

Laboratory Research

Microleakage and Marginal Gap of Adhesive Cements for Noble Alloy Full Cast Crowns

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Clinical Relevance

Recently introduced self-adhesive resin cements showed an improved sealing ability for noble alloy full cast crowns compared with a resin-modified glass ionomer or dual-cured resin cement.

SUMMARY

Very limited comparative information about the microleakage in noble alloy full cast crowns luted with different types of adhesive resin cements is available. The purpose of this study was to evaluate the microleakage and marginal gap of two self-adhesive resin ce-

ments with that of other types of adhesive luting cements for noble alloy full cast crowns. Fifty noncarious human premolars and molars were prepared in a standardized manner for full cast crown restorations. Crowns were made from a noble alloy using a standardized technique and randomly cemented with five cementing agents as follows: 1) GC Fuji Plus resin-modified glass ionomer cement, 2) Panavia F 2.0 resin cement, 3) Multilink Sprint self-adhesive resin cement, 4), Rely X Unicem self-adhesive resin cement with pretreatment, and 5) Rely X Unicem with no pretreatment. The specimens were stored in distilled water at 37°C for two weeks and then subjected to thermocycling. They were then placed in a silver nitrate solution, vertically cut in a mesiodistal direction and evaluated for microleakage and marginal gap using a stereomicroscope. Data were analyzed using a nonparametric Kruskal-Wallis test followed by Dunn multiple range test at a $p < 0.05$ level of significance. The Rely X Unicem (with or with no pretreatment) exhibited the smallest

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DOI: 10.2341/10-253-L

degree of microleakage at both tooth-cement and cement-crown interfaces. The greatest amount of microleakage was found for Panavia F 2.0 resin cement followed by GC Fuji Plus at both interfaces. No statistically significant difference in the marginal gap values was found between the cementing agents evaluated ($p>0.05$). The self-adhesive resin cements provided a much better marginal seal for the noble alloy full cast crowns compared with the resin-modified glass ionomer or dual-cured resin-based cements.

INTRODUCTION

Resin-based cements have become popular as luting agents because of the development of dentin bonding agents and direct-filling resin composite materials with improved properties. The clinical success of indirect resin-bonded fixed restorations relies on the retention and support derived from mechanical and/or chemical bonding of the resin luting agent to the tooth and restoration.

Dissolution, shrinkage on setting, and lack of adhesive bond of the luting agent to both tooth structure and restoration have been reported to be possible causes of microleakage.¹ The bonding efficiency of adhesive luting agents is influenced by several factors related to the material itself, such as monomer composition, filler content, and curing mode, and on the type of the substrate surfaces like enamel, dentin, alloys, ceramics, or composites.² Several studies have reported significant differences between adhesive luting agents in their ability to prevent interfacial leakage of cemented restorations.³⁻⁶

Conventional dentin bonding agents are based on smear layer removing or dissolving techniques. These adhesive systems need several steps of application and most of them are technique sensitive.⁷ The discrepancy between etching depth and adhesive penetration may lead to a large area of exposed collagen at the interface between the adhesive and prepared dentin surfaces resulting in postoperative sensitivity with luting agents that require a separate etching step.⁸ Resin cements that include self-etching primers have been introduced to overcome some of these limitations. The concept is using the smear layer as a bonding substrate, but with novel formulations that should etch beyond the smear layer into the underlying dentin.⁹

In addition, self-adhesive resin-based dental cements have been introduced recently, which advocate no pretreatment of tooth substance and thus

simplify the cementing procedure. A wide range of ceramic or metal-based restorations with indications ranging from inlays to fixed partial dentures are included.⁷ While the adhesive luting resins are excellent for bonding to base metal alloys, they have low chemical reactivity to the surfaces of noble alloys because of the lack of surface oxide layer.¹⁰

Very limited comparative information about the microleakage in noble alloy full cast crowns luted with different types of adhesive resin cements is available. The purpose of this study was to evaluate and compare the microleakage and marginal gap of two self-adhesive resin cements with that of other types of adhesive luting cements for noble alloy full cast crowns. The null hypothesis was that there is no difference in the sealing ability of self-adhesive resin cements with that of other adhesive luting agents in noble alloy full cast crowns.

