ORIGINAL ARTICLE

Microleakage of zirconia frameworks cemented with two types of phosphate monomer-based resin cements

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ABSTRACT

Objective: Resin cements containing phosphoric acid modified methacrylate monomers are commonly used for zirconia-based restorations. However, there are few studies of microleakage of zirconia frameworks cemented with these types of resin cements. The purpose of this study was to compare microleakage of zirconia frameworks cemented with two types of phosphate monomer-based resin cements after long-term thermocycling. **Materials and Methods:** Totally, 30 permanent premolars were randomly divided into two groups: Self-etching (SE) (Panavia™ F2.0, Kuraray Medical, Japan) and self-adhesive (SA) (RelyX™ U100, 3M ESPE, USA) resin cements. The teeth were prepared for zirconia frameworks, which were fabricated by TDS CAD/CAM. After cementation and thermocycling (20,000 cycles), specimens were immersed in 2% methylene blue dye solution for 24 h and were sectioned mesiodistally and buccolingually. Microleakage was recorded, and the data were analyzed using the Mann–Whitney U-test and Sign test ($\alpha = 0.05$). **Results:** All specimens showed significantly higher microleakage scores at the cementum margins than at the enamel margins ($P < 0.05$). The SE resin cement provided significantly lower microleakage scores than the SA resin cement at the enamel margin ($P < 0.05$); however, there were no statistically significant differences in microleakage between two resin cements at the cementum margins ($P > 0.05$). **Conclusion:** SE resin cement has a better sealing ability than SA resin cement at the enamel surface. Both resin cements presented high microleakage at the cementum margins, especially at the tooth/resin interface.

Key words
Phosphate based resin cement, resin cements, zirconia crown

INTRODUCTION

The popularity of zirconia ceramics for restorative dentistry has increased due to their superior mechanical properties, which are attributed to the unique characteristic known as transformation toughening. Apart from the mechanical properties and esthetics, the long-term clinical success of all ceramic restorations can be influenced by marginal adaptation. Poor marginal adaptation results in secondary caries, periodontal disease, pulp sensitivity, pulp necrosis, and esthetic problems.[1]

Microleakage can be defined as the passage of bacteria, fluids, molecules or ions between the tooth structure and the restorative material applied to it.[2] Several factors influence microleakage such as dental restorations, luting agents, and tooth structures. Zirconia is a nonsilica based ceramic and thus cannot be etched with hydrofluoric acid. Therefore, luting agents are important to improve the retention of zirconia ceramics. Several articles have recommended using phosphate monomer-based resin cements for luting zirconia-based restorations.[3-5] Resin cements containing phosphate esters of acidic monomers result in chemical bonding to metal oxides. Examples of phosphate monomer-based resin cements include Panavia™ F2.0 (Kuraray Medical, Tokyo, Japan) and RelyX™ U100 (3M ESPE, Minneapolis, MN, USA), which are self-etching (SE) and self-adhesive (SA) resin cements, respectively.

Some studies have reported acceptable bond strength between zirconia frameworks and tooth surfaces when bonded with phosphate base resin cements.[6,7] However, Ortorp *et al.*[8] found that approximately 7% of zirconia restorations failed due to loss of retention within 3 years and 27% of 3–5-unit zirconia bridges developed secondary caries after 10 years.[9] In addition, there are only a limited number of studies of the microleakage of zirconia

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ceramics after long-term thermocycling. Therefore, the aim of this study was to compare the microleakage of zirconia frameworks using two types of phosphate monomer-based resin cements. The null hypothesis was that there is no difference between the microleakage of zirconia frameworks cemented with two different types of phosphate monomer-based resin cements.

