



## Microleakage of Glass Ionomer-based Provisional Cement in CAD/CAM-Fabricated Interim Crowns: An *in vitro* Study

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### ABSTRACT

**Aim:** The aim of this study was to compare *in vitro* the marginal microleakage of glass ionomer-based provisional cement with resin-based provisional cement and zinc oxide non-eugenol (ZONE) provisional cement in computer-aided design and computer-aided manufacturing (CAD/CAM)-fabricated interim restorations.

**Materials and methods:** Fifteen intact human premolars were prepared in a standardized manner for complete coverage of crown restorations. Interim crowns for the prepared teeth were then fabricated using CAD/CAM, and the specimens were randomized into three groups of provisional cementing agents (n = 5 each): Glass ionomer-based provisional cement (GC Fuji TEMP LT™), bisphenol-A-glycidyl dimethacrylate (Bis-GMA)/triethylene glycol dimethacrylate (TEGDMA) resin-based cement (UltraTemp® REZ), and ZONE cement (TempBond NE). After 24 hours of storage in distilled water at 37°C, the specimens were thermocycled and then stored again for 24 hours in distilled water at room temperature. Next, the specimens were placed in freshly prepared 2% aqueous methylene blue dye for 24 hours and then embedded in autopolymerizing acrylic resin blocks and sectioned in buccolingual and mesiodistal directions to assess dye penetration using a stereomicroscope. The results were statistically analyzed using a nonparametric Kruskal–Wallis test. Dunn's *post hoc* test with a Bonferroni correction test was used to compute multiple pairwise comparisons that identified differences among groups; the level of significance was set at  $p < 0.05$ .

**Results:** All groups exhibited marginal microleakage; the Bis-GMA/TEGDMA resin-based provisional cement demonstrated the lowest microleakage scores, which were statistically different from those of the glass ionomer-based provisional cement and the ZONE cement.

**Conclusion:** The provisional cementing agents exhibited different sealing abilities. The Bis-GMA/TEGDMA resin-based provisional cement exhibited the most effective favorable sealing properties against dye penetration compared with the glass ionomer-based provisional cement and conventional ZONE cement.

**Clinical significance:** Newly introduced glass ionomer-based provisional cement proved to be inferior to resin-based provisional cement as far as marginal microleakage is concerned.

**Keywords:** Dye penetration, Interim restorations, Marginal microleakage, Provisional cements.

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### INTRODUCTION

Provisional cements are cements that are used to temporarily bond interim or definitive restorations. These cements should be of sufficient mechanical strength to retain the restoration for a limited period of time, yet weak enough so that the restoration can be retrieved without undue force that might damage the restoration or the teeth.<sup>1</sup>

Several materials can be used for provisional cementation, and these materials can be divided into groups based on their chemical composition (bases): Zinc oxide eugenol (ZOE) based, zinc oxide non-eugenol (ZONE) based, and resin based<sup>2</sup> cements are the earliest commercially available provisional cements in the market. The primary advantages of these cements are their low cost and their sedative and excellent antibacterial properties.<sup>3</sup> Unfortunately, their high solubility, hydrolytic breakdown, and low mechanical properties are major drawbacks; furthermore, the presence of eugenol in their composition may lead to allergic reaction in some

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patients.<sup>4</sup> Eugenol is also considered to be a free radical scavenger and therefore inhibits free radical polymerization in the resins.<sup>5,6</sup>

The drawbacks of ZOE cements and the necessity of longer cementation periods led to the introduction of non-eugenol-based and resin-based provisional cements. Zinc oxide non-eugenol cement materials are based on ZnO-poly-organic acid systems, typically polycarboxylic and polyacrylic acids. Non-eugenol provisional cements are compatible with resin provisional materials and permanent resin cements; they are also stronger, more retentive, and possess longer working and setting times than ZOE.<sup>2,7</sup> Unfortunately, they still have weak mechanical properties and high solubility. Resin-based provisional cements exhibit strong mechanical properties that are considered to be four times higher than those of ZOE and ZONE cements.<sup>8</sup> In addition, resin-based provisional cements have excellent retention, better esthetics, and low solubility.<sup>9</sup> Therefore, resin-based provisional cements are considered to be more suitable for long-term provisional cementation than ZOE and ZONE cements.