MATERIALS AND METHODS

Specimen Preparation

A total of 50 extracted noncarious permanent human molar and premolar teeth after debridement were stored in 0.5% chloramine T solution for one week and then in distilled water at room temperature up to three months until they were used.

For the standard full cast crown tooth preparations, the occlusal and axial surfaces were reduced by approximately 1.2 and 1 mm, respectively, using a converging angle of around 6°. The cervical preparation margins were finished as circular chamfers using torpedo-shaped diamond burs (D&Z, Geneve, Switzerland) with water-cooling. A new bur was used for every five preparations, and all preparation margins were entirely in dentin.

Impressions of the prepared teeth were taken with an addition silicone impression material (Affinis, Coltene, Altstätten Germany) and poured with type IV extra-hard stone, (Sheraaqua, Shera Werkstoff-Technologie GmbH & CoKG, Lemförde, Germany) following the manufacturer's instructions. The stone dies were trimmed and two coats of die spacer (Gradia separator, GC Corporation, Tokyo, Japan) were applied above the preparation margins. Subsequently, the wax patterns (Inlay wax, Dentecon Inc, VA, USA) were fabricated to model full crowns of 0.5 mm thickness. The wax patterns were then sprued and invested with a phosphate-bonded investment (Hinrivest KB, Type IV, Goslar Harz, Ernst Hinrichs GmbH, Goslar, Germany). The manufacturers' directions were followed for mixing, setting time, and wax burnout of the investment.

The wax patterns were cast using a type IV high-gold casting alloy (Degubond 4, DeguDent GmbH, Hanau, Germany having the following composition: Au and Pt metals 78.7%, Au 49.6%, Pd 29.0%, Ag 17.5%, Sn 3.0 %, Ir 0.1%, Ga 0.5%, Ta 0.1%, and Re 0.2%). The castings were divested, trimmed, and seated. The marginal fit of the castings on the prepared teeth was checked with a probe under a stereomicroscope (Olympus, SZX 12, Tokyo, Japan) at 20 \times magnification.

Cementation

The internal surfaces of cast frameworks were sandblasted with 50 μ m alumina particles at 20-bar pressure and then cleaned in alcohol ultrasonically for 10 minutes. A thin coat of Alloy primer (lot no. 00373A, Kuraray, Osaka, Japan) was applied only to the internal surfaces of crowns cemented by Panavia F 2.0 (Kuraray, Osaka, Japan) according to the manufacturer's recommendation. The teeth were also cleaned in alcohol and randomly divided into five test groups (n=10) for cementation procedures. The cementing agents used in this study were: 1) GC Fuji Plus resin-modified glass ionomer cement (GC Corp, Tokyo, Japan); 2) Panavia F 2.0 dual-cured self-etch resin cement (Kuraray); 3) Multilink Sprint dual-cured self-adhesive resin cement (Ivoclar-Vivadent, Schaan, Liechtenstein); 4) Rely X Unicem (3M ESPE, Seefeld, Germany) dual-cured self-adhesive resin cement with pretreatment; and 5) Rely X Unicem with no pretreatment. All cementing procedures were used according to the manufacturers' instructions at room temperature (23°C \pm 1°C) and relative humidity (50% \pm 5%). The composition and application modes of the tested cementing agents are provided in Table 1.

Cementation was performed by loading a thin layer of the cements into the interior surfaces of the restorations and applying finger pressure for 10 seconds. Then, a static load of 5.0 kg was applied axially on the restorations for five minutes, leaving the material to set in the self-curing mode. Finally, excess cements adhered to tooth surfaces were removed by a scaler. An oxygen-blocking gel (lot no. 00373A, Oxyguard II, Kuraray) was applied on the cement margins for three minutes in group 2 when Panavia F 2.0 was used and then removed with a cotton roll and water spray. Finally, after five minutes of self-curing, 20 seconds of light irradiation (Coltolux 75, Coltene, Whaledent, NJ, USA) at a light intensity of 800 mW/cm² were performed from each side of the specimens. Marginal fit was checked by both visual inspection and with a probe.

