

Microleakage of IPS Empress 2 Inlay Restorations Luted With Self-adhesive Resin Cements

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Clinical Relevance

Although self-adhesive resin cements reduce the duration of the luting process, their universal practice requires caution because the microleakage performance of some brands is very poor.

SUMMARY

Objective: To assess the microleakage of three self-adhesive and one etch-and-rinse resin cements when luting IPS Empress 2 (Ivoclar Vivadent, Liechtenstein) all-ceramic inlay restorations to the prepared cavities in extracted human molars.

Methods: The cylindrical Class V cavities were prepared on the buccal surfaces of 40 extracted

human third molars using diamond burs. The IPS Empress 2 ceramic inlays were placed with Multilink Sprint (Ivoclar Vivadent), RelyX Unicem (3M ESPE, USA), G-Cem (GC, Japan), or Variolink II (Ivoclar Vivadent) as the control group. After storage in distilled water at 37°C for 24 hours, samples were subjected to 1000 thermal cycles between baths of 5°C and 55°C, with a dwell time of 30 seconds. The microleakage scores were examined on the occlusal and gingival margins at 30× magnification after each sample was stained with 0.5% basic fuchsin and sectioned into three parts using a thin diamond blade (Isomet, Buehler, USA) (n=40). The extent of microleakage on both occlusal and gingival margins of the restorations was scored and recorded. The microleakage data were analyzed using Kruskal-Wallis and Mann-Whitney U-tests.

Results: Statistically significant differences were observed between the groups in both

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margins according to the Kruskal-Wallis and Mann-Whitney U-tests ($p < 0.05$). Microleakage scores on the occlusal margins were Variolink II < RelyX Unicem < G-Cem = Multilink Sprint. Microleakage scores on the gingival margins are Variolink II = RelyX Unicem < G-Cem < Multilink Sprint.

Conclusion: Self-adhesive resin cements displayed higher microleakage scores on the occlusal margins, whereas on the gingival margins RelyX Unicem showed comparable microleakage results with the control samples.

INTRODUCTION

Increasing esthetic demands have resulted in the development of tooth-colored restorative materials. Among these materials, glass ceramic inlay techniques have some advantages such as satisfactory physico-chemical properties, abrasion resistance, and color retention compared with the composite resins that have led to some problems when used in stress-bearing areas of the mouth.¹ IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein) glass ceramic is a heat-pressed, lithium disilicate-reinforced material using the "lost-wax" technique. This all-ceramic material has been introduced for single-unit restorations as well as for three-unit fixed partial dentures in the anterior region extending to the second premolar. The final restoration, made of a lithium disilicate-framework ceramic, offers clinical benefits in terms of machinability, polishability, and reduced wear of opposing tooth structure, with the advantages of increased biocompatibility, natural appearance, and superior esthetics.²⁻⁴

Marginal adaptation of ceramic inlays is one of the important features that could influence the durability of these restorations.^{5,6} Microleakage at the interface between the teeth and the restorative material still remains a problem, and unfortunately, no technique is available that completely eliminates this leakage.

Ceramic inlays are generally cemented using resin-based cements because these cements generally display superior mechanical properties^{7,8} and can increase the retention⁹ and fracture resistance of overlying ceramic materials.¹⁰ In addition, the flexural strengths of resin cements are higher than glass ionomer and resin-modified glass ionomer cements; thus, they are especially recommended for high-strength clinical situations.^{8,11} To date, a number of resin-based luting cements have been introduced by different manufacturers. Various

pretreatment steps to prepare the tooth's surface are necessary prior to the use of the resin cements to achieve adhesion to dentin. Depending on the type of luting cement, these steps include etching with phosphoric acid, priming, and bonding. In order to obtain optimal adhesive cementation, use of dentin bonding agents prior to the cementation of inlays is gaining wider acceptance. In previous studies, acceptable results were obtained with the bonding strategies described above.^{12,13} However, the clinical application of these steps is time consuming and technically sensitive. Thus, cements that form a comparable adaptation but avoid the complex bonding procedures are desirable.

Recently, new resin-based systems, named *self-adhesive* resin cements, have been put on the market. These cements were developed to meet the demand among dentists for luting agents that offer easy, quick, and universal application. During the application of these materials, no conditioners or bonding agents need to be applied to the dental hard tissues. Bonding values and mechanical properties of these products are claimed to be comparable to those of similar products currently available by the manufacturer.^{14,15}

The purpose of this *in vitro* study was to compare the adaptation of three self-adhesive and one etch-and-rinse resin cements on the microleakage of IPS Empress 2 all-ceramic inlay restorations by a dye penetration method.

