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ORIGINAL RESEARCH

In vitro evaluation of microleakage for different cementation materials used in prefabricated zirconia crowns

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Abstract

Background: Microleakage is a significant factor that leads to the failure of full-coronal restorations. Moreover, the choice of cementation material may affect microleakage. This study aimed to evaluate microleakage in prefabricated zirconia crowns (PZCs) when cemented with different luting cements. **Methods**: Thirty-six freshly extracted primary maxillary incisors were prepared for PZCs. The teeth were randomly divided into three groups (n = 12) to be lutation with one of two different resin-modified glass ionomer cements (Ketac Cem Plus and FujiCEM Evolve) or a self-adhesive resin cement (RelyX U200). After the cementation process, the teeth were thermocycled 6000 times at temperatures ranging from 5 °C to 55 °C with a dwell time of 15 seconds and a transfer time of 10 seconds, immersed in basic fuchsin solution and then sectioned buccolingually. Subsequently, the sections were scored based on observations made using a stereomicroscope at 30× magnification. Statistical analysis was conducted using one-way analysis of variance and the independent two-sample *t*-test. The significance level was set at $p < 0.05$. **Results**: The lowest microleakage score was attained by the self-adhesive resin cement group (mean 1.00). No statistically significant differences were found among the groups ($p = 0.467$). Moreover, no statistically significant difference was observed between the microleakage scores for the primary central and lateral teeth, regardless of the cementation material used ($p = 0.314$). **Conclusions**: According to the outcomes of this study, self-adhesive resin cements may be a promising alternative to resin-modified glass ionomer cements.

Keywords

Cementation; Crown; Dental cements; Microleakage; Primary teeth; Zirconia

1. Introduction

With the concept of preventive dentistry gaining increasing recognition, it is gradually becoming a fundamental aspect of pediatric dentistry. However, despite the use of preventive practices for early childhood caries, it remains one of the most prevalent diseases in children, posing a significant public health problem [1]. Early childhood caries is usually first observed in primary anterior teeth in the upper jaw, after which carious lesions progress rapidly in both anterior and posterior teeth. Notably, severely mutilated primary anterior teeth can lead to phonation difficulties, esthetic losses, loss of self-confidence, development of parafunctional habits, malocclusions, pain and pulp inflammation [2, 3].

The restoration of severely mutilated anterior teeth in primary dentition *involves* difficult treatment processes, primarily owing to the low cooperation of young children and the high esthetic expectations of parents [4, 5]. In pediatric dentistry, multiple options, both intra-coronal and full-coronal restoration, are available for restoring primary anterior teeth. However, for carious lesions that are multisurface and subgingival, full-coronal restorations are recommended since achieving isolation in such cases is difficult and the physical strength of the material used is extremely important. Several methods for full-coronal restoration of primary anterior teeth have been proposed over time. Prefabricated zirconia crowns (PZCs) are among the most popular options, as they provide clinically acceptable, long-term and esthetically successful restoration of the anterior primary teeth in pediatric patients [6]. Some advantages of PZCs include their biocompatibility, smooth surface, high fracture resistance and rapid adaptation to gingival tissue due to comparatively less plaque accumulation [2, 7, 8].

Luting cements fundamentally affect the microleakage, clinical success and longevity of PZCs [8]. Cements establish a mechanical and/or chemical bond between two different materials and fill the gap between the tooth and the restoration, thus protecting the tooth against microleakage [9].

In recent years, several PZC manufacturers have emerged due to the material's increasing use in pediatric dentistry.

Due to the inability to crimp PZC margins, the cementation process necessitates technical sensitivity, and manufacturers may recommend the use of different cements. While some manufacturers recommend bioactive cements or glass ionomer cements (GICs), others suggest resin-modified glass ionomer cements (RMGICs) or resin cements [10]. Nevertheless, there is insufficient evidence to determine the ideal luting cement for PZCs [10, 11].

The null hypothesis tested in this study is that there is no significant difference among cements in terms of microleakage. Although numerous case reports and studies are available pertaining to the cementation of zirconia crowns on permanent teeth, very limited research has been conducted on the cementation of PZCs on primary dentition. Therefore, the present study aimed to evaluate microleakage values observed in PZCs cemented to primary incisors using one of two different RMGICs or a self-adhesive resin cement (SARC).

2. Methods

The protocol of this *in vitro* study was approved by the Ethics Committee of the university to which the authors were affiliated at the time of the study (Decision no: 2020.04.01). This study evaluated the effect of three different luting cements on microleakage in PZCs.