Microleakage and Marginal Gap Evaluation

All tooth restoration specimens were stored in distilled water at 37°C for 10 days and then were subjected to 5000 cycles of thermocycling in water baths between 5°C and 55°C (immersion time 20 seconds; transfer time 10 seconds).

After thermocycling, the root apices were sealed with a light-cured resin composite, and the root surfaces were covered with two layers of nail varnish up to 2 mm below each crown margin. Then, the specimens were immersed into a unimolar silver nitrate solution (Crystal, Merk, Germany) for six hours, rinsed thoroughly, and then stored in a photochemical developer (D76, Eastman Kodak, Rochester, NY, USA) for 12 hours followed by an exposure to a 150-W flood lamp for six hours. The specimens were then embedded in a transparent self-cure acrylic resin (Rapid Repair, Meliodent, Heraeus Kulzer GmbH, Hanau, Germany). Each specimen was cut in a mesiodistal direction through the center of the restoration using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) with water-cooling. Each specimen featured four surfaces for analysis of microleakage and marginal gap.

The microleakage in the area of the tooth-cement interface was defined as linear penetration of silver nitrate starting from the restorative crown margins. Microleakage and marginal gap was determined with a stereomicroscope (Olympus, SZX 12). The images were taken at a resolution of 1024 \times 768 pixels. The selected magnification was based on 2.44 μ m equaling one pixel. Marginal gaps were defined as the perpendicular measurement from the crown margin to the tooth finish line at the external tooth surface. All measurements were recorded in microns.

Statistical Analysis

Since the raw data were not characterized by a normal distribution, a nonparametric Kruskal-Wallis test followed by Dunn multiple range test was used to analyze the statistical differences among groups. Pearson correlation was used to assess the correlation between two continuous variables. The selected level of statistical significance was $p < 0.05$.

RESULTS

Figure 1 represents the longitudinal section of restorations with silver nitrate penetration. The mean and standard deviation values for the microleakage and marginal gap findings, and the compar-

Table 1: Chemical Composition and Application Mode of the Cementing Agents Used

Cements	Main Composition	Application
Fuji Plus (lot no. 60327)	Powder: alumino-silicate glass Liquid: hydroxyethyl methacrylate (HEMA), polyacrylic acid, triethylene glycol dimethacrylate (TEGDMA)	1. Apply Fuji Plus conditioner for 20 s on the tooth and rinse and dry by blotting with a cotton pellet. 2. Mix a powder-to-liquid ratio of 2:1 for 20 s. 3. Light-cure (40 s).
Panavia F 2.0 (lot no. 51180)	Paste A: silanated barium glass; colloidal silica; bisphenol A polyethoxy dimethacrylate; 10-methacryloxydecyl dihydrogen phosphate; hydrophilic dimethacrylate; hydrophobic dimethacrylate; benzoyl peroxide; camphorquinone Paste B: silanated barium glass; silanated titanium oxide; sodium fluoride colloidal silica; bisphenol A polyethoxy dimethacrylate; hydrophilic dimethacrylate; hydrophobic dimethacrylate; N,N-diethanol-p-toluidine; sodium 2,4,6-trisopropyl benzene sulfinate	1. Mix ED primer A+B (1:1). 2. Apply on the tooth and gently air-blow after 30 s. 3. Mix paste A+B (1:1) for 20 s. 4. Apply and self-cure (5 min) and light-cure (40 s).
Multilink Sprint (lot no. K04660)	Dimethacrylates, adhesive monomers, fillers, initiators/stabilizers	1. Mix cement. 2. Apply and self-cure (5 min) and light-cure (40 s).
Rely X Unicem (lot no. 241332)	Powder: glass powder, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators	With pretreatment: 1. Apply a 35% phosphoric acid etchant (Ultra-Etch, Ultradent Products Inc, Utah, USA) for 15 s, rinse, and blot dry. 2. Apply an adhesive (Single Bond 2, 3M ESPE, MN, USA) for 10 s, dry, and light cure for 20 s. 3. Mix cement. 4. Apply, self-cure (5 min), and light-cure (40 s).
		Without pretreatment: 1. Mix cement. 2. Apply, self-cure (5 min), and light-cure (40 s).

ison of data among groups obtained for the various cementing agents are shown in Table 2.