METHODS AND MATERIALS

A total of 40 extracted, caries-free human third molars were used in this study. Immediately after extraction, the teeth were scraped of any residual tissue tags, pumiced, and washed under running tap water. The teeth were stored in distilled water at +4°C until required, a period not exceeding one week. Standardized, nonbeveled cylindrical Class V cavities were prepared on the buccal aspects of each tooth with round internal angles, 1 mm below the cemento-enamel junction, using cylindrical (3.8 mm in diameter and 1.8 mm in length) (041-038C, MDT Micro Diamond Technologies Ltd, Afula, Israel) and 6° conical diamond burs (702.8KR, Abrasive Technology, London, UK). Standardization of the cavity size was accomplished by using the cylindrical diamond burs in similar dimensions with the prepared cavities and using the hand piece in a parallelometer during preparation. After preparation, the teeth were randomly divided into four groups. The bonding agents and resin cements used in this study are described in Table 1.

Table 1: The Bonding Agent and Resin Cements Used in This Study

Cement	Variolink II	Multilink Sprint	RelyX Unicem	G-Cem
Type	Etch-and-rinse resin cement	Self-adhesive resin cement	Self-adhesive resin cement	Self-adhesive resin cement
Etching	Scotchbond Etchant	—	—	—
Priming	Syntac primer	—	—	—
Bonding	Syntac adhesive	—	—	—
Manufacturer	Ivoclar Vivadent, Liechtenstein	Ivoclar Vivadent, Liechtenstein	3M ESPE, USA	GC, Japan
Mixing	Hand	Automix syringe	Hand	Capsule
Active components	Base paste: Bis-GMA, UDMA, TEGDMA, filler	Base paste: Dimethacrylates, ytterbium trifluoride, glass filler, silicon dioxide, initiators, stabilizers and pigments	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers	Powder: Fluoro-alumino-silicate glass, initiator, pigment
	Catalyst paste: Bis-GMA, UDMA, TEGDMA, filler	Catalyst paste: Dimethacrylates, ytterbium trifluoride, glass filler, silicon dioxide, adhesive monomer, initiators, stabilizers and pigments	Catalyst paste: Methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments	Liquid: Urethane dimethacrylate, dimethacrylate, 4-Methacryloyloxyethyl trimellitate, distilled water, phosphoric acid ester monomer, silicon dioxide, initiator, inhibitor
Definitions: Bis-GMA, bisphenol-glycidyl methacrylate; TEGDMA, triethylene-glycoldimethacrylate; UDMA, urethane dimethacrylates.				

Impressions were made with polyvinyl siloxane material (Imprint II VPS, 3M ESPE AG, Seefeld, Germany) and poured into a vacuum-mixed polyurethane die material (Alpha Die MF, Schültz-Dental GmbH, Rosbach, Germany) according to the manufacturer's instructions. IPS Empress II ceramic inlays were fabricated according to the manufacturer's instructions and then glazed.

Ceramic inlays were etched with the hydrofluoric acid for 20 seconds, and then a layer of silane coupling was applied to the ceramic bonding surface for 60 seconds and air-dried.

Products used for cementation were as follows: in group 1, Multilink Sprint (Ivoclar Vivadent, Schaan, Liechtenstein); in group 2, G-Cem (GC, Tokyo, Japan); in group 3, RelyX Unicem (3M ESPE AG) self-adhesive resin cements; and in group 4, Variolink II (Ivoclar Vivadent) etch-and-rinse resin cement. All resin cements were applied to the

diamond bur-prepared dentin surface with smear layer; however, self-adhesive luting agents were applied without separate dentin conditioning.

The cavities of self-adhesive resin cement groups were cleaned with fluoride-free pumice and water after cavity preparation. In groups 1 and 2, the cavities were rinsed with water and air-dried for two to four seconds to remove excess moisture, leaving the dentin surface with a slightly glossy appearance before cementation with Multilink Sprint and RelyX Unicem.^{16,17} In group 3, the cavity surfaces were rinsed with water and gently dried by blowing with oil-free air until the prepared surfaces appeared dry before cementation with G-Cem.¹⁸ After tooth preparation, the self-adhesive resin cements were applied to the cavities. Inlay restorations were seated into the cavities using light pressure and cured briefly (one to two seconds) with light. Then the excess material was removed using a scaler. The

restoration margins were covered with glycerin gel. The luting agents were light-cured for 20 seconds. The light activating unit was Optilux 501 (Kerr, Orange, CA, USA), which was tested prior to each sample. The output of this unit did not drop below 500 mW/cm². After the final polymerization the glycerin gel was rinsed off.