2.1 Sample size

The study design *involved* three groups. A power analysis indicated the number of samples to be a total of 36 teeth, with 12 in each group. The sample size was calculated to have an effect size of 0.55, type 1 error 0.05 and power of 0.82 using G*Power (version 3.1.9.7, Heinrich Heine University Dusseldorf, Dusseldorf, NRW, Germany) [12]. Thirty-six maxillary primary anterior teeth (18 centrals and 18 laterals) were randomly divided into three groups using the randomization procedure according to https://www.randomizer.org.

2.2 Sample selection

A total of 36 freshly extracted primary anterior teeth (18 centrals and 18 laterals) that had been extracted due to trauma or dental caries were carefully cleaned with a scaler to remove soft tissue remnants, disinfected in 0.1% thymol solution and stored in distilled water at 4 °C. Teeth with hypoplasia, fractures, cracks or a root resorption rate of more than one-third were excluded from the study. Before the teeth were further processed, previous restorations or caries were removed until caries-free dentin was exposed. In cases of pulp exposure, an RMGIC (Vitrebond Plus, 3M ESPE, St. Paul, MN, USA) was used to seal the exposed area.

2.3 Preparation of teeth

To prepare the crowns, PZCs with an appropriate mesiodistal size were first selected. All tooth preparations were performed by the same operator (BEAE). Each tooth was securely embedded in self-cured acrylic resin blocks (Integra, BG Dental, Turkey) up to 2 mm below the cemento-enamel junction, positioned upright and centred within the acrylic block. Silicone impression material was used to prepare the molds to ensure the standardization of the acrylic blocks. The teeth were prepared using diamond burs in accordance with the "NuSmile ZR crowns technical guide" [13]. Try-in crowns (NuSmile, TX, USA) were used to control the preparation, and Nusmile ZR PZCs (NuSmile, TX, USA) were used for cementation as the final crowns. The preparation was deemed complete upon confirmation that the crowns were passively seated (Fig. 1). At the end of the preparation process, the teeth were washed using an air-water spray and then dried gently.

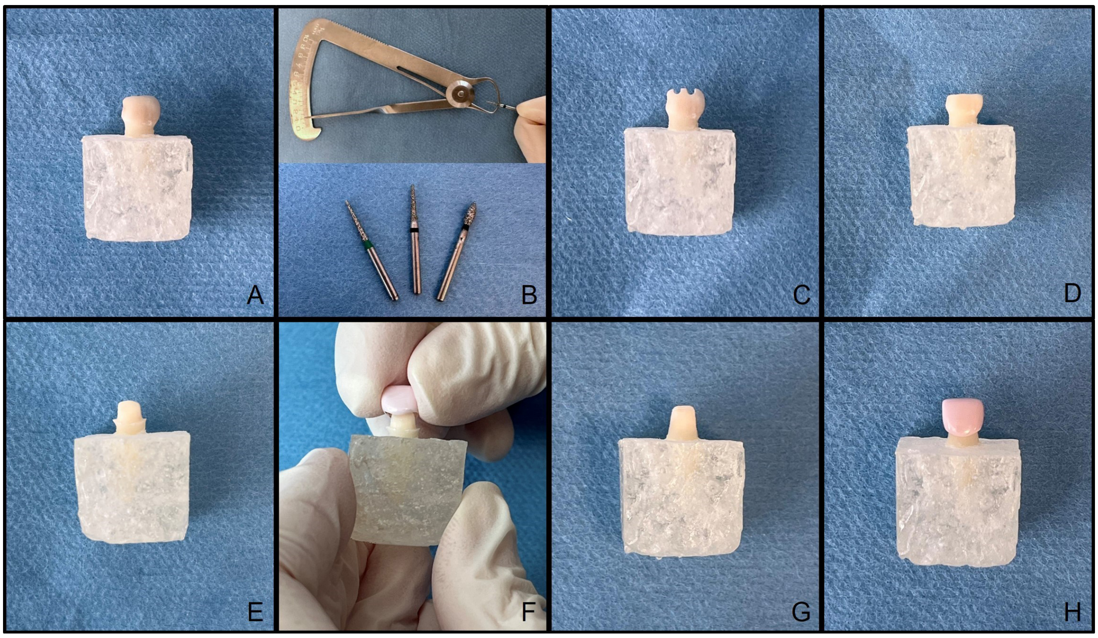

FIGURE 1. Preparation steps for PZCs. (A) Primary incisor tooth before preparation (B) Diamond burs used for preparation (C) Incisal depth cuts (D) Incisal preparation (E) Facial, interproximal and lingual shoulder preparation (F) Test fitting before subgingival preparation (G) Subgingival feather-edge preparation (H) Final test fitting.