Kruskal-Wallis nonparametric analysis revealed statistically significant differences among groups ($p < 0.05$). The smallest degree of microleakage at the tooth-cement and cement-crown interfaces was observed for the Rely X Unicem self-adhesive resin cement with pretreatment followed by the Rely X Unicem with no pretreatment. There was no statistically significance difference between Rely X Unicem resin cement with and with no pretreatment ($p > 0.05$). The greatest amount of microleakage was found for Panavia F 2.0 resin cement followed by GC Fuji Plus resin-modified glass ionomer cement at both tooth-cement and cement-crown interfaces, and there was no significant difference between them ($p > 0.05$).

The mean microleakage values for the Rely X Unicem with and without pretreatment were significantly lower than those of Panavia F 2.0 and GC

Fuji Plus at both interfaces ($p < 0.05$). At the tooth-cement interface, the degree of microleakage for Multilink sprint self-adhesive resin cement was significantly higher than that of Rely X Unicem with pretreatment ($p < 0.05$), but not significantly different from that of Rely X Unicem with no pretreatment or GC Fuji Plus cements ($p > 0.05$). At the cement-crown interface, no statistically significant difference ($p > 0.05$) in the mean microleakage values was observed between Multilink Sprint and Rely X Unicem (with and with no pretreatment). In addition, there was significant difference in the mean microleakage values between Multilink Sprint and Panavia F 2.0 at both tooth-cement and cement-crown interfaces ($p < 0.05$).

The average marginal gap in specimens ranged from $123 \pm 73 \mu\text{m}$ for the self-adhesive resin cement (Multilink Sprint) to $272 \pm 138 \mu\text{m}$ for the resin-modified glass-ionomer (GC Fuji Plus) cement. No

statistically significant difference in the marginal gap values was found between cementing agents tested ($p > 0.05$).

For the Rely X Unicem self-adhesive resin cement (with and with no pretreatment), Multilink Sprint, and Panavia F 2.0 resin cement, no correlation was found between the microleakage (tooth-cement and cement-crown interfaces) and marginal gap values ($p > 0.05$). The only weak direct correlation between microleakage and marginal gap data was observed for the Fuji Plus resin-modified glass ionomer cement.

DISCUSSION

Marginal seating of cast crowns is influenced by several factors such as physicochemical interactions between cement, tooth structure and casting metal, cement viscosity, and possible effects of dentin bonding agents.¹¹ The type, composition, and other characteristics of cementing agents also have an effect on the degree of microleakage.¹² Adhesive luting agents could provide a reduction of microleakage, which is the principal cause of pulpal disease and sensitivity.¹³

From the results of the present study, the smallest degree of microleakage both at the cement-crown and tooth-cement interfaces was obtained by the Rely X Unicem self-adhesive resin cement with pretreatment followed by Rely X Unicem without pretreatment. Similar to this observation in noble alloy full cast crowns, a lower degree of leakage at the tooth-cement interface has been reported by Rely X Unicem in another study¹⁴ when compared with that of Panavia F 2.0 dual-cured resin cement or resin-modified glass ionomer cement (GC Fuji Plus). These findings may indicate that this luting agent is able to provide a sufficient seal at the interface between the noble alloy, cementing agent, and tooth structure.