One of the primary functions of provisional cements is to provide a seal and prevent marginal leakage that may jeopardize the biological health of the dentin–pulp complex.<sup>10</sup> Several studies have investigated the sealing ability of provisional cements. A poor sealing ability was reported for ZOE and ZONE cements due to their weak mechanical properties and high solubility; meanwhile, resin-based provisional cements have been reported to have more favorable sealing properties.<sup>11–13</sup>

The first glass ionomer-based provisional cement was recently introduced onto the market. The sealing ability of this cement has not been widely investigated. Therefore, the purpose of this study was to compare *in vitro* the marginal microleakage of glass ionomer-based provisional cements with resin-based provisional cements and ZONE provisional cements in computer-aided design and computer-aided manufacturing (CAD/CAM)-fabricated interim restorations. The null hypothesis is that there would be no significant difference between these provisional cements in terms of marginal microleakage.

## MATERIALS AND METHODS

### Sample Preparation

Fifteen freshly extracted intact human maxillary premolar teeth were collected and stored in distilled water at room temperature until use. The teeth were dried and the roots were painted with nail polish 1 mm apical to the cemento-enamel junction. They were then embedded in transparent cold-cure acrylic resin (Eco Cryl Cold, Protechno, Spain, lot #14-34951) utilizing a mold obtained from 19 mm diameter cylindrical polyethylene pipe.

The teeth were then prepared for complete coverage restoration in a standardized manner using a rotary diamond-cutting instrument in a high-speed handpiece with water spray coolant with flat occlusal reduction, a 1 mm radial shoulder finish line, and 1.5 mm axial reduction. To ensure that all of the preparations had an even reduction, a depth cutter bur (MADC15-009M-FG; NTI-Kahla GmbH, Germany) was used first, and then the preparation was finished using a tapered diamond for the round shoulder bur (847KR-018F-FG; NTI-Kahla GmbH, Germany).

Digital impressions of the prepared teeth were made using a 3shape scanner (D810 3D Scanner, Copenhagen, Denmark). The interim crowns were then designed in the dental laboratory using a CAD program (Dental System™ 3shape, Copenhagen, Denmark). The interim crowns were designed with a wall thickness of 1 mm and a cement space of 25 µm. The STereoLithography data set was saved and imported into a milling center (Cercon Brain Expert system; Degudent, Hanau, Germany). The interim crowns were machine milled from a 20 mm high Cercon base polymethyl methacrylate (PMMA) disk that was composed of highly cross-linked methyl (Cercon; DeguDent GmbH, Germany: Batch# 005366122220) (Fig. 1). The interim crowns were removed from the disk and placed on the prepared teeth; the crowns were adjusted on the prepared teeth as necessary, and marginal fit was confirmed using a sharp probe.

The teeth with their interim crowns were randomly allocated into three groups (n=5 each), and each group received a different provisional cement (Table 1). The cements were mixed according to the manufacturer's instructions at room temperature (23±1°C). The resin cement was available in a dual cartridge with a static auto-mix system, and the glass ionomer-based cement was available as a dual-cartridge paste–paste system for hand mixing (Fig. 2). The ZONE was available as a