2.4 Cementation of crowns

After they were prepared, the teeth were randomly divided into three cementation groups (n = 12 for each group, 6 centrals and 6 laterals): Group 1 used an RMGIC (NC44770, Ketac Cem Plus, 3M ESPE, St. Paul, MN, USA), Group 2 used another RMGIC (2010011, FujiCEM Evolve, GC, Tokyo, Japan) and Group 3 used an SARC (7308167, RelyX U200, 3M ESPE, St. Paul, MN, USA). The chemical compositions of the tested cementation materials are presented in Table 1. Each luting cement material was prepared and used in accordance with the manufacturer's instructions. Subsequently, the PZCs were filled with luting cement according to their respective groups and then placed on the incisors. The teeth were stabilized with finger pressure, and the SARC group was additionally light cured for 20 seconds per surface with an LED dental curing light (Monitex Blue Lex LD-105, Fomed Dental Inc., Taipei, Taiwan). Finger pressure was maintained for 5 minutes after seating the crowns (approximately 8 minutes after the cement mixture was initiated), as recommended by the manufacturer, to ensure complete polymerization. All residual cement was carefully removed with a probe. Following cementation, the samples were allowed to bench set for 1 hour. Subsequently, all samples were stored in distilled water at 37 °C for 24 hours. Afterward, they were thermocycled 6000 times at temperatures ranging from 5 °C to 55 °C with a dwell time of 15 seconds and a transfer time of 10 seconds.

2.5 Microleakage tests

After thermocycling, the root surface was coated with two layers of acrylic varnish to within 1 mm of the restoration margin, and the apical foramen was covered with dental wax to prevent dye leakage. Subsequently, the prepared specimens were kept in a 2% basic fuchsin dye solution (V7N548058A, Carlo Erba Reagents GmbH, Val de Reuil, France) for 24 hours and then rinsed. Next, the teeth were embedded in acrylic blocks (Integra, BG Dental, Turkey) and sectioned buccolingually into two parts at the mesiodistal midpoint using a microcut device (Micracut 201, Metkon Instruments, Bursa, Turkey) with water cooling. Dye penetration in the sections was assessed using a stereomicroscope (Nexius Zoom, Euromex, Arnhem, Holland) at 30× magnification. The scoring was conducted by a blinded observer, following the criteria proposed by Stepp *et al.* [12]. The microleakage scoring criteria are presented in Table 2.

2.6 Statistical analysis

The obtained data were analyzed using IBM SPSS V23 (Armonk, NY, USA). Compliance with normal distribution was determined using the Shapiro-Wilk test. One-way analysis of variance was employed to compare the normally distributed scores among the three groups. The independent two-sample *t*-test was used to analyze normally distributed scores between paired groups. When evaluating the study outcomes, a significance level of $p < 0.05$ was considered.

3. Results

There was no failure in any of the sample during the tests. The descriptive statistics (mean and median microleakage scores and standard deviations) of the three groups (n = 12 for each group) are presented in Table 3. The mean microleakage scores were 1.58 for Group 1, 1.33 for Group 2 and 1.00 for Group 3. These results showed comparable microleakage values, with no statistically significant differences among the groups ($p = 0.467$). In particular, the lowest microleakage value was observed for Group 3, followed by Group 2 and Group 1. In the microscopic evaluation, the minimum microleakage score was 0 (Fig. 2A) and the maximum score was 4 (Fig. 2B).

Table 4 presents the comparison statistics pertaining to pairwise groups, exhibiting no statistically significant differences in microleakage scores ($p > 0.05$). Moreover, no statistically significant difference was observed between the scores for the primary central and lateral teeth, regardless of the cementation material used ($p = 0.314$).

TABLE 1. Chemical composition of the tested luting cements in the present study.