MATERIALS AND METHODS

Totally, 30 permanent upper premolars without any lesions were stored in 10% formalin solution for 2 weeks.[10] They were embedded into clear epoxy resin and positioned with the long axis of the teeth perpendicular to the floor of the mold. The abutment teeth were prepared using an operating instruction SCHICK-ceramic-milling set; Master S3-Nr. 2650 (Schick-Dentalgeräter, Schemmerhofen, Germany) and diamond rotary cutting instruments under water cooling. Rounded-shoulder finishing margins (1.0 mm circumference) with 6° convergence angles were produced at the cementoenamel junction (CEJ) on the proximal surfaces of the preparation using high-speed, round-end, tapered diamonds (number 837314016; Jota AG, Rüthi, Switzerland) with diameters of approximately 1.0 mm and 6° of taper. Preparation heights of 4.0 mm were achieved using high-speed, cylindrical diamonds (number 837314016; Jota AG). The transition from the axial to the occlusal surfaces was rounded with round white stones (number3 -10-02-03; Accord, Bangkok, Thailand). During the study, all specimens were stored in distilled water and kept in an incubator at 37°C. All prepared specimens were sent to the dental laboratory to fabricate the zirconia frameworks via the TDS® CAD/CAM (Spec dental lab, Bangkok, Thailand). A wall thickness of 0.5 mm and a cement space of 30 μm were chosen for the frameworks. The flat occlusal surface of the frameworks was prepared parallel to the horizontal plane. There was no surface treatment of the zirconia frameworks. The specimens were divided into two groups (n = 15): SE group and SA resin cements (SA group) [Table 1]. The zirconia frameworks were cemented according to the cement manufacturers' instructions. Panavia™ F2.0 (Kuraray Medical, Tokyo, Japan) was used in the SE group. Liquid A and liquid B (ED primer 2.0) were mixed in 1:1 ratio and applied to the tooth surfaces. Paste A and paste B were then mixed in a 1:1 ratio (within 20 s) and applied on the inner surfaces of the framework. The zirconia framework was then seated on the abutment teeth, using a loading device, for 7 min with a load of 50 N.[11,12] The cement was partially light-cured for 3 s, and the excess cement removed with the aid of an explorer. The cement was then light-cured for 40 s on all four sides of the specimen to accelerate polymerization. Finally, a protective gel, Oxyguard II® (Kuraray Medical) was applied on the margin for 3 min to prevent the formation of an oxygen-inhibited layer and then rinsed it off with distilled water. RelyX™ U100 (3M ESPE; St. Paul, MN, USA) was employed in the SA group. This cement does not require any prior tooth surface treatment before bonding. Base and catalyst pastes were mixed together on a mixing pad for 20 s. Resin cement was applied on the inner surface of the framework and the process utilized was the same as with the SE group. After 24 h storage in distilled water at 37°C, all specimens were thermocycled for 20,000 times between 5°C and 55°C with a dwell time of 20 s in each bath. The interval time between baths was 10 s.[13]

After thermocycling, the specimens were dried, and the root surfaces were covered with two layers of acrylic fingernail polish. A 1 mm window was created, situated below the crown margin to prevent dye penetration to other areas of the specimens. The teeth were immersed in 2% methylene blue dye solution for 24 h and kept in an incubator at 37°C. After rinsing off the methylene blue dye solution with water for 30 s, the teeth were dried and embedded in clear epoxy resin to prevent fracture of the de-bonded framework during sectioning.

The embedded specimens were sectioned buccopalatally and mesiodistally by an Accutom-50 precision cut-off and grinding machine (Struers, Compenhagen, Denmark) with a water-cooled diamond saw (Struers, Compenhagen, Denmark) to produce four pieces (eight surfaces) of specimens [Figure 1]. Each surface was evaluated for the extent of dye penetration at the margins along the tooth-cement interface under an Eclipse E400 POL microscope (Nikon, Tokyo, Japan) at ×250 magnification. The CEJ line was not straight because the rounded

Table 1: Composition of two resin cements tested

Materials	Compositions	Batch number	Manufacturers
SE primer (ED primer 2.0)	Primer A: HEMA, 10-MDP, 5-NMSA, water, accelerator Primer B: 5-NMSA, accelerator, water sodium benzenesulfinate	051357	Kuraray Medical Inc., Okayama, Japan
Panavia F2.0 (SE resin cement)	Paste A: 10-MDP, dimetacrylates, silanated silica, chemical and photoinitiators Paste B: Dimethacrylates, sodium aromatic sulfate, accelerator, sodium fluoride, silanated barium glass	051357	Kuraray Medical Inc., Okayama, Japan
RelyX U100 (SA resin cement)	Base: Glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated silica, sodium persulfate Catalyst: Glass fiber, dimethacrylates, silanated silica, p-toluene sodium sulfate, calcium hydroxide	357659	3M ESPE, St. Paul, MN, USA

