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Microleakage of All-ceramic Crowns Using Self-etching Resin Luting Agents

CP Trajtenberg • SJ Caram • S Kiat-amnuay

Clinical Relevance

Among the self-adhesive resin cements, Panavia F 2.0 demonstrated less microleakage than RelyX Unicem or Multilink, whether or not a die spacer technique was used.

SUMMARY

Self-etching adhesive systems are a new generation of materials that possess acidic methacrylates that can generate self-adhesion. There is limited data reported on the marginal leakage of ceramic restorations bonded with self-etching adhesive materials.

This study assessed and compared the amount of microleakage of bonded ceramic crowns using three different types of self-etching adhesive systems with and without a die spacer.

Eighteen human molars were prepared for all-ceramic IPS Empress crowns and the teeth were randomly assigned to each experimental group. The buccal side had the preparation finish line 1.5 mm below the CEJ, and the lingual finish line was 1.5 mm above the CEJ, creating margins in enamel and dentin. Two die-spacing techniques were used (three layers or no layer of die spacer). Each crown restoration was cemented with one of three self-etching resin luting agents (Panavia F 2.0, Multilink and RelyX Unicem). The specimens were thermally cycled for 1000 cycles, then immersed in a 5% methylene blue dye solution for 24 hours. The teeth were then rinsed, embedded in clear epoxy resin and sectioned. A total of 60 sections were evaluated for each type of resin luting agent using digital image analysis at 70x magnification. A novel formula, using mean percentage of microleakage, was developed by dividing the extent of dye penetration along the tooth/resin luting cement interphase and the total perimeter of the tooth crown surface. The data were analyzed using three-way analysis of variance at the 0.05 level of significance. Fisher's PLSD intervals were calculated for comparing significant means.

Panavia F 2.0 showed a lower degree of microleakage than RelyX Unicem and Multilink

*Cynthia Paulina Trajtenberg, DDS, MS, assistant professor, Department of Restorative Dentistry and Biomaterials, The University of Texas Dental Branch at Houston, Houston, TX, USA

Santiago J Caram, DDS, MS, former resident, Department of Prosthodontics, The University of Texas Dental Branch at Houston, Houston, TX, USA

Sudarat Kiat-amnuay, DDS, MS, assistant professor, Department of Restorative Dentistry and Biomaterials, The University of Texas Dental Branch at Houston, Houston, TX, USA

*Reprint request: 6516 M D Anderson Blvd, Suite #493, Houston, TX 77030, USA; e-mail: Cynthia.Trajtenberg@uth.tmc.edu

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at both the enamel and dentin margins. Interactions of the main effects (cement, margin and die spacer technique) were all highly significant ($p \le 0.004$). The degree of microleakage was higher on the dentin margins than on the enamel margins ($p < 0.0001$). The degree of microleakage for the die spacer group was not significantly different from the group with no die spacer technique ($p > 0.1$).

Overall, Panavia F 2.0 showed the least microleakage, followed by RelyX Unicem and Multilink, respectively.

INTRODUCTION

An alternative to metal-ceramic restorations is the all-ceramic restoration. The use of all-ceramic restorations has substantially increased over the last 20 years for the restoration of anterior and posterior teeth. This increase has been attributed to improvements in the physical properties and handling of these ceramic materials, as well as the development of a newer generation of ceramic bonding systems.

IPS Empress (Ivoclar Vivadent, Amherst, NY, USA) was introduced to the public in the 1990s. Since then, it has been a reliable, highly esthetic restorative material. A feldspar porcelain, consisting of 63% SiO_3 and 18% Al_2O_3, is used as the basic material. In addition, leucite crystals are used as the crystalline part of the ceramic.[1-5] The gap between the ceramic restoration and tooth surface is filled with a resin luting agent that holds the restoration to the tooth through a mechanism of bonding.[6]