Material	Manufacturer	Lot no	Composition
RMGIC (Ketac Cem Plus)	3M ESPE, St. Paul, MN, USA	NC44770	Paste A: Radiopaque fluoro-alumino-silicate glass, 2-hydroxyethyl methacrylate (HEMA), water, reducing agent, opacifying agent, dispersion aid. Paste B: Methacrylated polycarboxylic acid, nonreactive zirconia silica filler, HEMA, bisphenol A-glycidyl methacrylate (Bis-GMA), water, potassium persulphate, photoinitiator.
RMGIC (FujıCEM Evolve)	GC, Tokyo, Japan	2010011	Paste A: Fluoro-alumino-silicate glass, HEMA, dimethacrylate & bisphenol-A ethoxylate dimethacrylate (Bis-MEPP), inhibitor, silicon dioxide, photoinitiator, pigments. Paste B: Polyacrylic acid, polybasic carboxylic acid, water, ytterbium fluoride, initiator, silicon dioxide.
SARC (RelyX U200)	3M ESPE, St. Paul, MN, USA	7308167	Base: Methacrylate monomers containing phosphoric acid groups, silanated fillers, methacrylate monomers, initiator components, rheological additives, stabilizers. Catalyst: Methacrylate monomers, alkaline (basic) fillers, silanated fillers, stabilizers, initiator components, rheological additives, pigments.

TABLE 2. Microleakage scores.

Scores	Criteria
0	Dye penetration at crown margins only
1	Dye penetration at crown margins and around cement
2	Dye penetration at crown margins and throughout cement
3	Dye penetration to 1/3 of tooth structure
4	Dye penetration throughout tooth structure and pulp

TABLE 3. Mean microleakage scores and standard deviation of three groups.

Cements	Number of samples	Mean ± SD	Median (Min–Max)	One-way analysis	*p* value
Group 1 (Ketac Cem Plus)	12	1.58 ± 1.16	1.00 (0.00–4.00)	$F = 0.778$	$p = 0.467$ NS
Group 2 (FujiCEM Evolve)	12	1.33 ± 1.07	1.00 (0.00–3.00)		
Group 3 (RelyX U200)	12	1.00 ± 1.21	0.50 (0.00–3.00)		

F: One-way analysis test statics; SD: standard deviation; NS: not significant; Min: minimum; Max: maximum.

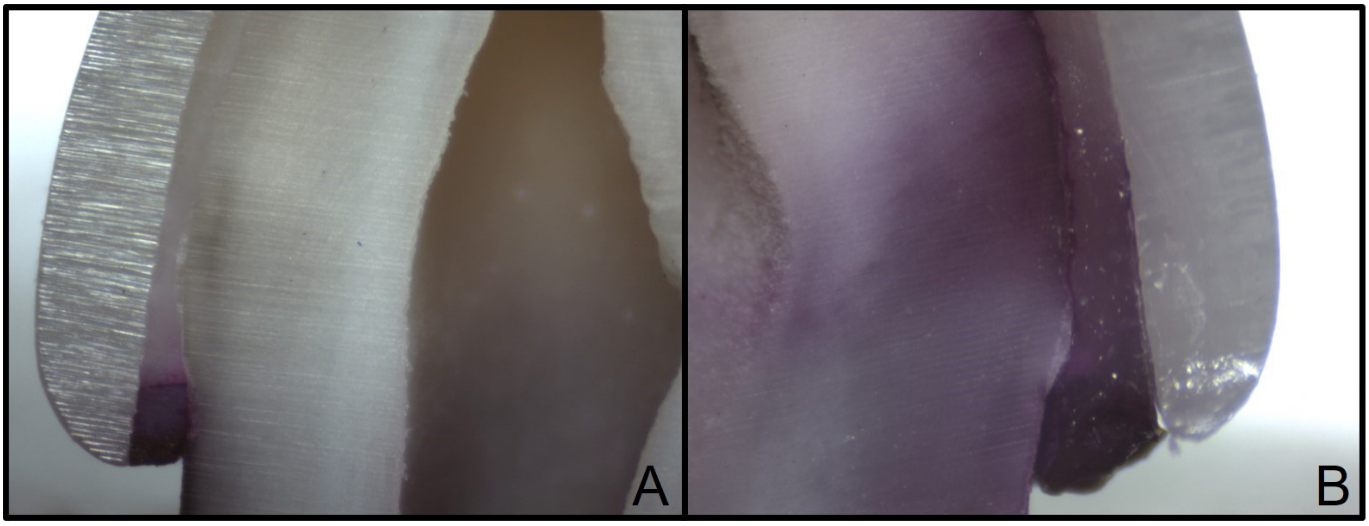

FIGURE 2. Representative microscopic images taken from sections at 30× magnification. (A) Score 0: Dye penetration at crown margins only. (B) Score 4: Dye penetration throughout tooth structure and pulp.

TABLE 4. Comparison of pairwise groups.