SE – Self-etching; 10-MDP – 10-Methacryloyloxydecyl dihydrogen phosphate; HEMA – 2-hydroxyethyl methacrylate; 5-NMSA: N-methacryloyl 5-aminosalicylic acid; SA – Self-adhesive

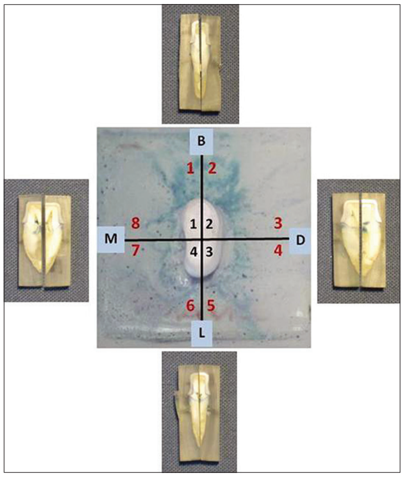

Figure 1: Specimen was sectioned buccopalatally and mesiodistally to produce four pieces. A total of eight surfaces were measured

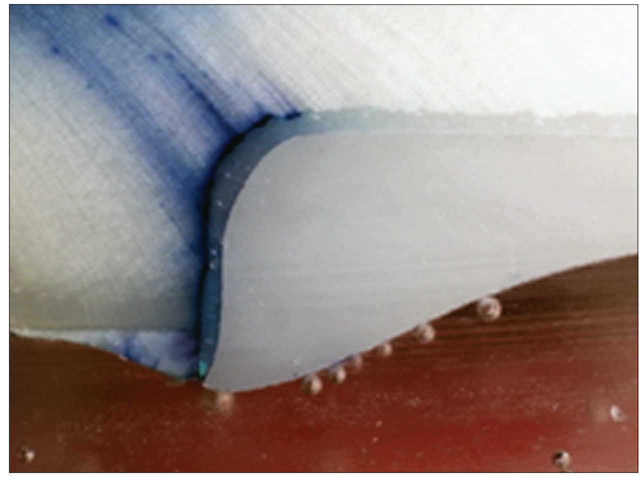

Figure 2: Representative specimen of the self-adhesive group shows dye penetration along the margin at the resin cement/tooth interface and penetrating along the full length of the cervical shoulder (score 3)

shoulder finishing margin was prepared at the CEJ on the proximal surfaces. As such, the buccal and palatal aspects were located at the enamel margins while the mesial and distal aspects were located at the cementum margins.

The extent of dye penetration within each surface of the sections was evaluated and recorded by one operator according to the following scores:

- 0 = No leakage
- 1 = Leakage within one-third of the cervical shoulder
- 2 = Leakage within two-thirds of the cervical shoulder
- 3 = Leakage along the full length of the cervical shoulder
- 4 = Leakage up to one-third of the axial wall
- 5 = Leakage up to two-thirds of the axial wall
- 6 = Leakage along the full length of the axial wall
- 7 = Leakage extending onto the occlusal aspect.

The microleakage scores between two groups were analyzed using the Mann–Whitney U-test. In addition, the microleakage scores at the enamel and cementum within the same group were analyzed using Sign test.

RESULTS

The microleakage at the enamel and cementum margins was evaluated using an eight-point scale, and the results are shown in Tables 2 and 3. There was greater dye penetration along the margin at the resin cement/tooth interface than at the resin cement/zirconia interface [Figure 2]. Mean and median of microleakage scores of SE and SA groups are shown in Table 2. The SE group had a slightly lower microleakage score than the SA group at the enamel margin.

About 30% of specimens in the SE group demonstrated a score of 2 with regards to microleakage at the enamel margin. 55% of the SA group demonstrated a score of 3 with regards to microleakage at the enamel margin [Table 4]. In addition, 28.33% of the SE group exhibited a score of 4 with regards to microleakage at the cementum margin while 40% of the SA group exhibited a score of 3 with regards to microleakage at the cementum margin [Table 3].