Adhesion to a ceramic surface is based on the combined effects of micromechanical interlocking and chemical bonding. Hydrofluoric acid is mainly used to increase the surface area and create microporosities within the glass phase of the ceramic substrate. Chemical bonding is achieved by the use of a silane agent through a bifunctional coupling molecule. One end of the molecule bonds to the hydrolyzed silicon dioxide at the ceramic surface, while the other end bonds to a methacrylate group that copolymerizes with the adhesive resin.[7-8] When conventional resin luting agents are used, adhesion to tooth structure is generated by the use of phosphoric acid[6] followed by the application of adhesive monomers.[9-11]

The application of a separate acid-etching step is unnecessary when using self-etching resin luting agents. These materials have become popular for their simplicity and because they require fewer procedural steps when compared to previous systems that used separate acid-conditioning and primer/adhesive steps. The monomers in self-etching composite luting agents contain phosphorulated methacrylates that have the ability to generate self-adhesion. Further, the presence of phosphoric acid groups within the material creates an acidic bonding surface environment. The low pH environment that is created provides for demineralization of the tooth surface, which, in turn, allows for subsequent penetration of the resin cement into the demineralized bonding surface. Once the resin cement polymerizes, micromechanical retention is achieved between the cement and tooth.

Recently, a novel self-bonding resin luting agent, RelyX Unicem, has gained attention, due to its ability to bond to tooth structure without using separate etchant and adhesive protocols. Other self-etching cements (Multilink and Panavia F 2.0) require the mixing of primers to activate the self-etch mechanism; however, they do not require a further rinsing step.

Clinically, if the adhesive bond or the adhesive bonding process is compromised, marginal microleakage between the tooth and restorative material(s) is likely to occur. Marginal leakage could result in further bond failure, create the potential for secondary caries and, if left untreated, advance to pulpal pathology.

Classically defined, microleakage is the diffusion of substances, such as bacteria, oral fluids, molecules and/or ions into a fluid-filled gap or into a structural defect that is present or one that occurs between restorative materials and tooth structure.[12-13]

A die-spacer (die relief) is often used in the fabrication of crowns to provide space for the luting cement. This space helps to reduce resistance of the cementing material to flow, which facilitates complete seating of the restoration and helps to ensure that the cement is extruded from beyond all preparation margins, thereby providing a good marginal seal. The resistance of cementing materials to flow may result in a crown that does not fully seat. This may create marginal discrepancies that will lead to microleakage.[14]

Placing a small perforation (vent hole) in the occlusal aspect of the restoration to facilitate escape of the cement has been suggested to allow for a more complete seating of the restoration.[15-16] Several studies described a trend toward a decrease in elevation (an increase in the accuracy of the occlusion after cementation) following cementation of die-spaced crowns.[17-19] These observations have been attributed to a decrease in hydrolic forces within the cement film,[20] improved outflow of cement and a decrease in contact between the inner surface of the crown and the tooth.[21]

Currently, there is no comprehensive reported data on marginal leakage of all-ceramic crowns bonded with self-etching resin luting agents or reports of the value of using a die spacer technique with these materials when compared to not using a die spacer. This study assessed microleakage (if any) at the tooth structure/ceramic crown interface using three different types of self-etching resin luting agents with and without the use of a die spacer

METHODS AND MATERIALS

Eighteen extracted human mandibular molars, free of cracks, caries and restorations, were used in this study. The teeth were stored in a 0.2 % sodium azide solution for no more than three months. Each tooth was debrided using an ultrasonic scaler. A single operator prepared the teeth for conventional full veneer crowns using a modified flat end taper diamond bur (30 µm, #6847KR), a diamond esthetic trimming egg (30 µm, #6347) and a diamond end cutting (30 µm, #8839) all from Brasseler USA, Savannah, GA, USA. Each preparation had an approximately 20 ± 4 degree taper. Each preparation was made with an approximate 1.0 mm shoulder. The buccal finish line was prepared 1.5 mm below the CEJ and the lingual finish line 1.5 mm above the CEJ, creating restorations with margins in both dentin and enamel.