In group 4, the enamel and dentin margins of inlay cavities were first cleaned with fluoride-free pumice and water and then etched with a phosphoric acid gel (Uni-etch, Bisco, IL, USA) for 15 seconds. They were rinsed thoroughly with water for approximately five seconds and air-dried for two to four seconds to remove excess moisture, leaving the dentin surface with a slightly glossy appearance.¹⁹ One drop of the primer (Syntac, Ivoclar Vivadent) was applied to the cavity surfaces for 15 seconds and gently air-dried. A layer of bonding resin (Syntac, Ivoclar Vivadent) was applied with a brush for 10 seconds, spread gently with air. After application of enamel bonding agent (Heliobond, Ivoclar Vivadent) to the cavity and the bonding surface of the ceramic inlay restorations, the cavities were filled with Variolink II (Ivoclar Vivadent) and inlays were placed into the cavities using light pressure and cured briefly (one to two seconds) with light. Then the excess material was removed using a scaler. The restoration margins were covered with glycerin gel. The resin cement was light-cured for 40 seconds. After final polymerization the glycerin gel was rinsed off.

Excess material was removed with finishing diamond burs and flexible discs. The restoration margins were finished with silicone polishers (Astropol F and Astropol P, Ivoclar Vivadent).

After cementation, specimens were stored in distilled water at 37°C for 24 hours and then subjected to 1000 thermal cycles between baths of 5°C and 55°C, with a dwell time of 30 seconds. The teeth were subsequently coated with nail varnish 1 mm short of the restoration margins to seal open dentin tubules. The dye penetration test was conducted in a 0.5% basic fuchsin dye solution for 24 hours. The teeth were then rinsed, and Class V restorations were sectioned into three parts longitudinally in a bucco-lingual plane with a slow-speed diamond blade (Struers, Ballerup, Denmark). The sections from the centers of the restorations were 2-mm thick, whereas the other sections were 1-mm thick. In this way, four surfaces (either mesial or distal surfaces of 1-mm-thick samples and both mesial and distal surfaces of 2-mm-thick samples) were obtained from one restoration for microleakage

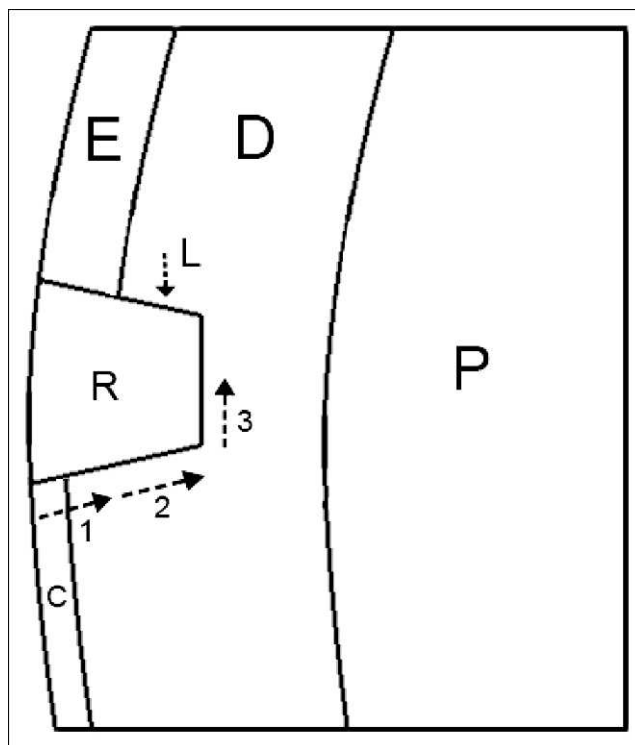


Figure 1. Dye penetration between restoration and tooth. E, enamel; D, dentin; C, cementum; R, inlay restoration; L, luting cement; P, pulp.

evaluation. A total of 160 surfaces (n=40) were evaluated at 30× magnification under a stereomicroscope (Olympus Co, Tokyo, Japan) by two examiners who were calibrated prior to the study.

