different interfaces on which the restoration relies to remain in place. The bond strength of both of these interfaces should be optimized, because the lower one is to determine the final bond strength of the cemented restoration.

The adhesion between dental ceramics and composite resin cements can be significantly increased as a result of physicochemical interaction across the interface involving the adhesive and the ceramic surface obtained by micromechanical retention provided by hydrofluoric acid etching of the lithium disilicate-based ceramic followed by chemical coupling of the etched surface by the application of a silane coupling agent.

The bonding performance between dentin and resin luting materials can also be dramatically improved by incorporating an adhesive bonding system in the cementation protocol of the indirect restorations prior to application of the composite resin cement.

However, rehabilitation with indirect restorations in cases with caries and/or deep dentin preparations will increase the need to treat the cavities with an antibacterial agent prior to restoration placement. Furthermore, open dentin tubules will need to be preserved from the potential negative effect of the external environment until the cementation of the indirect restoration.

It has been advocated that the bonding strength of restorations to dental hard tissues may decrease depending on specific combinations between traditional antibacterial agents and adhesive bonding systems. Also, recently developed adhesive system with self-antibacterial activity as effective as the traditional cavity disinfectants has been reported to provide high bond strength to dental hard tissues.

Immediate-dentin-bonding (IDB), also called immediate-dentin-sealing or resin-coating, expressed by using dentin bonding agent to seal freshly cut dentin prior to definitive impression making in order to protect the dentinal tissue from bacterial infiltration and saliva leakage during the provisionalization period, has yielded improved bond strength.

The aim of this in vitro study was to comparatively evaluate the microtensile bond strength (MTBS) of glass ceramic inlay system to bur-cut dentin using luting protocol involving an adhesive system featuring antibacterial properties or a traditional cavity disinfectant combined with a regular adhesive system with and without IDB cementation technique. The null hypothesis was that there would be no difference in MTBS between the different antibacterial luting protocols with different cementation techniques.

Material and Methods

A priori power-analysis was conducted by using statistical software (GPower 3.1; Heinrich Heine University, Dusseldorf, Germany) to detect the number of specimens (n) for 80% statistical power of the test results. Referencing a recent study with matching material and methods, n was calculated to be at least 14 for 1-β=0.80 with type I error at α=0.05.

Twenty impacted full-grown human third molar teeth with intact integrity were used for the study within strict compliance with the Helsinki Declaration Principles. The teeth were cleaned, embedded up to 1-2 mm from cervical line into a hard dental stone flat-base, and stored in saline solution under room temperature conditions.

The occlusal aspect of the teeth was flattened to the central fossa perpendicular to the long axis, and standardized Class I inlay cavity preparations with 6-degree occlusal divergence and size of 6-, 3- and 2-mm in length, width and depth, respectively (Figure 1), were made using diamond burs in a high-speed handpiece under water spray cooling. Cavity preparation procedures were all done by an experienced Prosthodontist consecutively at one go, after pre-exercising on 2 molars for self-calibration. After preparation, the remaining 18 molars were randomly assigned to 2 main groups of 9; each to 1 cementation technique, with or without IDB (IDB or NIDB). Each of the groups was further divided into 3 subgroups of 3; 2 to 2 different antibacterial luting protocols, traditional (T) and experimental (E); and 1 to a control (C) group. All test groups were then prepared as follows.
The cavities in group IDB-T and NIDB-T were treated with traditional cavity disinfectant (Cavity Cleanser; Bisco, Schaumburg, IL, USA) containing 2% chlorhexidine-digluconate (CHX) for 60 seconds and dried prior to dentin bonding application. A regular 2-step self-etch adhesive bonding system (Clearfil SE Bond; Kuraray, Okayama, Japan) was immediately applied on the cavity walls according to the manufacturer’s instructions.

The cavities in group IDB-E and NIDB-E were not treated with separate cavity disinfectant, but only with a 2-step self-etch adhesive bonding system (Clearfil Protect Bond; Kuraray) according to the manufacturer’s instructions, containing MDPB (12-methacryloyloxydodecylpyridinium bromide) active monomer with antibacterial cavity cleansing effect directly in its chemical structure along with fluoride-releasing component.