Impressions of the 18 preparations were made using light and regular body addition polyvinyl siloxane impression material (President, Coltene/Whaledent, Mahwah, NJ, USA). Individual plastic dies (Dup-Die Cups, Portola Packaging, Inc, San Jose, CA, USA) were used to impress each tooth specimen. Master dies were poured in dental gypsum (Die-Keen, Heraus Kulzer, Armonk, NY, USA) and prepared for crown construction. All the master dies were numbered and aligned with their respective tooth preparations.

Nine master dies were treated with three layers of die spacer (Blue Classic Die Spacer, Belle de St Claire, KerrLab Sybron Dental Specialties, Orange, CA, USA) on the axial and occlusal walls. Die spacer was stopped approximately 1 millimeter short of the line angle between the shoulder and axial wall. No die spacer was applied to the other half of the master dies.

IPS Empress crowns (Ivoclar Vivadent) were fabricated following the manufacturer's instructions for waxing, spruing, investing, pressing, divesting, polishing and finishing. A single operator fabricated the wax copings on the master dies. The sprues were cut to a length of 5 mm and carefully attached to the patterns. The position of the wax pattern was checked to make sure it was at least 10 mm within the restriction of the selected ring diameter. The wax copings were invested in a phosphate bonded investment material (Ivoclar Vivadent). A trained technician operated the pressing machine (IPS-Empress P500, Ivoclar Vivadent) and programmed the pressing cycle P05: ramped temperature of 700°C, pressing temperature of 700°C, holed for 20 minutes, then vacuumed at 700°C. Two ingots of the Empress material were used for every ring, which contained three wax patterns. After bench cooling, the all-ceramic crowns were removed from the investment ring using cutting discs and an air-abrasion unit with glass beads.

After complete divesting of the ceramic restorations, the sprues were separated from the copings using a Visionflex open meshed double-sided disc (Course-125 µm, 6934B Brasseler USA). Spray solution (Occlude, Pascal Co, Inc, Bellevue, WA, USA) was used to indicate high spots that would interfere with proper seating of the copings. The intaglio surfaces of the ceramic crowns were adjusted to fit the master dies using a round diamond bur (30 µm, #8801, Brasseler USA). Prior to the cementing procedure, the fit of the ceramic crowns on the teeth was examined thoroughly. Crown margins were checked with an optical light microscope (OptisPec, Microenterprises, Norcross, GA, USA) at a 3.6x magnification in order to verify that the crown margins were smooth and adapting to the preparation margin correctly. The internal surface of each ceramic crown was steam-cleaned for approximately five seconds before cementation. Hydrofluoric acid 8.9% (Porcelain Etch, Ultradent, South Jordan, UT, USA) was applied for 30 seconds, rinsed with water for 15 seconds and dried for 15 seconds using an air-water syringe. Silane (Ultradent) was applied over the etched ceramic surface for one minute, then allowed to evaporate.