No distinct demineralization or hybridization in dentin has been observed by Rely X Unicem.^{8,15,16} The bonding mechanism is similar to glass ionomers with an intermediate interfacial layer incorporating partially dissolved smear layers.^{15,17} This luting agent is a self-adhesive and dual-cured luting agent incorporating two setting reactions—a dual-cured redox reaction for polymerization of the resinous phase and an acid-base reaction resulting in the formation of calcium phosphates. Bonding with dentin is established by ionized phosphoric acid-methacrylates of the monomer mixture. Ionization occurs either *in situ* from the water of dentin or from



Figure 1. Longitudinal Section Showing Silver Nitrate Penetration.

the water produced during the neutralization reaction of the phosphate monomers with the basic filler.^{15,18} This variety of interactions seems to enable Rely X Unicem to generate self-adhesion to tooth surface, resulting in an effective seal of the tooth-cement interface,¹⁵ and therefore, the lowest microleakage values among all the adhesive cements evaluated.

Several studies have shown the weak adhesion of Rely X Unicem to enamel.^{6,19-21} These studies have suggested that Rely X Unicem has a better adhesion on dentin than on enamel surfaces and that acid-etching enamel prior to luting is necessary. In this study, the preparation margins for full cast crowns were located in dentin. The results showed that an additional pretreatment for the Rely X Unicem using an etch-and-rinse adhesive system (Single Bond 2, 3M ESPE) did not improve marginal seal of cast crowns at dentin significantly compared with that of this luting agent with no pretreatment. This is in agreement with the results of other investigations.^{7,16} However, Rely X Unicem with pretreatment showed the lowest degree of microleakage, and due to the high standard deviations, differences between specimens cemented using this luting agent with and without additional pretreatment might not have been identified. Further studies employing larger sample sizes in this regard are required.

The extent of microleakage at the cement-crown interface for full cast crowns luted by the Multilink Sprint self-adhesive resin cement was statistically comparable to that of Rely X Unicem (with or

Table 2: Microleakage, Marginal Gap Mean Values (μm), and Kruskal-Wallis Statistical Analysis of the Groups Tested*

Cement Material	Microleakage				Marginal Gap	
	Tooth-Cement Interface		Cement-Crown Interface		Mean (SD)	Mean Rank
	Mean (SD)	Mean Rank	Mean (SD)	Mean Rank		
Fuji Plus	1574.06 (592.80) ^{c,d}	33.25	1542.81 (623.50) ^{b,c}	34.95	272.50 (138.26) ^a	37.10
Panavia F 2.0	2501.87 (727.64) ^d	41.80	2586.25 (723.47) ^c	42.70	165.31 (97.63) ^a	23.45
Multilink Sprint	1077.81 (502.51) ^{b,c}	26.00	577.5 (169.64) ^{a,b}	20.45	123.37 (63.8) ^a	17.85
Rely X Unicem (with pretreatment)	229.36 (104.69) ^a	10.25	138.73 (79.54) ^a	9.95	153.62 (63.62) ^a	24.05
Rely X Unicem (with no pretreatment)	445.02 (164.08) ^{a,b}	16.20	438.75 (171.83) ^a	19.45	157.18 (47.27) ^a	25.05

* Different letters (in each column) indicate statistically significant difference between groups ($p < 0.05$).

without pretreatment) or the resin-modified glass ionomer cement (GC Fuji Plus). The same finding was also obtained at the tooth-cement interface except when comparing with that of Rely X Unicem with pretreatment of tooth surfaces. Although no statistically significant difference in microleakage was observed between Rely X Unicem with and without pretreatment, there was a considerable amount of increased microleakage at the tooth-cement interface by Multilink Sprint when compared with that of the Rely X Unicem with additional pretreatment. It should be noted that no bonding system was used for Multilink Sprint cement in this study. Different results might have been obtained if additional pretreatment was applied with this type of self-adhesive resin cement. This may need to be explored in future studies.

