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Sealing Properties of Three Luting Agents Used for Complete Cast Crowns: A Bacterial Leakage Study

O Zmener • CH Pameijer • SMH Rincon
SA Serrano • C Chaves

Clinical Relevance

The results of this study demonstrated that Rely X Luting Plus, a resin-modified glass ionomer cement, exhibited the most favorable sealing properties against bacteria during a 60-day observation period in comparison to a self-etching resin cement Maxcem Elite and a conventional glass ionomer cement Ketac Cem. The bacterial leakage model used for this experiment proved to be a useful method to determine the sealing properties of luting agents used for cementation of cast restorations.

Osvaldo Zmener, DDS, Dr Odont, Postgraduate Department of Endodontics, Faculty of Medical Sciences, School of Dentistry, University of El Salvador/Argentine Dental Association, Buenos Aires, Argentina

*Cornelis H Pameijer, DMD, MScD, DSc, PhD, Department of Reconstructive Sciences, University of Connecticut, Simsbury, School of Dental Medicine, Farmington CT, USA

Sandra M Hernandez Rincon, DDS, MSc, Faculty of Medical Sciences, School of Dentistry, University of El Salvador/ Argentine Dental Association, Buenos Aires, Argentina

Susana Alvarez Serrano, DDS, Postgraduate Department of Endodontics, Faculty of Medical Sciences, School of Dentistry, University of El Salvador/Argentine Dental Association, Buenos Aires, Argentina

Carolina Chaves, DDS, Postgraduate Department of Endodontics, Faculty of Medical Sciences, School of Dentistry, University of El Salvador/Argentine Dental Association, Buenos Aires, Argentina

*Corresponding author: 10 Highwood, Simsbury, CT 06070; e-mail: cornelis@pameijer.com

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SUMMARY

Objective: **To assess the sealing properties of three different luting materials used for cementation of full cast crowns on extracted human premolars.**

Methods: **Thirty noncarious human premolars were prepared in a standardized fashion for full cast crown restorations. All margins were placed in dentin. After impressions of the preparations, stone dies were fabricated on which copings were waxed, which were cast in type III alloy using standardized laboratory methods. Teeth were randomly assigned to three groups of 10 samples each (n=10), for which the following cements were used: 1) a resin-modified glass ionomer cement, Rely X Luting Plus (3M ESPE, St Paul, MN, USA); 2) a self-adhesive resin cement, Maxcem Elite (Kerr Corporation, Orange, CA, USA); and 3) a glass ionomer cement, Ketac Cem (3M ESPE), the latter used as control. After cementation the samples were allowed to bench-set for 10**

minutes, stored in water at 37°C, subjected to thermal cycling (2000×, between 5°C and 55°C, dwell time 35 seconds), and then stored in sterile phosphate buffer for seven days at 37°C. Subsequently, the occlusal surface was carefully reduced until the dentin was exposed. Finishing on wet sand paper removed the gold flash caused by grinding. After sterilization, the specimens were subjected to bacterial microleakage in a dual chamber apparatus for 60 days. Bacterial leakage was checked daily. Data were analyzed using the Kaplan-Meier survival test. Significant pairwise differences were analyzed using the log-rank test followed by Fisher exact test at a $p<0.05$ level of significance.

Results: **Rely X Luting Plus showed the lowest microleakage scores, which statistically differed significantly from Maxcem Elite and Ketac Cem ($p<0.05$).**

Conclusions: **Rely X Luting Plus cement displayed significantly lower microleakage scores than a self-adhesive resin-based and conventional glass ionomer cement.**

INTRODUCTION

Inadequate marginal adaptation of complete cast metal crowns, porcelain fused to metal restorations, ceramic crowns, and inlays/onlays is one of the most important shortcomings that can affect the wash-out of a luting agent, resulting in bacterial penetration between restoration and dentin, thus compromising the durability of a restoration.[1] As demonstrated in a previous study,[2] the choice of an appropriate cementing agent may be a determining factor concerning the degree of microleakage. Unfortunately, contemporary luting agents are not totally effective in preventing marginal bacterial microleakage, which may lead to cement dissolution causing secondary decay, possible pulpal inflammation, and hypersensitivity[3] as well as loss of retention.[4] Currently, glass ionomer, resin, or resin-modified glass ionomer (RMGI) cements are routinely being used by the dental profession. Conventional glass ionomer luting cements bond to dentin by ionic bonds with hydroxyapatite, while resin-based cements bond to dentin through micromechanical interlocking. RMGIs are hybrid formulas and composed of fluoro aluminosilicate glasses, polyacrylic acid, and resin composites and contain photo or chemical initiators and methacrylate monomers.[5] RMGIs bond to dentin through a combined ionic bond between polyacrylic acid and hydroxyapatite and a micromechanical interlocking with collagen and dentinal tubules. Previous studies have shown that resin-based luting materials possess superior mechanical properties over conventional glass-ionomer cements,[6,7] which may be a factor in sealing capacity as well as resistance to displacement.[4] To date, limited information on microleakage at the interface between tooth structure and full cast crowns luted with different types of self-adhesive cements is available.[2,8,9]