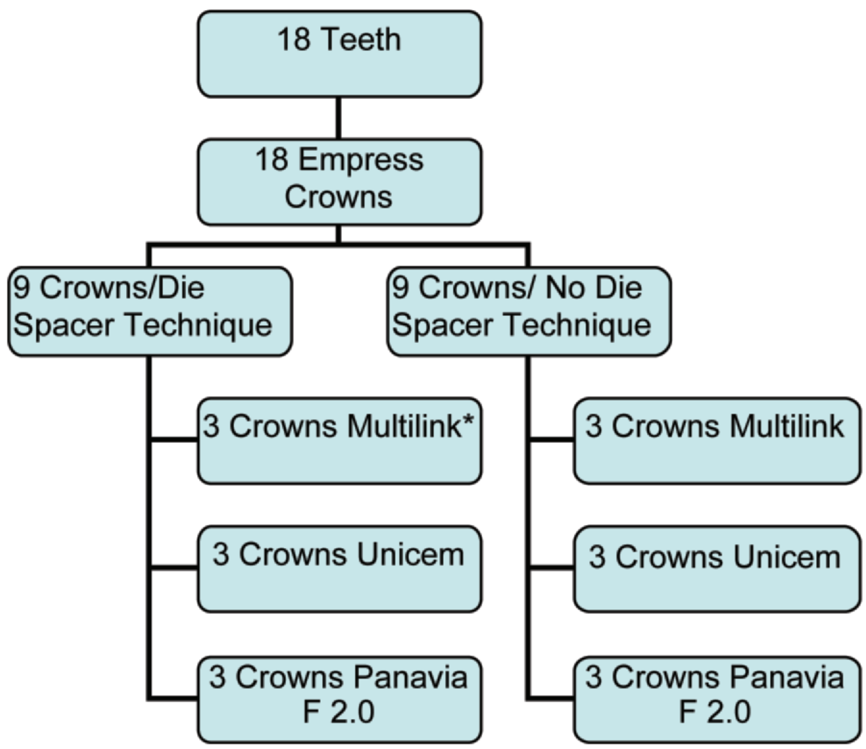
Three self-etching resin luting agents were used: Multilink (Ivoclar Vivadent), Panavia F 2.0 (Kuraray America Inc, NY, NY, USA), and RelyX Unicem (3M ESPE). All the cements were used according to the manufacturers' recommendations as follows:

Multilink: Primers A and B were applied to moist dentin using a microbrush with slight pressure for 15 seconds, then air dried. Multilink cement was applied onto the restoration and seated under digital pressure for two minutes. The excess cement was removed with the aid of an explorer and left to self-cure.

Panavia F 2.0: ED Primer II A&B were combined and applied to moist dentin for 30 seconds, then gently air dried. Pastes A and B were mixed and applied to the crown and seated under digital pressure for two minutes. The cement was partially light-cured for two-to-three seconds, then removed with the aid of an explorer. The cement was light-cured for 20 seconds on each surface using a conventional halogen light Elipar Trilight (3M ESPE Dental Products).

RelyX Unicem Aplicap: This cement did not require a primer solution prior to bonding. The cement capsule was put into the system activator and opened. The capsule was inserted into an amalgamator mixing device Rotomix (3M ESPE Dental Products) for 10 seconds. The cement was dispensed onto the restoration, and the restoration was seated using light digital pressure for two minutes. The cement was allowed to self-cure for five minutes. The excess cement was removed with the aid of an explorer in the gel state.

Table 1: *Experimental Design*



**5 Replications per tooth specimen*

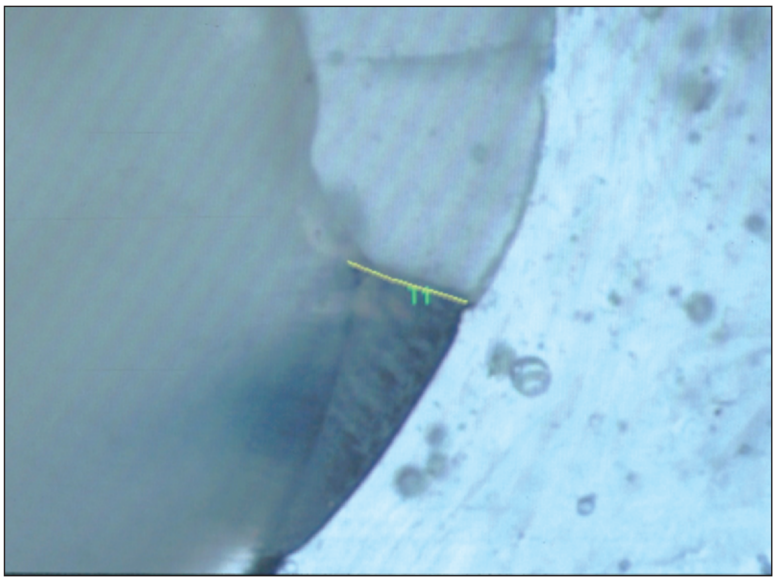


Figure 1. *Digital image analysis of one section with SPOT 3.2 software (Diagnosis Instrument Inc). Dye penetration recorded in millimeters (70x magnification).*

To ensure complete marginal adaptation, the bonded crown margins were checked with a fiberoptic light microscope at a magnification of 3.6x (OptisPec).