Reduction of perfect margin areas for Multilink Sprint at dentin and enamel has been reported in a recent study.¹⁹ Mazzitelli and others²² assumed that the acidic monomers of Multilink Sprint might not be properly neutralized so that they retain their etching potential, affecting the polymerizing reaction and jeopardizing adhesion. Besides, its phosphonic acid base etching system absorbs water in long-term water storage and therefore jeopardizes adhesion.¹⁹

In the present study, Panavia F 2.0 self-etching dual-cured resin cement exhibited greater microleakage at both interfaces than the two self-adhesive resin cements. Similar observation has been reported by Piwowarczyk and others¹⁴ for full cast crowns

made of noble alloy. The self-etching primers of Panavia F 2.0 are water-based and contain aromatic sulphonate salts to effectively catalyze polymerization of the acidic monomers and to reduce oxygen inhibition. Excess water at the interface may interfere with the polymerization of the acidic monomers of the primer within the hybrid layer resulting in plasticization. The same may occur in the primer films formed on the dentin surface, where the acidic monomers are not neutralized, being in contact with dentin.²³ Diffusion of this film into the luting agent may protonate the amine-reducing agents and thus inhibit the chemical curing component of the setting mechanism, providing reduced interfacial strength.^{24,25} However, similar or higher bond strengths to different restorative materials have been shown for Panavia F 2.0 resin cement when compared with those of Rely X Unicem in other investigations.^{14,26,27} Furthermore, Behr and others¹⁹ have observed a better marginal adaptation of all-ceramic inlays with Panavia F 2.0 when compared with that of two self-adhesive resin cements (Multilink Sprint, Rely X Unicem). In general, different microleakage patterns may be caused by different factors, depending on the type of substrates involved, such as type of restorative materials and structure and composition of the enamel or dentin, age and location of teeth, and the experimental conditions. Therefore, there is a large variation in leakage data from one laboratory to another, depending on the technique used and the manipulative variables adopted during placement of the bonding agents.

By contrast with the resin cements, the hydrophilic formulation of the resin-modified glass-ionomer cements like GC Fuji Plus can compensate for initial setting contraction by subsequent expansion due to water uptake. This difference in chemical behavior might explain the lower microleakage values found for this cement than that of Panavia F 2.0 resin cement.¹⁴

In this study, microleakage at the tooth-cement interface was comparable to that of the cement-crown interface. This is in contrast with other studies in which microleakage was predominantly located at the tooth-cement interface.^{2,12,13} Although a thin coat of metal primer was applied on the cast crowns cemented by Panavia F2.0 as the manufacturer recommends, a considerable degree of microleakage was observed for this cementing agent at the cement-crown interface. However, it should be noted that marginal seal at the metal crown-cement interface is mainly dependent on the combination of alloy composition, metal surface treatment, metal primer, and the cement used.²⁸

The results of marginal gap were least favorable for the GC Fuji Plus resin-modified glass ionomer cement. The self-adhesive resin cement Multilink Sprint showed the smallest degree of marginal gap followed by Rely X Unicem with and without pretreatment. The marginal gap quality of a given type of cementing agent may be codetermined by its specific physicochemical properties.¹⁴ However, no statistically significant difference in the degree of marginal gap was observed among all the cementing agents evaluated. On the other hand, Piwowarczyk and others¹⁴ reported the greatest and lowest degree of marginal gap for the resin cements (Panavia F and Rely X Unicem) and the resin-modified glass ionomer cement (GC Fuji Plus), respectively.

Moreover, the results of the present study revealed no correlation between marginal gaps and microleakage. This is in agreement with other investigations that showed no regular influence of marginal gaps on microleakage in full cast crowns.^{4,13,14}

CONCLUSIONS

- Within the limitations of this study, it can be concluded that the Rely X Unicem self-adhesive resin cement provided a much better marginal seal for the noble alloy full cast crowns. This was followed by the Multilink Sprint self-adhesive resin cement.
- It should be noted that the results obtained from this *in vitro* study cannot necessarily be extrapolated to the clinical situation because of the complex

oral environment. Further long-term clinical studies on the efficacy of self-adhesive resin cements are required.

Acknowledgement

We thank Dr. M.J. Kharazifar for his invaluable assistance in the statistical analysis

(Accepted 8 January 2011)

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