The purpose of the present *ex vivo* study was to compare the sealing property of three different luting materials (a resin-modified glass ionomer, a self-etching resin-based cement, and a conventional glass ionomer cement) in complete cast gold crowns by means of a bacterial leakage test. The null hypothesis was that there is no significant difference with respect to marginal leakage among the three tested materials.

MATERIALS AND METHODS

Specimen Preparation

Thirty intact permanent human maxillary and mandibular premolars with fully developed roots, extracted for orthodontic reasons, and showing comparable crown length and size were used. The study received exemption from the Ethics Committee of the Argentine Dental Society. All teeth were prepared by a single operator as described by Pameijer and others.[10] Briefly, the teeth were prepared freehand using a No. 4138 high-speed diamond chamber bur (KG Sorensen, Ind e Com Ltda, SP, Brazil) using copious oil-free water-cooling. The occlusal surface was reduced perpendicular to the long axis, penetrating the dentin, while a medium chamber finish line was established circumferentially in dentin, approximately 0.5 mm short of the cementoenamel junction. All preparations had a height of ±5 mm with a total angle of convergence of approximately 12°, while all line angles were rounded. A new bur was used for every five preparations. From each individual tooth, preliminary impressions were made with Putty Soft, type I body vinyl polysiloxane (President, Coltene/Whaledent, Mahwah, NJ, USA) using individual custom-made acrylic trays. Final impressions were made with light body vinyl polysiloxane type III (President), and dies were poured in type IV extra-hard stone (Densell Mix, GDK, Densell Dental Technology, Buenos Aires, Argentina) following the manufacturers' instructions. The stone dies were trimmed and one coat of die relief (Renfert Pico-Fit, Renfert GmbH, Hilzingen, Germany) was applied, taking care to stay ±1.5 mm short of the margins of the preparation. Subsequently, wax patterns (Whip Mix Corporation, Louisville, KY, USA) were fabricated to model copings ± 0.5 mm thick with flat occlusal surfaces. Using standard laboratory techniques, the wax patterns were invested in phosphate-bonded investment (Ivoclar Vivadent, Amherst, NY, USA) and cast in type III gold (AlbaDent, Fairfield, CA, USA). The castings were divested, and checked for accuracy under a stereomicroscope (Carl Zeiss, Oberkochen, Germany) at 20× magnification. The internal fitting surface was air abraded with 50 μm aluminum oxide. At all times, other than when they were worked on, the teeth were stored in a sterile phosphate buffer (SPB) solution. After checking for fit and retention, each tooth and matching coping were numbered, individually bagged, and autoclaved. After sterilization, 10 samples (n=10) were randomly assigned to each of three treatment groups.

Cementation

The luting agents used in this study are described in Table 1. In group 1, Rely X Luting Plus (3M ESPE, St Paul, MN, USA), a resin-modified glass ionomer cement, was used. Group 2 was cemented with Maxcem Elite, a self-adhesive resin cement (Kerr Corporation, Orange, CA, USA), and group 3 used Ketac Cem (3M ESPE), a conventional acid-base reaction glass ionomer cement. Prior to cementation the teeth were thoroughly rinsed with sterile oil-free water-spray and dried with filtered compressed air. Care was taken to avoid excessive drying. Cementation was performed under aseptic conditions by a single operator and strictly according to the manufacturers' instructions. The castings were filled with the cement mixture and immediately seated, following which they were kept under finger pressure for 2 minutes, Then, for the samples of group 1 and 2, an oxygen inhibition gel (DeOX; Ultradent Products Inc, South Jordan, UT, USA) was applied to the margins and left in place for 4 minutes, after which excess cement was removed with a scaler. After bench setting for an additional 10 minutes, the marginal fit was checked by visual examination and a probe, and the samples stored for 24 hours in SPB at 37°C. They were subsequently subjected to 2000 thermal cycles in water baths at 5°C and 55°C with a dwell time of 35 seconds and once more stored in