The extent of the microleakage was scored according to the following criteria (Figure 1):

- 0 = no leakage visible;
- 1 = penetration of dye along the cavity wall, but less than half the length;
- 2 = penetration of dye along the wall, but short of the axial wall;
- 3 = penetration of dye to and along the axial wall.

Leakage scores at occlusal and gingival margins for each group were compared with the nonparametric Kruskal-Wallis test. Multiple comparisons were performed as pairwise comparisons using the Mann-Whitney U-test. Significance was considered at the 0.05 level.

RESULTS

The microleakage scores are shown in Table 2. The mean and median dye penetration scores, standard deviation, minimum, maximum, and interquartile range values are shown in Table 3. According to the

Table 2: Frequency of Microleakage Scores in Study Groups, n (%)

	Occlusal Margin				Gingival Margin			
	0	1	2	3	0	1	2	3
Variolink II	18 (45)	13 (32.5)	7 (17.5)	2 (5)	24 (60)	8 (20)	6 (15)	2 (5)
Multilink Sprint	4 (10)	18 (45)	12 (30)	6 (15)	1 (2.5)	6 (15)	13 (32.5)	20 (50)
RelyX Unicem	12 (30)	13 (32.5)	11 (27.5)	4 (10)	22 (55)	10 (25)	5 (12.5)	3 (7.5)
G-Cem	6 (15)	10 (2.5)	16 (40)	8 (20)	15 (37.5)	9 (22.5)	11 (27.5)	5 (12.5)

Kruskall-Wallis and Mann-Whitney U-tests, there were significant differences between the groups in both margins ($p < 0.05$).

Nearly half of the Variolink II samples (45%) revealed no visible leakage at the occlusal margins, followed by 30% of RelyX Unicem, 15% of G-Cem, and 10% of Multilink Sprint samples. Only 20% or less of all groups showed dye penetration along the axial wall at the occlusal margin. Variolink II samples showed the lowest leakage scores at the occlusal margins ($p < 0.05$). RelyX Unicem's leakage score was higher than that of Variolink II ($p < 0.05$), whereas it was lower than those of the Multilink Sprint and G-Cem samples ($p < 0.05$). There were no statistically significant differences between the occlusal margin leakage scores of G-Cem and Multilink Sprint samples.

More than half of the Variolink II (60%) and RelyX Unicem (55%) samples revealed no visible leakage at the gingival margins, followed by 37.5% of G-Cem and 2.5% of Multilink Sprint samples. Half of the

Multilink Sprint samples (50%) showed dye penetration along the axial wall at the gingival margin, whereas the ratio of score 3 was under 13% for the other tested agents. Multilink Sprint samples showed the highest leakage scores at the gingival margins ($p < 0.05$). G-Cem's leakage score was lower than that of the Multilink Sprint samples ($p < 0.05$), whereas it was higher than that of the RelyX Unicem and Variolink II samples ($p < 0.05$). There were no statistically significant differences between the gingival margin leakage scores of the RelyX Unicem and Variolink II samples.

There were no significant differences between the microleakage at the enamel and dentin margins in Variolink II samples. The microleakage at the enamel margins was greater than that at the dentin margins in the RelyX Unicem and G-Cem samples ($p < 0.05$), whereas the microleakage at the dentin margins was greater than that at the enamel margins in the Multilink Sprint samples ($p < 0.05$).

Table 3: Descriptive Analysis of Study Groups

	Occlusal Margin						Gingival Margin					
	Mean	SD	Median	Min	Max	Interquartile Range	Mean	SD	Median	Min	Max	Interquartile Range
Variolink II	0.83	0.903	1	0	3	1	0.65	0.921	0	0	3	1
Multilink Sprint	1.5	0.877	1	0	3	1	2.30	0.823	2.5	0	3	1
RelyX Unicem	1.18	0.984	1	0	3	2	0.73	0.96	0	0	3	1
G-Cem	1.65	0.975	2	0	3	1	1.15	1.075	1	0	3	2

Abbreviations: Max, maximum; Min, minimum; SD, standard deviation.