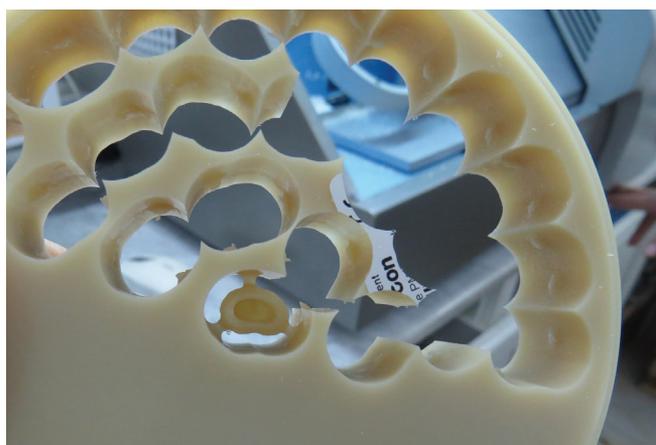
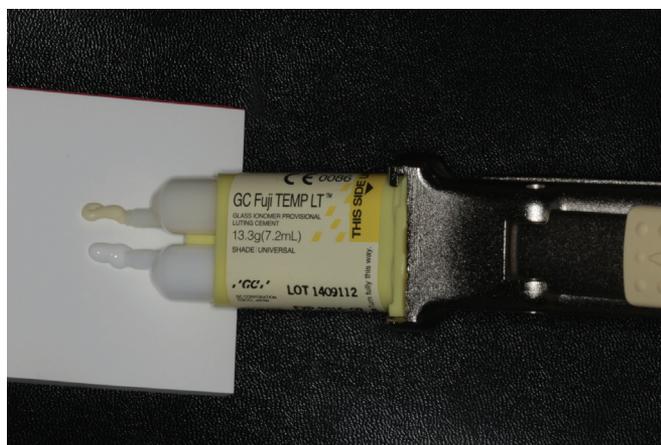


Fig. 1: Provisional crown milled from Cercon base PMMA disk

**Table 1:** Description of provisional cementing agents used in this study

Brand	Type	Composition*	Manufacturer	Lot No
GC Fuji TEMP LT™	Glass ionomer cement	Base: Fluoro-alumino-silicate glass (amorphous); Catalyst: Polyacrylic acid, Glycerin	GC America, Alsip, Ill, USA	1409112
UltraTemp REZ	Resin cement	Base: Mono-2-(Methacryloyloxy) Ethyl Succinate, hydroxy propyl methacrylate, GDMA-Phosphate; Catalyst: Bis-GMA, TEGDMA	Ultradent Products, Inc., South Jordan, USA	BBBL6
Temp-Bond NE	ZONE cement	Base: Zinc oxide (ZnO); Catalyst: Ortho-ethoxybenzoic acid (EBA), Octanoic acid, Carnauba wax	Kerr Corp, Orange, California, USA	5257682

\*According to the information provided by the manufacturer; GDMA: 1, 3-glycerol dimethacrylate



**Fig. 2:** Glass ionomer-based cement (dual-cartridge paste-paste system) used in the study

two-tube system for hand mixing. The glass ionomer-based cement and the ZONE were mixed as follows: The two pastes were dispensed in equal lengths onto a mixing pad provided by the manufacturer. Then, the pastes were mixed with a metal cement spatula until a homogeneous color was obtained. A small quantity of cement necessary to obtain an even distribution was applied just occlusally to the cavosurface margin of the interim crowns. Then, the crowns were seated with finger pressure and placed under a controlled axial load of 5 kg for 10 minutes at room temperature to ensure complete seating. After 10 minutes, the excess cement was removed with a curette. Then, the teeth were stored in distilled water for 24 hours in an incubator at  $37 \pm 1^\circ\text{C}$ .

### Thermocycling Process

The specimens were subjected to 100 thermal cycles. Each thermal cycle consisted of immersing the specimens alternatively in water baths maintained at 5 and  $55^\circ\text{C}$  with 20 seconds of dwell time and 15 seconds of transition time.