Table 1: *Description of Cementing Agents Used in This Study*

Material/Lot	Material Type	Manufacturer	Composition
Rely X Luting Plus			
Lot N386694	Paste/paste, dual syringe direct dispensing through clicker dispensing system	3M ESPE, St Paul, MN, USA	Paste A: Radiopaque fluoro aluminosilicate (FAS glass) opacifying agent, HEMA, water, proprietary reducing agent
			Paste B: nonreactive zirconia silica filler methacrylated polycarboxylic acid, HEMA, Bis-GMA, water, potassium per sulphate
Maxcem Elite			
Lot 3493741	Paste/paste, dual syringe direct dispensing through a mixing tip	Kerr Corporation, Orange, CA, USA	Glycerol phosphate dimethacrylate co-monomers (mono-, di-, and tri-), functional methacrylate monomers, water, acetone, ethanol, barium, glass-fumed silica, sodium hexafluorosilicate (complete formulation not available)
Ketac Cem			
Lot 460448	Powder/liquid	3M ESPE, St Paul, MN, USA	Powder: glass powder, polycarboxylic acid, pigments
			Liquid: water, tartaric acid, conservation agents

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate.

SPB for seven days at 37°C. In preparation for the bacterial leakage test, the occlusal surfaces of the gold copings were reduced until the dentin was exposed using a fine high-speed diamond bur with light pressure and copious water-cooling. This was followed by sanding the occlusal surface on wet garnet paper of 400 and 600 grit (3M ESPE) to remove gold flash that may have been caused by the diamond bur. Subsequently, the entire root surface 1 mm below the margin of the copings was coated with two layers of nail polish and the samples subjected to bacterial microleakage.

Bacterial Leakage Setup

For this experiment, a slight modification of the dual chamber test apparatus described by Imura and others[11] was used. The tip of 1.5-mL Eppendorf plastic tubes (upper chamber) was cut and the sample was pushed (crown first) through the opening until approximately one half of the crown protruded through the end of the tube. The junction between the sample and the tube was sealed with sticky wax, making sure the crown margin was situated in the upper chamber. The tubes were put into glass vials (lower chamber) containing 10 mL of sterile trypticase soy broth (TSB; Difco Laboratory, Detroit, MI, USA) in such a way that the occlusal dentin/cement interface was submerged in the broth of the lower chamber (Figure 1). The junction between the tube and the glass vial was then sealed with two layers of cyanoacrylate (Ciano, Anaerobicos IWT, Buenos Aires, Argentina) and finally covered with sticky wax. The entire test apparatus was sterilized with ethylene oxide gas for 12 hours and then incubated at 37°C for 72 hours to verify sterility. If the TSB broth showed turbidity, the test set-up was discarded and replaced by a new one and the process repeated.

Bacterial Leakage Test

This phase of the study was carried out in a microbiology laboratory by a microbiologist under strict sterile conditions. The upper chamber was filled with 1 mL of TSB containing 24-hour growth of *Enterococcus faecalis* ATCC 29212 (10^8 colony-forming units/mL). The inoculated apparatus was incubated for 60 days at 37°C. The upper chamber was reinoculated every five days with fresh cultures of the microorganism. The TSB broth in the lower chamber was checked daily for turbidity, which, when observed, was an indication that bacterial leakage had occurred along the crown/cement or cement/dentin interface. Once turbidity was detected, the day of the observation was recorded. Samples from both chambers were then incubated on blood agar plates to check bacterial viability by morphologic observation and Gram staining. The number of teeth demonstrating bacterial leakage and the days on which leakage occurred were recorded for each group.