The cavities in group IDB-C and NIDB-C were treated only with the regular self-etch adhesive bonding system (Clearfil SE Bond; Kuraray) that was used in group IDB-T and NIDB-T according to the manufacturer’s instructions. Each of these subgroups served as control for its corresponding main group; with or without IDB cementation technique.

Two weeks after the preparation of the inlay cavities the temporary fillings were removed and the cavity surfaces were wiped with ethanol-soaked cotton pellet. The internal surfaces of the inlay ceramic restorations were treated with 35-45% phosphoric acid gel (K-etchant Gel; Kuraray) and silane coupling agent (Clearfil Porcelain Bond Activator; Kuraray) according to the manufacturer’s instructions. Dual-cure adhesive resin cement (Panavia F 2.0 Light; Kuraray) was applied on the cavity walls and the restorations were fitted into their corresponding teeth. The excess cement was removed and polymerization accomplished by using LED (light-emitting diode) light for 20 seconds, upon the occlusal aspect of every tooth.

After being kept for 1 week in 100% aqueous environment at room temperature, the cemented restorations along with the teeth were sectioned by using a slow-speed diamond saw precision cutting machine (IsoMet 1000; Buehler, Lake Bluff, IL, USA) in order to prepare the test specimens for microtensile testing according to previous studies. Vertical sections were made, both at mesio-distal and bucco-lingual directions along the long axes of the teeth so that to fabricate I-shaped bars with 1.37 × 1.26 mm rectangular cross section, consisting of ceramic-top and dentin-bottom halves, and a luting agent junction in between. Fourteen test specimens (n=14/group) were randomly selected among the I-shaped bars within each test group. The assembled specimens for all groups (IDB-T, IDB-E, IDB-C, NIDB-T, NIDB-E, and NIDB-C) were then consecutively subjected to tensile load by using a testing...
The microtensile load was 5.96 MPa (median: 5.99 MPa) for IDB-T, 7.23 MPa (median: 7.55 MPa) for IDB-E, 6.68 MPa (median: 6.56 MPa) for IDB-C, 7.24 MPa (median: 7.20 MPa) for NIDB-T, 6.98 MPa (median: 6.30 MPa) for NIDB-E, and 7.02 MPa (median: 6.99 MPa) for NIDB-C (Figure 5). The calculated microtensile load mean values and standard deviations, median values, detected minimum and maximum range values are presented in detail for each group in Table 1. The Kruskal-Wallis test revealed that there was no statistical difference between any of the tested groups ($p>0.05$) (Table 2).

Table 1. Microtensile load mean values and standard deviations, median values, minimum and maximum range values for tested groups with different luting protocols (MPa)

| Group   | n  | Mean | Std. D. | Min. | Max. | Median |
|---------|----|------|---------|------|------|--------|
| IDB-T   | 14 | 5.96 | 1.34    | 3.58 | 7.76 | 5.99   |
| IDB-E   | 14 | 7.23 | 2.98    | 2.85 | 10.84| 7.55   |
| IDB-C   | 14 | 6.68 | 2.07    | 4.13 | 9.71 | 6.56   |
| NIDB-T  | 14 | 7.24 | 2.07    | 4.30 | 10.88| 7.20   |
| NIDB-E  | 14 | 6.98 | 2.46    | 4.18 | 11.09| 6.30   |
| NIDB-C  | 14 | 7.02 | 2.33    | 3.18 | 11.09| 6.99   |

Table 2. Kruskal-Wallis results of microtensile loading for tested groups with different luting protocols ($p>0.05$)

| Group   | n  | Mean Rank | df | $\chi^2$ | P   |
|---------|----|-----------|----|----------|-----|
| IDB-T   | 14 | 34.43     | 5  | 2.384    | 0.794|
| IDB-E   | 14 | 44.71     |    |          |     |
| IDB-C   | 14 | 40.86     |    |          |     |
| NIDB-T  | 14 | 47.50     |    |          |     |
| NIDB-E  | 14 | 44.21     |    |          |     |
| NIDB-C  | 14 | 47.29     |    |          |     |
SEM monitoring for mode of failure of the specimens revealed either cohesive in nature character within resin cement (Figure 6), or adhesive-cohesive (mixed) in nature character mostly within resin cement along with partially involved areas at the junction between resin cement and ceramic restoration (Figure 7). Hybrid layer was also involved with the cohesive failure mode in some specimens but only for NIDB subgroups (Figure 8).