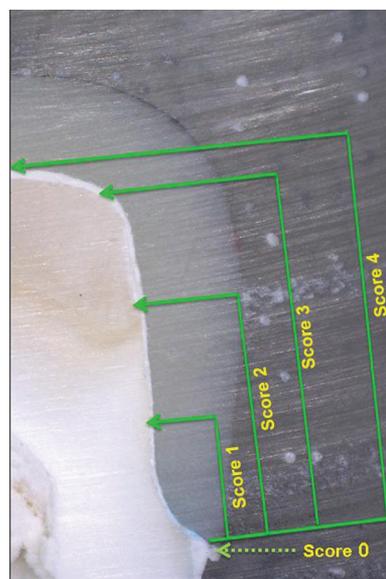
Next, the specimens were again placed in distilled water at  $23 \pm 1^\circ\text{C}$  for 24 hours. Then, the specimens were dried and painted with nail polish three times over the entire interim crown, except for a 2-mm ring area from the restoration cavosurface; this was done by placing 2 mm-wide strips of paper around the circumference of the interim crown and securing them with adhesive tape

prior to applying the nail polish. After the polish dried completely, the specimens were stored in freshly prepared 2% aqueous methylene blue solution at room temperature for 24 hours and then removed and gently washed with tap water for 10 minutes. The nail polish was scratch removed and then the specimens were dried. The coronal portion was then embedded in transparent acrylic resin (Eco Cryl Cold, lot #14-34951) and left on the bench top to polymerize.

### Microleakage Assessment

After the resin was set, each tooth was sectioned longitudinally in the buccolingual and mesiodistal directions using a diamond disk (Super Diamond Disk no. 800.104.355.524.190; NTI-Kahla GmbH, Germany) under dry conditions. This sectioning yielded four separate measurement points per specimen, which were examined under a stereomicroscope (Hamilton, Hamilton International s.r.l. Lazio, Italy) at an original magnification of 20 $\times$ . The extent of the dye penetration was scored by a single operator according to the 5-point scale adopted by Tjan et al<sup>14</sup> (Fig. 3):

1. No microleakage.
2. Microleakage less than one-third of the axial wall length.



**Fig. 3:** The 5-point scale scoring system, which was used in the study

3. Microleakage more than one-third but less than two-thirds of the axial wall length.
4. Microleakage all along the axial wall length.
5. Microleakage on the occlusal surface.

The value of the marginal microleakage assigned to each specimen was the average of the scores of dye penetration recorded from the four measurement points.

### Statistical Analysis

A nonparametric Kruskal–Wallis test was applied to determine if there were differences in microleakage scores among different provisional cement groups. Dunn's procedure with a Bonferroni correction was used for pairwise comparisons when the Kruskal–Wallis tests were significant. The level of significance was set at  $p < 0.05$ . We performed the statistical analysis using the Statistical Package for the Social Sciences version 20.0 software (SPSS Inc., Chicago, IL, USA).

### RESULTS

The means and standard deviations of the microleakage scores for each provisional cement are provided in Table 2. Statistical analysis (Kruskal–Wallis) revealed that the distributions of microleakage scores were not similar for all groups based on a visual inspection of a boxplot. The distributions of microleakage score were statistically significantly different among groups,  $\chi^2(2) = 9.946$ ,  $p = 0.007$ .

Pairwise comparisons were performed using Dunn's (1964) procedure with a Bonferroni correction for multiple comparisons (Table 3). This *post hoc* analysis revealed that there were statistically significant differences ( $p < 0.05$ ) in microleakage scores between bisphenol-A-glycidyl dimethacrylate (Bis-GMA)/triethylene glycol dimethacrylate (TEGDMA) resin cement and

ZONE-based cement. No statistically significant differences were found between glass ionomer-based cement and ZONE-based cement.