The specimens were thermocycled (Forma Scientific Inc, Model 2095, Marietta, OH, USA) for 1000 cycles between two water baths at 5°C and 55°C with a dwell time of 30 seconds in each bath. Following thermocycling, the apices of the teeth were sealed with a resin-modified glass ionomer restorative GC Fuji II LC (GC America Inc, Alsip, IL, USA). The entire surface of each specimen was then covered with two coats of acrylic fingernail polish up to a 1 mm area from the crown margins. The teeth were soaked in an aqueous solution of 5% methylene blue dye for 24 hours at room temperature (22°C). Following dye exposure, the teeth were rinsed thoroughly with a water syringe for 30 seconds, dried and invested with the apices downward in clear, fast-cure epoxy resin (Buehler, Lake Bluff, IL, USA) within a custom silicone rubber die. Resin blocks were allowed to polymerize for 24 hours.

Each embedded specimen was sectioned bucco-lingually with an Isomet slow-speed, water-cooled diamond saw (Buehler), producing five sections (replications) from each tooth. A summary of the experimental design of the study is presented in Table 1.

A digital image analysis system (Image Pro Plus 4.51, Media Cybernetics Inc, Bethesda, MD, USA) was used to measure and qualitatively evaluate the degree of dye penetration with the use of a built-in digital color camera (Sony Model DXC-960 MD) and fiberoptic light at a magnification of 90x. Dye penetration was measured in millimeters. A traveling micrometer was used to take the measurements on both margins on the enamel and dentin of each tooth section. The percentages of dye penetration were calculated according to the extent of dye penetration at the tooth/luting agent and the total tooth perimeter length (Figures 1 and 2).

The data were analyzed using three-way analysis of variance (StatView, Cary, NC, USA). The means were compared at the 0.05 level of significance using Fisher's PLSD intervals.

RESULTS

The resin luting agents tested in this study showed some degree of microleakage. Table 2 lists the means and standard deviations of the percentages of microleakage for the three different resin luting agents and the two die spacer techniques. Analysis of variance (Table 3) showed highly significant differences

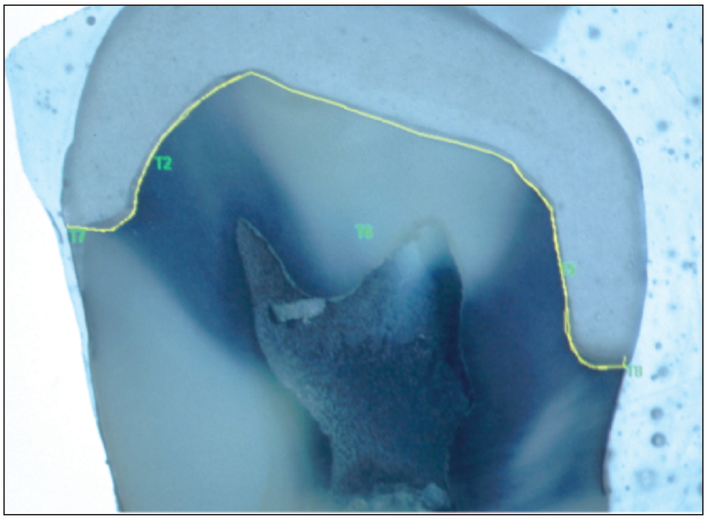


Figure 2. *Intaglio/abutment interface traced and recorded in millimeters.*

Figure 3. *Digital image analysis of Panavia F 2.0 with die spacer.*

in the degree of microleakage among resin luting agents (p<0.0001, power = 1.0) and tooth substrates (p<0.0001, power =0.99). There was no significant difference in the degree of microleakage between the die and no die spacer groups (p>0.1056). Interactions of cement/tooth substrate (p<0.013), die spacer/tooth (p<0.016) and cement/die spacer/tooth substrate (p<0.035) were all significant at the 0.05 level of significance. Fisher's PLSD intervals for comparing resin luting agents, tooth substrates and die spacer techniques were 3.0, 2.5 and 2.5, respectively.