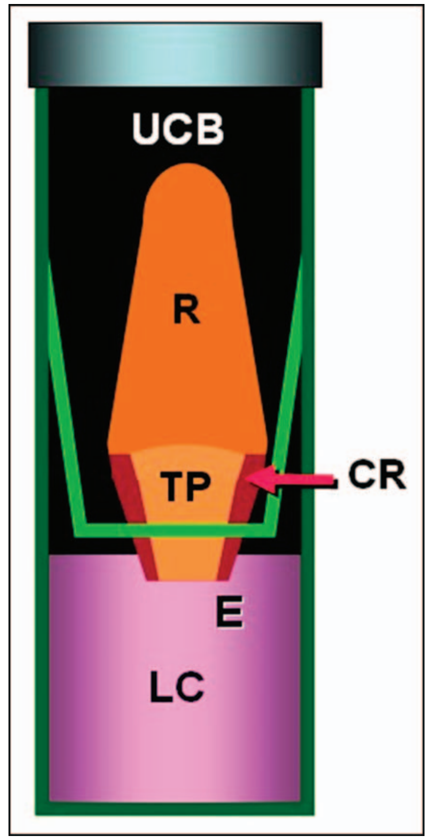

Figure 1. *Schematic set-up of the bacterial leakage system. CR, cast crown; E, exit for bacteria; R, root; TP, tooth preparation; TSB, lower chamber containing TSB broth; UCB, upper chamber conaining TSB broth with* E faecalis.

Statistical Analysis

The length of time until leakage was detected was compared among the three groups using the Kaplan-Meier survival analysis. Significant pairwise differences were analyzed using the log-rank test followed by Fisher exact test. The selected level of statistical significance was $p<0.05$. The presence of *E faecalis* was also checked.

RESULTS

Before the bacterial leakage test, two samples in group 1 and one in group 2 showed evidence of contamination. They were sterilized again according to the procedure described above. The results for the

Table 2: *Samples Showing Turbidity Per 20-Day Interval*

Group/Material	n	Experimental Period, d			
		1-20	21-40	41-60	No Leakage
1. RelyX Luting Plus	10	0	0	1	9 (90.0%)
2. Maxcem Elite	10	0	2	3	5 (50.0%)
3. Ketac Cem	10	0	3	4	3 (30.0%)

three groups are shown in Table 2. In group 1, turbidity did not occur until 55 days (one sample), whereas no leakage was observed in nine samples (90.0%). In group 2, leakage occurred at 38, 40, 42, 43, and 50 days (one sample each). In this group, five samples (50.0%) did not show leakage. In group 3, turbidity was seen at 22 days (one sample), 30 days (one sample), 33 days (one sample), 43 days (two samples), 44 days (one sample), and 47 days (one sample), whereas 3 samples (30.0%) did not show leakage. Fisher exact test revealed that the mean bacterial leakage values for Rely X Luting Plus were significantly lower than those of Maxcem Elite and Ketac Cem ($p<0.05$), while no significant differences were found between Maxcem Elite and Ketac Cem ($p>0.05$). The median survival time for Ketac Cem (ie, absence of bacterial leakage) was 60 days (with a 95% confidence interval of 45.8-74.2), while for Rely X Luting Plus and Maxcem Elite it could not be determined as it was greater than 60 days, the time interval the experiment lasted. Bacteriologic testing of the contents of the lower chamber with the samples that demonstrated leakage revealed viable *E faecalis*.

DISCUSSION

Marginal fit of full cast restorations has always been of concern to clinicians since poor adaptation is related to microleakage.[8] The chemical composition and the type of luting material used for cementing full cast metallic crowns has a bearing on fluid and bacterial microleakage.[2,12,13] Bacterial leakage at the crown margins and between the luting material and dentin is an important factor involved in secondary decay as well as pulpal and/or gingival inflammation.[3,14,15] The use of conventional dyes or stains has provided considerable information about this issue.[8,13,16,17] However, it seems unlikely that testing agents in a molecular state would be totally suitable to provide information on microleakage of particulate matter or aggregates of macromolecules such as large enzymes, bacteria, and/or bacterial by-products.[18] In the present study, we used an *ex vivo* method to analyze the penetration of bacteria at the margins of cast gold crowns luted with different types of cements. Controversy exists in the literature whether or not *ex vivo* and *in vivo* microleakage testing correlate and if results from *ex vivo* experiments can be correlated with the clinical situation.[19] In clinical practice, other factors such as material biocompatibility, ease of use, and specific requirements for each individual case (eg, height of residual tooth structure, preparation angle, and location of the preparation margins) may influence the choice of cement to be used.[20]

E faecalis was chosen because it exists in the normal oral flora in humans and is frequently found in mixed infections with other aerobes and facultative anaerobes.[21] For evaluation, a qualitative approach (presence or absence of turbidity) during a 60-day period was used. Although this observation period was similar to what has been reported in the endodontic literature,[22,23] it should be emphasized that 60 days is a short period considering much higher expectations of longevity of a restoration. In spite of the fact that the experimental conditions used in this study did not include a broader range of factors that affect marginal leakage of full cast crowns in a clinical environment,[24] the test nevertheless allows for a comparison of different materials under strictly controlled conditions.