Cements	Mean ± SD	*t*-test statics	*p*
Group 1 (Ketac Cem Plus)	1.58 ± 1.16	$t = 0.547$	0.590 NS
Group 2 (FujiCEM Evolve)	1.33 ± 1.07		
Group 1 (Ketac Cem Plus)	1.58 ± 1.16	$t = 1.205$	0.241 NS
Group 3 (RelyX U200)	1.00 ± 1.21		
Group 2 (FujiCEM Evolve)	1.33 ± 1.07	$t = 0.715$	0.482 NS
Group 3 (RelyX U200)	1.00 ± 1.21		

(t-test: independent two-sample t-test; SD: standard deviation; NS: not significant).

4. Discussion

Zirconia, which possesses esthetic characteristics similar to those of natural teeth, is a prosthetic material that exhibits high compatibility with gingival tissues and offers superior mechanical strength [14, 15]. Zirconia crowns have long been employed by dentists to restore teeth in permanent dentition [5]. Currently, PZCs are used as durable and esthetic restorative materials for anterior and posterior teeth in primary dentition [10]. A clinical study published in 2023 showed that PZCs on primary molars are more durable and maintain their retention longer than those on primary anterior teeth [6]. This result suggests that the choice of luting cement plays a lesser role in primary molars than in primary anterior teeth. Therefore, primary anterior teeth were chosen for investigation in this study instead of molars. In addition, in clinical studies evaluating PZCs on primary anterior teeth, primary incisors (central and lateral) have frequently been assessed [16–19]. We hypothesized that the exclusion of canines might be due to potential anatomical and occlusal differences; therefore, we did not include primary canines in the study methodology.

Choosing the appropriate luting cement is a crucial factor in ensuring long-lasting restoration. Recent studies have highlighted the significance of choosing the appropriate luting cement for the long-term success and survival of PZCs [8, 11, 20, 21]. The ideal cementation material should ensure durable adhesion between the restoration and the tooth structure while also preventing microleakage [22]. However, comparative studies evaluating microleakage in PZCs luted with different cements are quite limited [8, 12, 20, 23]. To our knowledge, few studies in the literature have investigated microleakage in PZCs applied to anterior teeth in primary dentition [8]. Therefore, this *in vitro* study was designed to provide clinicians with new insights into the cementation of PZCs in primary teeth. For this purpose, two different RMGICs, which are commonly used in similar clinical indications and are particularly recommended for zirconia crowns, were compared with each other and with an SARC, whose use in pediatric dentistry has increased in recent years.

The findings showed that the SARC exhibited lower microleakage values than the RMGICs, but there were no significant differences among the groups. Based on this result, the null hypothesis tested in the study was accepted. Along the same line, in a study published in 2024, the microleakage values of PZCs and custom-made zirconia crowns applied to primary anterior teeth using different cements were compared. For both types of crowns, RMGIC exhibited higher microleakage values compared to SARC; however, the difference was not found to be statistically significant [8]. Similarly, in their *in vitro* study on primary molar teeth, Al-Haj Ali and Farah [23] assessed the microleakage values of PZCs cemented using GIC, RMGIC or SARC. The researchers observed higher microleakage values in the RMGIC group than in the SARC group, although there was no significant difference. The findings of this study are consistent with those of the present study. In a study investigating the microleakage levels of various cements used in PZCs on primary molar teeth, the researchers found that SARC exhibited significantly lower microleakage values compared to both GIC and RMGIC [20]. Furthermore, the results of two studies in which stainless steel crowns were cemented with SARC or RMGIC showed that the SARC group had lower microleakage values, but the difference was not significant [24, 25]. Overall, the findings of these studies are similar to those of the current study.

Previous studies that examined the microleakage values of crowns cemented on permanent teeth using different kinds of luting cement also presented findings similar to those of the present study [26, 27]. Chang *et al*. [26] found that when zirconia crowns were cemented with SARC or RMGIC, there was no difference in microleakage values between the groups. In another *in vitro* study examining the microleakage values in cast metal crowns cemented with different cements, resin cement achieved the lowest microleakage score, followed by RMGIC and GIC, although the differences among the groups were not statistically significant. The researchers of this study attributed the results to the filler content of the resin cement, which improved the marginal wear resistance [27].