Panavia resin luting agent showed the lowest degree of microleakage both at enamel and dentin with and without the use of a die spacer (1%), followed by RelyX Unicem at the enamel margins with and without die spacer (2%), RelyX Unicem at the dentin margins with (10%) and without die spacer (12%), Multilink resin luting agent at the enamel margins with die spacer (13%), at enamel and dentin without die spacer (15%), and Multilink resin luting agent at dentin margins with die spacer showed the highest degree of microleakage (28%). Among the cements, Multilink resin luting agent showed the highest degree of microleakage.

Digital image analysis revealed minimal or no leakage for IPS Empress crowns bonded with Panavia luting agent using a three-layer die spacer (Figure 3) or no die spacer (Figure 4).

Table 2: *Means and Standard Deviations in Percentages of Microleakage*

	Die Spacer		No Die Spacer	
	Enamel	Dentin	Enamel	Dentin
Multilink	13.12[a] (13.12)	28.38 (15.27)	15.36[a] (11.16)	15.16[a] (17.32)
RelyX Unicem	1.78[c] (0.79)	12.18[a] (4.32)	2.47[c] (1.24)	10.00 (3.88)
Panavia F 2.0	0.76[b] (0.85)	1.12[b] (0.77)	0.90[b] (0.96)	1.05[b] (1.50)

There are no significant differences in groups with the same superscript letter (p>0.05).

Table 3: *Analysis of Variance for Microleakage*

	DF	Sum of Squares	Mean Square	F-Value	*p*-Value	Power
Cement	2	9043.92	4521.96	62.33	<.0001	1.000
Die Space	1	192.01	192.01	2.65	.1056	.349
Tooth	1	1399.31	1399.31	19.29	<.0001	.997
Cement-Die Space	2	267.62	133.81	1.84	.1613	.367
Cement-Tooth	2	653.98	326.99	4.51	.0124	.768
Die Space-Tooth	1	429.86	429.86	5.92	.0160	.678
Cement-Die Space-Tooth	2	497.86	248.93	3.43	.0346	.633
Residual	168	12187.54	72.54			