Table 2: Means (±SD) and medians of microleakage scores of zirconia frameworks luted with two resin cements

Groups	Mean microleakage scores (±SD)		Median		*P*
	Enamel margins	Cementum margins	Enamel margins	Cementum margins	
SE	2.1 (±1.2)	2.9 (±1.4)	2	3	0.004
SA	2.5 (±1.2)	2.9 (±1.5)	3	3	0.002
P	0.021	0.076			

SD – Standard deviation; SE – Self-etching; SA – Self-adhesive

The number of specimens with no microleakage at the enamel margins was higher than at the cementum margins. All resin cement groups demonstrated a maximum microleakage score of 5, indicating that leakage penetrated to two-thirds of the axial wall.

The Mann–Whitney U-test showed that there were statistically significant differences between the two resin cements at the enamel margins ($P < 0.05$). However, there were no statistically significant differences between the two resin cements at the cementum margins ($P > 0.05$). Moreover, the Sign test showed that the microleakage scores at the cementum margins were significantly higher than the enamel margins in both resin cements ($P < 0.05$).

DISCUSSION

The null hypothesis was rejected; there was a significant difference in microleakage between the SE and SA

Table 3: Microleakage scores at cementum margins of two resin cements

Groups	Number of surfaces (%)								Total surfaces
	0	1	2	3	4	5	6	7	
SE	1 (1.7)	11 (18.3)	12 (20)	11 (18.3)	17 (28.3)	8 (13.3)	0 (0)	0 (0)	60 (100)
SA	2 (3.3)	11 (18.3)	3 (5)	24 (40)	10 (16.73)	10 (16.7)	0 (0)	0 (0)	60 (100)

SE – Self-etching; SA – Self-adhesive

Table 4: Microleakage scores at enamel margins of two resin cements

Groups	Number of surfaces (%)								Total surfaces
	0	1	2	3	4	5	6	7	
SE	5 (8.3)	14 (23.3)	20 (33.3)	15 (25)	4 (6.7)	2 (3.3)	0 (0)	0 (0)	60 (100)
SA	4 (6.7)	8 (13.3)	9 (15)	33 (55)	5 (8.3)	1 (1.7)	0 (0)	0 (0)	60 (100)

SE – Self-etching; SA – Self-adhesive

systems at the enamel margin. The microleakage with Panavia F2.0 (SE resin cement) at the enamel margin was less than with the RelyX group (SA resin cement). This is attributed to the SE property of Panavia F2.0, which can slightly demineralize the enamel. However, the microleakage at the cementum margin between the two groups was not significantly different. The cross-sectional views of the specimens in this study showed that the two groups presented with similar microleakage patterns, in that most specimens had preferential dye penetration along the margin at the resin cement/tooth interface compared to the resin cement/zirconia interface [Figure 2]. This was most likely due to the adhesion characteristics of the SA and the SE resin cements where smear layers were still presented on the tooth surface. The SE and SA resin cements were developed to eliminate the clinical steps of etching and rinsing; however, despite the acidity of these two resin cements, they were unable to completely remove the smear layer. RelyX Unicem is an SA resin cement, which consists of alkaline fillers and multifunctional phosphoric acid methacrylates. The latter is responsible for its SE ability. Some studies have shown that this resin cement is unable to demineralize the smear layer completely.[14,15] No distinct decalcification or infiltration of dentin had occurred, and no clear hybrid layer, or resin tags were observed.[14,15] However, this material had been shown to chemically bond with calcium derived from hydroxyapatite, which enhanced the bonding performance.[16] The ED primer 2.0 of Panavia F2.0, which is an SE resin cement, could etch through the smear layer to partially demineralize the underlying dentin.[15] The bonding mechanism with dentin relied on the infiltration of the resin monomers into the partly demineralized collagen meshes, which subsequently formed a hybrid smear layer after polymerization.[15] Microleakage took place at any defect site at the bonding interface, with the resin incompletely infiltrating into demineralized dentin, resulting in permeable hybrid layers.[17] Consequently, the tooth structure beneath restorations could have been demineralized by lactic acid produced by *Streptococcus mutans*. This has been known to result in secondary caries, restoration detachment, and pulpal pathosis.[17,18] It was suggested that the hybridized smear layer may have had an adverse effect on the quality of the bonding interface, as it was the weakest structure. It has been clinically reported that the permeable hybrid layer could be degraded over a 1–3-year period.[19,20]