### DISCUSSION

In this *in vitro* study, the marginal microleakage of the first commercially available glass ionomer-based provisional cement was compared with Bis-GMA/TEGDMA resin-based provisional cement and ZONE cement using the dye penetration method, which has been a popular method for microleakage assessment.<sup>15</sup> Resin cement was selected because its good sealing ability has been reported in the literature; ZONE was selected for comparison because it is one of the oldest and most commonly used cements for provisional cementation.<sup>2,13</sup>

In this experimental setup, every effort was made to standardize all the factors that could affect microleakage (i.e., tooth preparation, provisional restoration fabrication, the cementation technique, and the thermocycling process). During tooth preparation, control of the amount of reduction was attained using depth cutter burs. In addition, the margin configuration was a radial shoulder instead of a deep chamfer because a shoulder exhibited smaller marginal gap in previous reports.<sup>16,17</sup> For the thermocycling process, the small number of thermocycles which was performed corresponded to the short period of service of provisional restoration in the oral cavity. Moreover, the temperature range of the cycles is more important than the number of cycles.<sup>18,19</sup> In this study, we used a CAD/CAM system to fabricate the interim crowns because it improves the average quality of the prosthetic adaptation and yields a reduction in both the marginal gap and cement film thickness compared with that obtained with conventional manufacturing methods.<sup>17,20</sup> This system also allows us to design and mill the interim crowns to ensure reliability in reproducing the same restoration wall thickness, cement space, and marginal gap in a large number of interim crowns; this consistency was obvious in the stereomicroscopic photographs of the specimens (Fig. 4).

The Bis-GMA/TEGDMA provisional resin cement was the most effective material in terms of preventing microleakage under interim crowns. This result is consistent with previous reports and can be attributed to two factors: The low solubility of resin cements and the presence of the comonomer TEGDMA in the cement formula, which enhances hygroscopic expansion due to the increased water uptake; this hygroscopic expansion compensates for polymerization volumetric shrinkage in the resin cement. Moreover, the presence of the comonomer TEGDMA lowers the cement's viscosity and increases its hydrophilicity, thereby improving adaptation to tooth structure and hence the sealing ability.<sup>21,22</sup>

**Table 2:** Microleakage mean scores, standard deviation (SD), standard error (SE), and median of the three cements

Cement	Glass ionomer (Fuji TEMP LT™)	Resin (Ultra Temp REZ)	ZONE (Temp- Bond NE)
Mean	3.25	0.90	3.50
SD	0.50	0.28	0.17
SE	0.22	0.13	0.08
Median	3.50	1.00	3.50

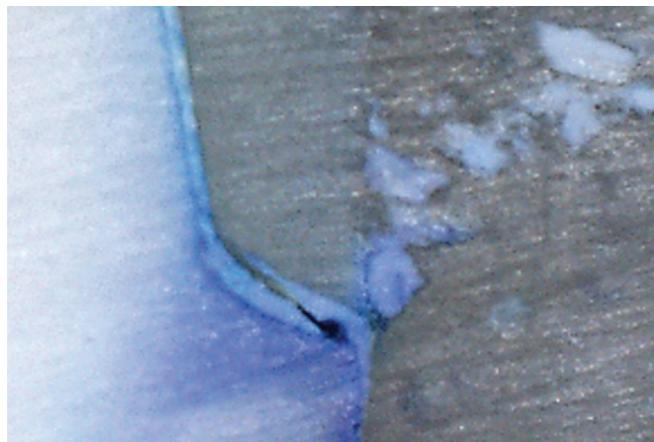
**Table 3:** Summary of Dunn's procedure with a Bonferroni correction test for pairwise comparisons of cement groups

Samples 1–2	Test statistic	Std. error	Std. test statistic	Sig.	Adj. Sig.
Resin–GI	6.900	2.772	2.489	0.013	0.038*
Resin–ZONE	–8.100	2.772	–2.922	0.003	0.010*
GI–ZONE	–1.200	2.772	–0.433	0.665	1.000

\*Statistically significant difference ( $p < 0.05$ ); GI: Glass ionomer cement



**Fig. 4:** Stereomicroscopic image showing the quality of CAD/CAM-fabricated interim crown [group 2 (resin-based cement – UltraTemp® REZ)]



**Fig. 5:** Stereomicroscopic image showing cracks in the cement film due to thermal stresses [group 3 (ZONE cement – TempBond® NE)]