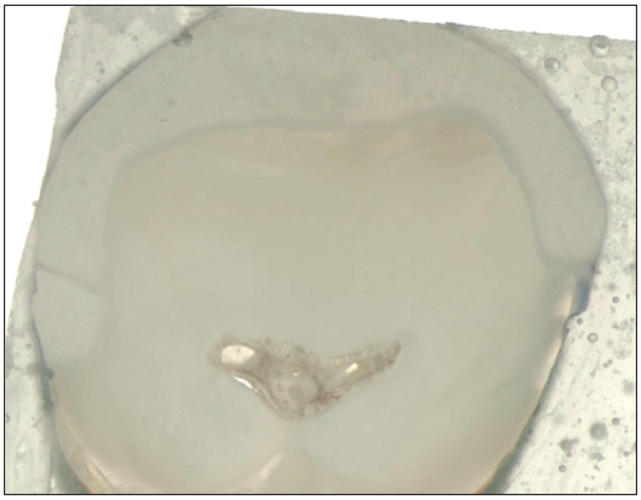


Figure 4. *Digital image analysis of Panavia F 2.0 with no die spacer.*

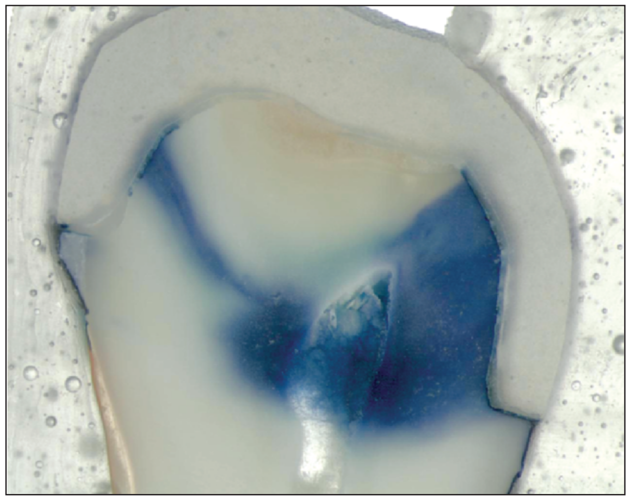


Figure 5. *Digital Image analysis of Multiink with die spacer.*

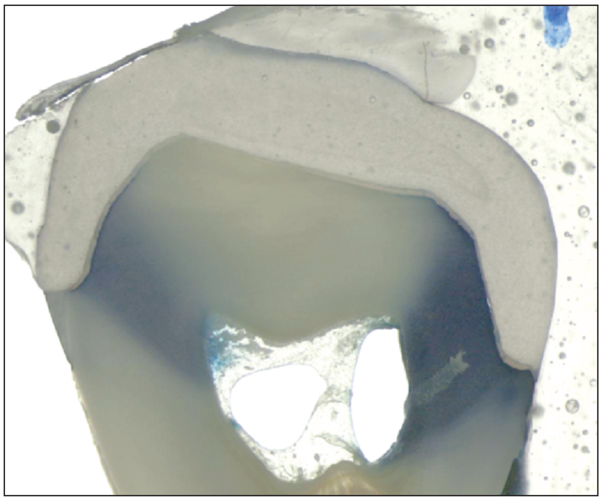


Figure 6. *Digital image analysis of Multilink with no die spacer.*

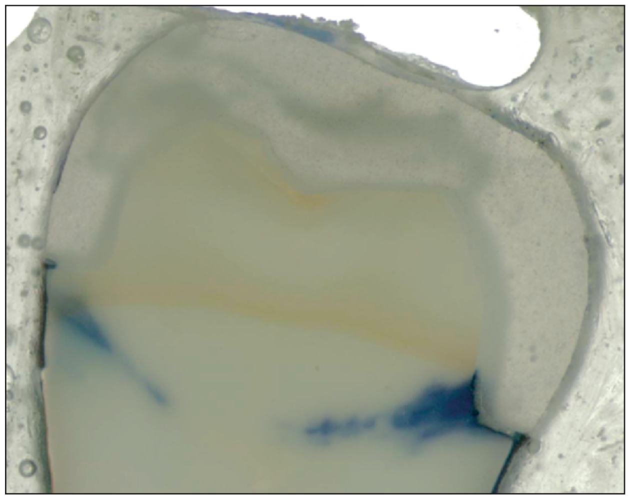


Figure 7. *Digital image analysis of RelyX Unicem with die spacer.*

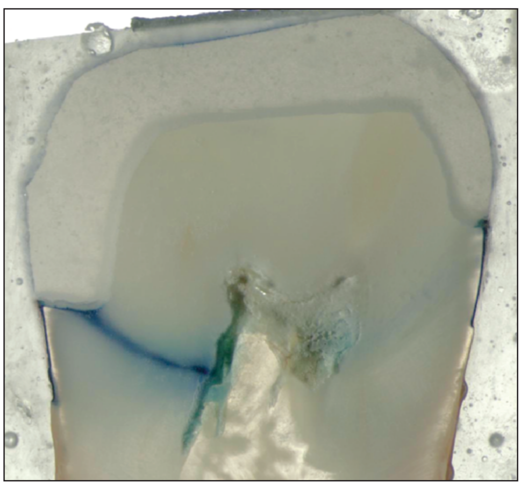


Figure 8. *Digital image analysis of RelyX Unicem with no die spacer.*

IPS Empress crowns bonded with Multilink luting agent showed extensive leakage, usually observed at both axial walls of the tooth preparation with dye spacer (Figure 5) and with no die spacer (Figure 6).