The results of this study show that both resin cements had significantly higher microleakage scores at the cementum margins than at the enamel margins. This is in agreement with the results of previous studies[17,21-23] Piemjai *et al.*[23] stated that this was caused by differences in compositions of enamel, dentin, and cementum. Dentin and cementum have more organic compositions (primarily of collagen and protein polysaccharides) than enamel.[24] The composition of enamel is largely inorganic hydroxyapatite, and its organic phase is keratinous, not collagenous. Thus, there are no areas around the collagen fibers that can facilitate microleakage, whereas dentin and cementum have spaces remaining around the collagen fibrils in demineralized dentin. This is thought to be the reason suggested for the microleakage occurring at the dentin and cementum margins more than at enamel margins. Therefore, it is suggested that clinicians should be cautious when using these types of resin cements in cases where restorative margins extend below the CEJ.

It should be mentioned that formalin was used as a disinfectant in this study. Some studies reported that formalin could decrease the microleakage due to formaldehyde reacting with several protein molecules, which could lead to the cross-linking of protein molecules followed by collagen fixation.[25,26] However, other studies suggested that formalin did not have any effect on the dentin bond strength.[10,27] Although formalin might have affected the microleakage at the cementum margin, the microleakage observed from the two resin cements was found not to be significantly different ($P > 0.05$) in this study. In addition, the microleakage at the enamel margin was found to be significantly different ($P < 0.05$); however, enamel is mainly inorganic structure. And as stated, even though formalin was used in this study, its effect was negligible.

Another point worth mentioning is that the zirconia framework used in this study had no surface treatment prior to cementation, as this project focused on the effects of the resin cements and microleakage. Sandblasting or air abrasion may interfere with microleakage analysis. Sener *et al.*[28] reported on the microleakage scores of zirconia crowns with no surface treatment and the results showed the highest scores of 3 (leakage along the full length of axial wall) and 4 (leakage over the occlusal surface) in DC-Zircon and Cercon zirconia systems, respectively, which similar to the results in this study. However, another study showed that the inner surface of zirconia (Cercon) with surface treatment (silica coating) presented an average microleakage score of 0.8 (±0.79), indicating the leakage did not exceed 2/3 of the chamfer preparation.[29] Osório *et al.*[30] proposed that Procera crowns had the lowest microleakage when the inner surfaces were treated with aluminum oxide blasting followed by silane application. As such, the microleakage of zirconia restorations may be reduced if their inner surfaces are treated with silica or aluminum oxide.

Several *in vitro* experiments have been performed to evaluate microleakage around restorations including radioisotope study, neutron activation analysis, chemical tracer, bacterial studies, and dye penetration.[31] Dye penetration is a common method for microleakage evaluation employing various tracers (such as basic fuchsin, eosin, methylene blue and silver nitrate).[2,31,32] One study reported that basic fuchsin was used most often in previous studies (40.7%) followed by methylene blue (22%) and silver nitrate (17%).[32] In this study, the teeth were immersed in 2% methylene blue dye solution for 24 h. Heintze *et al.*[33] found that there was greater dye penetration at dentin than enamel margins among basic fuchsin, methylene blue and silver nitrate, but this was not statistically significantly different. They proposed that dyes were probably not suitable for testing the sealing ability of restorations *in vitro* because their molecules were very small, which could lead to an overestimation.[33] Mente *et al.*[34] reported that the molecular size of various dyes ranged from 1 to 2 nm, which is smaller than that of bacteria (200–2000 nm). However, lactic acid produced from cariogenic bacteria may be consistent in size with that of dye. Future study may focus on additional materials used with phosphate-based resin cements in order to improve the marginal integrity and reduce microleakage.

CONCLUSION

Within the limitation of this study, the following conclusions may be drawn:

- Self-etching resin cement had a better sealing ability than self-adhesive resin cement at the enamel margins. Both types of resin cements showed higher leakage at the tooth/resin cement interface than at the zirconia/resin cement interface
- Both resin cements showed significantly higher microleakage at the cementum margins than at the enamel margins.

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