In general, glass ionomer-based cements are one of the most popular materials for permanent cementations used in clinics.<sup>7</sup> Their characteristics include excellent biocompatibility i.e., nonirritating and low cytotoxicity to tooth structure. The tooth-like coefficient of thermal expansion maintains the marginal seal, and their chemical adhesion to teeth is achieved via polyacrylic acid bonds to calcium ions on the surface of enamel or dentin; these cements also possess high dimensional stability in wet environments and undergo very little expansion during setting (only 1.7–1.8%). In addition, ion leaching and fluoride releases, which are from the fluoroaluminosilicate glass component, increase the resistance of enamel and dentin to acid dissolution and act as a bacteriostatic agent.<sup>5,23,24</sup> The glass ionomer-based provisional cement used in this study had the same composition as conventional glass ionomer cements with the addition of glycerin. As noted above, it is available as a dual-cartilage paste–paste system and not as a powder liquid system like other glass ionomer-based luting cements. Unfortunately, this provisional cement exhibited a high microleakage score, which was not significantly different from that of the ZONE cement. This result may be due to the high solubility of the glass ionomer cements in water; its setting reaction is sensitive to moisture conditions, especially during its initial set stage and during the first 24 hours. In this experimental setup, the initial crown cementation was performed in a dry environment. After 10 minutes, the specimens were placed in a water bath without applying a protective coat of varnish, petroleum jelly, or resin on the crown margins; therefore, the cement was subjected to both gain and loss of water. This process led to high solubility and increase

in microleakage.<sup>25</sup> Another explanation is the presence of glycerin in the cement formula – a highly hygroscopic substance – that led to more water absorption and therefore high cement solubility and voids in the cement.<sup>26</sup> One more explanation is that the extracted teeth used in this study had low water content, which was accompanied by drying of the prepared teeth prior to cementation and resulted in lower bond strengths between the cement and the tooth, leading to higher marginal microleakage. The same finding was reported by Rosenstiel and Rashid<sup>27</sup> who found that excessive dryness leads to lower bond strengths and postoperative sensitivity in crowns cemented with glass ionomer cement.

The ZONE cement exhibited the highest microleakage score of all cements examined in this study, which is consistent with previous reports.<sup>11,12</sup> Its high solubility, inferior flow properties, larger film thickness, and weaker mechanical properties are major drawbacks that result in its poor sealing ability. Furthermore, despite the specimens not being subjected to any mechanical loading, it was obvious in some of the stereomicroscope photographs of the ZONE group that the thermal cycles alone produced significant strains and microshifts of the restoration interfaces, thereby causing cracks in the cement film (Fig. 5). These cracks are an indication of weak mechanical properties combined with a mismatch of the linear coefficient of thermal expansion among the cement, the tooth structure, and the provisional resin crown.<sup>28</sup>

Although every effort was made to simulate the oral environment, real clinical scenarios are too complex to be fully reproduced by experimental setups; it is important to address the limitations of this *in vitro* study of microleakage assessment. First, the initial setting of the cement in a lab environment, which has a lower temperature and humidity than intraoral conditions, may lead to cement desiccation, which may affect the initial setting reaction and therefore cement properties. Second, the presence of

mastication forces in clinical situations and the presence of positive intrapulpal pressure and continual outflow of fluids and plasma in dentinal tubules, which have a dramatic effect on cement adhesion to tooth structure and the microleakage score, were not simulated in this experimental setup.<sup>29</sup> Furthermore, the dimensions of methylene blue were extremely small compared with those of a typical bacterium (500–100 nm). Therefore, the microleakage scores of *in vitro* dye penetration studies may not be clinically relevant.<sup>30</sup> Additional investigations and clinical studies with larger sample sizes are necessary in order to gain more insights into the clinical performance and sealing abilities of glass ionomer-based provisional cements.

## CONCLUSION

Within the limitations associated with this *in vitro* study, resin-based provisional cements exhibited significantly less microleakage after the dye penetration test compared with glass ionomer-based provisional cements and ZONE-based provisional cements.

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