IPS Empress crowns bonded with RelyX Unicem resin luting agent showed a moderate degree of microleakage only involving one of the axial walls of the tooth preparation (Figures 7 and 8).

In general, all testing conditions showed a higher degree of leakage at the dentin margins than at the enamel margins.

DISCUSSION

Among the resin luting agents, differences in the degree of microleakage were found. Two of the cements (Panavia F 2.0 and Multilink) still required a primer on the tooth surface in order to activate the self-etching capabilities of the cement; whereas RelyX Unicem is an encapsulated cement and did not require any type of priming of the tooth surface for the activation of its self-adhesive mechanism.

In this study, Panavia F 2.0 was light-cured. Rosentritt and others[22] investigated the *in-vitro* marginal adaptation of all-ceramic Empress 1 inlays cemented with several resin cements: Panavia F, RelyX Unicem, Variolink 2, Dyract Cem and Fuji Plus, which were either light-cured or self-cured. Panavia F had the lowest marginal leakage values. The authors found higher microleakage values at the cement-tooth interface when these cements were self-cured than when they were light-cured at both the enamel and dentin margins. Another study[23] found microleakage values for RelyX Unicem to be moderate, with no pretreatment of the cement-tooth interface.

Restorations cemented with Multilink exhibited the greatest amount of microleakage. Panavia exhibited the least. This might be due to differences in the pH of the acidic primers between the two cement monomers, with Multilink perhaps creating a weaker etching pattern on the tooth surface, resulting in less resin infiltration and a thinner hybrid layer. On the other hand, the cement viscosity could have an effect on the seating of the crowns, resulting in gap openings that could have contributed to varying degrees of microleakage.[24]

The results in this study demonstrated no significant differences in microleakage with or without an internal relief to allocate the resin cement. This finding suggests that using die spacer for the resin cement might not play as important a role as it does with heavier acid-base, non-adhesive conventional luting cements. There are several reports indicating the effect of cement space to decrease the seating discrepancies of fixed partial denture restorations,[14,17,19,21] although these studies did not indicate whether the relief technique would also contribute to a decrease in marginal leakage. Dietschi and others[25] studied the marginal and internal seal of the adhesive interface of crowns using dye infiltration tests, and one of the variables considered was cement thickness (80, 150 and 300 µ). They found that cement thickness did not influence the marginal seal. The current study is in agreement with this finding.

The type of tooth substrate had an effect on the degree of microleakage. Differences in microleakage were observed between margins bonded to enamel and margins bonded to dentin. It is well-documented that bonding to the enamel structure is more predictable due to its higher mineral content than resin infiltration in dentin, which is characterized by a much higher hydrated collagen structure and less mineralized tissue.[6,9,26-27] Bonding studies to dentin have demonstrated deficiencies on the sealing capacity to dentin compared to that of enamel.[23-25,28-29] It could be assumed from the study that a better seal would result when bonded margins are located on the enamel surface.

There is no restoration that could reproduce a perfect marginal fit to the prepared tooth. Luting cements are used to fill the gap discrepancy between the restoration and the abutment. The results of this study support the concept that type of acidic monomer and location of the preparation margin significantly influence the rate of microleakage. Future studies could focus on evaluation of the marginal leakage of non-etchable, high strength ceramics at the nano level using other self-etching resin cements and assessing whether acidity levels of primers in self-adhesive cement can impact the degree of bonding and therefore microleakage.

CONCLUSIONS

1. Panavia F 2.0 resin luting agent showed the least degree of microleakage at both the enamel and dentin margins, followed by RelyX Unicem and Multilink resin luting agents, respectively.
2. The use of a conventional relief technique did not improve the marginal seal of IPS Empress crowns against marginal leakage.
3. Marginal leakage was higher at margins bonded to dentin and less when bonded to enamel.

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