DISCUSSION

Different techniques have been described for studies of margin quality. The most widely accepted method is the dye penetration test.²⁰ In our study, 0.5% basic fuchsin solution was used for the dye penetration test. All restorations had previously undergone thermal cycling in order to subject the restorations to thermal expansion and contraction challenges. The different thermal expansion coefficients of tooth tissue from the restorative materials may lead to gap formation.²¹ As such, to assess the *in vitro* performance of resin materials, thermal cycling is the common method used to simulate the long-term stresses to which the resin restorations are exposed.²²

In the present study, the performance of self-adhesive resin cements on the microleakage of IPS Empress II inlays was evaluated in Class V cavities. The reason for studying Class V cavities was that 1) Class V cavities have unfavorable configuration factor resulting in high contraction scores within an adhesively fixed resin material, 2) Class V restoration margins are located in enamel as well as in dentin, 3) preparation and restoration of Class V lesions are minimal and relatively easy, thereby somewhat reducing practitioner variability, and 4) it is easier to standardize the preparation of Class V cavities than Class II cavities.^{23,24}

An essential requirement for a perfect restoration is the perfect adhesion to the tooth surface.²⁰ The adhesion of ceramic restorations to tooth surface depends on the properties of the dental cements and their bonding procedures. The tooth pretreatment with acid, primer, and bonding is very important for resin cement because these procedures affect the condition of the dentin and the smear layer.

Self-adhesive resin cements without pretreatment with acid, primer, and bond demonstrated higher microleakage scores than etch-and-rinse resin cement at the occlusal margins. In addition, the microleakage at the enamel margins was greater than that at the dentin margins in the RelyX Unicem and G-Cem samples. The greater leakage at the occlusal margins with self-adhesive resin cements may be attributed to their insufficient ability to etch the smear layer-covered enamel surface and lack of development of adequate micromechanical retention. High viscosity that the cements have after mixing and the short interaction time that they have with the tooth surface before light-curing may be the reasons for the inadequate micromechanical retention on enamel. The initial low pH may not be

sufficient to etch the enamel when the etching time is not adequate or when the neutralization reactions take place rapidly. The use of resin cement including a separate acid application step prior to the adhesive application results in good micromechanical retention because it adequately etches enamel.²⁵ Previous *in vitro* studies also reported that the etching potential of the self-adhesive cements was lower than that of resin cements applied by a phosphoric acid.^{26,27} Moreover, significantly better marginal adaptation in enamel margins was indicated with resin cements when compared with self-adhesive ones.^{28,29}

Similar to enamel margins, Variolink II showed good performance at dentin margins. However, varying results were obtained at dentin margins with self-adhesive cements, similar to the previous findings.^{29,30} RelyX Unicem's leakage score was similar to that of Variolink II, while it was lower than those of the Multilink Sprint and G-Cem samples. The results of a majority of previous studies were consistent and demonstrated that RelyX Unicem performed comparably to other multistep resin cements on dentin.^{31,32} The authors reported that the specific multifunctional phosphoric-acid methacrylates of this cement react with the tooth surface in multiple ways, resulting in an effective seal. Besides the formation of complex compounds with calcium ions, different kinds of physical interactions such as hydrogen bonding or dipole-to-dipole interactions were supposed to favorably affect the adhesion of this self-adhesive cement.³³

The different microleakage results of the self-adhesive cements may be explained by their different functional monomers and different chemical compositions. Differences in pH values may affect the etching ability of these cements to enamel and dentin, resulting in less than ideal adhesion with subsequent microleakage. G-Cem includes 4-methacryloxy-ethyl trimellitate anhydride, which bonds by a chelating reaction to calcium ions in apatite.³⁴ In addition, this cement was applied to the dry dentin surfaces, contrary to the other cements tested, because it is a water-based system and these systems require drier dentin surfaces for improved adhesion.³⁵ However, its leakage scores were higher than those of RelyX Unicem. The relatively weak bonding potential and the high molecular weight of the functional monomer may be the reason for the failure of the supposed chemical reaction within a clinically reasonable time.³⁶

Multilink Sprint showed the worst performance at the dentin margins. In addition, the microleakage at

the dentin margins was greater than that at the enamel margins in Multilink Sprint samples. The reason for this result may be the mild discrepancy between demineralization and the infiltration depths recorded for Multilink Sprint by Monticelli and others,³⁰ resulting in a deeper diffusion of noncured, nonneutralized acidic monomers. These residual monomers may retain their etching potential, forming an unprotected dentin zone and jeopardizing adhesion.³⁷

CONCLUSION

Within the limitations of this *in vitro* study, self-adhesive resin cements displayed higher microleakage scores on the occlusal margins than the control (Variolink II) group. On the gingival margins, RelyX Unicem showed comparable microleakage results with the control group.

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