**Discussion**

In the current study, the MTBS results for the tested group pairs were found to be statistically insignificant. Therefore, the null hypothesis that there would be no difference in MTBS between the different antibacterial luting protocols with different cementation techniques failed to be rejected.

According to the results of the present study none of the tested adhesive luting protocols could demonstrate superiority among each other in terms of dentin bond strength. Okuda et al. investigated deep dentin bond strength of indirect restorations and reported similar results for the adhesive system with self-antibacterial activity in combination of the resin cement used in
this study, regarding both cementation techniques. De Castro et al.25 showed that combination of the traditional antibacterial agent and the regular adhesive system used in the current study did not have significant effect on the MTBS to dentin. On contrary, Magne et al.23 found IDB to significantly affect the MTBS of indirect restorations luted to superficial dentin using the regular adhesive system alone as in the control groups of this study, reporting nearly 8-fold higher bond strength values. This much difference in the results and the overall miscorrelation of the present study with that of Magne et al.23 might be attributed to the fact that Class I cavity configuration along with the presence of deep dentin may result in lower bond strengths to the cavity floor26,27.

The failure mode results observed in this study are in consistency with the previous literature21,23,28. Okuda et al.21, Magne et al.23 and Murata et al.28 also reported similar cohesive fracture character for their tested groups with and without IDB. The modes of failure of the specimens in the present study suggest that resin cement may be the weakest link for this choice of materials and methods of their application.

Due to variations in materials and methods the direct comparison of the results of the present study to previous ones was difficult.

IDB technique enables coverage and protection of open dentin tubules on cut dentin immediately after cavity preparation21. The sealed dentin gets protected from infiltration and leakage of bacteria until the delivery of indirect final restoration, thus, enhances patient comfort pre- and post-cementation as a result of partial or complete reduction of post-operative sensitivity23.

Using separately antibacterial agent and adhesive bonding system prolongs the cementation procedure and makes it more technique sensitive. Furthermore, the antibacterial reliability of a CHX agent is expected to drop significantly in the area of its application in a short while7,17. However, the MDPB monomer featuring antibacterial properties remains active within the resin material even after polymerization29, in that for years30. Additionally, existence of a fluoride-releasing component in the structure of adhesive system may also inhibit bacterial growth and promote remineralisation in dentinal tissue surrounding cavity area31.

In the light of the evidence, choosing to use the adhesive system with self-antibacterial activity along with IDB cementation technique for the cementation of the tested glass ceramic inlay system seems to be more favourable.

The microtensile test method was used to evaluate dentin bond strength of the specimens in this study. This test method can provide closer loading results in respect to the true ultimate strengths of the materials being tested24. However, microcracks and other defects can possibly occur during the production of specimens by using slow-speed diamond saw precision cutting machine, which may cause premature failure of the bond24. Therefore very careful specimen preparation protocol is crucial24.

The results of this study should be interpreted considering the used brands of tested materials along with their methods of application under controlled in vitro conditions. The results should be corroborated with clinical studies.

**Conclusions**

Within the limitations of this in vitro study, the following conclusions were drawn; The antibacterial luting protocol involving either an adhesive system featuring antibacterial properties or a traditional cavity disinfectant combined with a regular adhesive system did not have an influence on the MTBS of the used glass ceramic inlay system to bur-cut dentin. The IDB cementation technique did not have an influence on the MTBS with the currently used luting materials and glass ceramic inlay system.

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