It should be noted that our test was focused only on crown margins placed on dentin. As previously has been demonstrated,[3,25] the degree of microleakage is higher on dentin than on enamel, therefore a worst-case scenario was tested. Another untested variable in this study was the cement thickness between the restoration and dentin. As per protocol, die-relief spacer was used in order to decrease the seating discrepancies of the castings,[26,27] albeit it has been previously shown that the marginal seal is not negatively influenced by the cement thickness.[25] However, this variable needs further research. Occlusal load stress, which normally occurs under *in vivo* conditions, is another variable that was not tested in the current study and should be taken into consideration when marginal leakage of cemented crowns is tested. Prior to being subjected to bacterial microleakage, all samples had undergone thermal cycling, which is a method to simulate the long-term stresses to which a restoration is exposed under clinical conditions.[13,16,28] However, it has been reported that higher microleakage values were registered when thermocycling was followed by load-cycling[29] an issue that also needs to be investigated more extensively.

The results demonstrated that Rely X Luting Plus had significantly lower leakage scores than Maxcem Elite and Ketac Cem, suggesting that Rely X Luting Plus provided an acceptable marginal seal for up to 60 days. These results are in agreement with Pameijer and others[10] who reported that gold copings cemented with Rely X Luting Plus demonstrated good sealing properties. Furthermore, Rossetti and others[17] reported that Rely X Luting cement (3M ESPE) showed significantly lower leakage than a resin-based luting agent or a zinc phosphate cement. Rely X Luting cement and Rely X Luting Plus have chemically similar formulas and differ only in that the first is a powder/liquid formulation, while the second comes as a paste/paste and is delivered by a clicker dispenser. This chemical similarity let led us to suppose that the physico-chemical interactions with the tooth surface are the same for both formulations.

When compared to Rely X Luting Plus, the bacterial leakage rates were significantly more pronounced for Maxcem Elite and Ketac Cem. According to reports in the literature, bacterial penetration indicates that potential gaps are present at the tooth-cement interface that may lead to secondary decay, pulpal inflammation, and periodontal disease.[3,14,15] Maxcem Elite is a contemporary self-adhesive resin cement with 69% filler content by weight. Previous studies[30,31] have shown adhesive failure between materials of resinous nature and dentin. As demonstrated by Frassetto and others,[32] Maxcem Elite exhibited high volumetric contraction stresses and greater shrinkage values than other tested resin cements. In the present study, the leakage patterns registered for Maxcem Elite did not show significant differences with the conventional glass ionomer cement Ketac Cem. Similar to Maxcem Elite, high volumetric contraction stresses and low-bond strength capacity to dentin have also been reported for Ketac Cem.[33] Indeed, the higher bacterial leakage scores by both materials may be explained because of high contraction stresses that may compromise the bonding leading to an increase in microleakage.

The lower sealing ability of Ketac Cem is in agreement with previous observations of Pameijer and others[10] and a study of White and others[12] who reported that only 29% of samples cemented with Ketac Cem revealed zero leakage. Furthermore, Piemjay and others[34] reported that the acid-base cement Ketac Cem exhibited greater microleakage when tested with a 0.5% basic fuchsin dye for 24 hours. The bacterial leakage data reported here also corroborates favorably with a pilot leakage study published previously[35] using methylene blue dye in which Ketac Cem demonstrated high leakage patterns. On the contrary, Tung and Coleman[36] and Coleman and others,[37] reported that, among other luting agents, Ketac Cem was effective in preventing leakage of detectable molecular concentrations of lipopolysaccharide and dextran. These contradicting data from one laboratory to another may be the consequence of using different experimental conditions as well as by variables such as tooth preparation, treatment of the dentin surfaces, and application of the luting material.

In summary, the current study demonstrated that Rely X Luting Plus showed significant differences with Maxcem Elite and Ketac Cem in its ability to better seal the interface crown/cement and cement/dentin against bacterial microleakage, while Maxcem Elite and Ketac Cem showed comparable microleakage results. Therefore, the null hypothesis must be partially rejected.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- The method described here appeared to be a suitable method for testing the sealing ability of luting materials against bacterial microleakage when used for cementation of cast crown restorations.
- Cast crowns cemented with Rely X Luting Plus provided an acceptable marginal seal up to 60 days and showed significantly lower bacterial leakage when compared to Maxcem Elite and Ketac Cem.

- The stability and the sealing properties of the tested materials at the restoration/tooth interface after more than 60 days need to be investigated further.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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