Furthermore, variations in thermal expansion coefficients and the dimensional change of cements can affect microleakage values [27, 28]. In the current study, thermocycling was applied to simulate temperature changes that occur in the mouth. As a result of these temperature changes, microleakage may occur at the restoration margin, as differences in the thermal expansion coefficients lead to dimensional changes [28]. In this context, Sidhu *et al*. [29] reported that while tooth tissue and resin cements expand at high temperatures, RMGIC contracts when exposed to such temperatures. This leads to stress at the tooth-cement interface at the restoration margin, which increases the risk of microleakage. Therefore, the stress exerted by temperature changes during thermocycling is higher in the RMGIC groups than in the SARC group [26].

The bonding strength of cements to tooth tissues and zirconia may also affect microleakage [30]. SARCs have two bonding mechanisms with dental tissues, the first of which is the chemical interaction between the phosphoric acid groups in the cement content and the hydroxyapatite in dental tissues [22]. Additionally, SARCs can remove part of the smear layer and infiltrate the dental tissue without any pretreatment as a result of methacrylate monomers, which contain phosphoric acid groups, thus enabling micromechanical bonding [22, 31]. This high bond strength allows effective hermetic sealing of both the dental tissue and the cement interface, resulting in low microleakage values [32]. In previous studies that examined the bond strength between zirconia and primary teeth when luted with different cements, the SARC groups exhibited higher bond strength than the RMGIC groups ($p >$ 0.05) [11, 14]. Similarly, Prylinska-Czyzewska *et al*. [30] investigated the shear bond strength between bovine teeth and zirconia cemented with different materials and showed that SARC had a significantly higher bond strength than RMGIC. In a systematic review and meta-analysis published in 2024, SARCs were found to provide significantly higher immediate and delayed bond strength to zirconia than RMGICs [33].

The lower microleakage values obtained for the SARC group may be explained by the fact that the RMGICs used in Groups 1 and 2 contained 2-hydroxyethyl methacrylate (HEMA). Since HEMA is hydrophilic, it may have adversely affected some of the physical properties of the luting cement. Although HEMA usually appears to compensate for polymerization shrinkage by absorbing water in the early stages, continued water absorption during ageing processes, such as thermocycling, can have negative effects on the material's structure. In this context, researchers have reported that hydrophobic resin cements can help reduce the likelihood of failure of the tooth-cement interface during thermocycling [34]. Although no statistically significant difference was observed between the two RMGICs used in this study in terms of microleakage values, lower values were obtained in Group 2. These results are thought to be attributed to the characteristic differences of the monomers in the composition of the RMGICs. The RMGIC in Group 1 contains bisphenol A-glycidyl methacrylate (Bis-GMA), whereas the RMGIC in Group 2 contains bisphenol-A ethoxylate dimethacrylate (Bis-MEPP). In this context, Bis-MEPP, defined as a rigid and hydrophobic monomer, has been reported to have lower water absorption and solubility compared to Bis-GMA, and it has been suggested that this may influence the mechanical properties of the cement [35–37]. In addition to the comparison of microleakage values, there is a difference of approximately 45 seconds between the groups in terms of working and setting time (the time from the initiation of cement mixing to final set). This difference is largely due to the rapid polymerization of the RMGIC in Group 2. From the perspective of pediatric dentistry, it is thought that this situation may be clinically significant.

In full-coronal restorations, cement plays a fundamental role in ensuring ideal marginal adaptation and preventing microleakage [20]. In their study, Al-Haj Ali and Farah [23] inspected different prefabricated crowns luted with different cements. They found that PZCs showed statistically significantly higher microleakage values than stainless steel crowns cemented with the same materials. This finding may be attributed to differences in marginal adaptation. Moreover, since it is impossible to shape PZC margins, due to which a passive fit must be provided, the choice of luting cement becomes even more crucial for PZCs.

It is well known that *in vitro* studies cannot fully reflect clinical conditions and therefore cannot be equated with the results obtained in clinical studies. Although no single *in vitro* laboratory test can fully predict the clinical outcomes of full-coronal restorations, *in vitro* studies can provide clinicians with crucial insights into the factors that may influence these clinical outcomes. Among the limitations of this study, the use of the dye penetration technique should be noted. Although this technique is commonly used in microleakage studies, the findings of a dye penetration-based microleakage test should not be considered a definitive measure for determining the clinical performance of cements [38]. Another limitation of this study is the inability to fully standardize cement thickness in PZCs. To minimize this limitation, all preparations were performed by a single operator. However, this variable can only be fully standardized with custom-made crowns. Nevertheless, microleakage values may not always show proportional results to cement thickness. In a study evaluating the effect of different marginal cement thicknesses on microleakage in zirconia crowns, it was reported that crowns with a 25 μm cement gap exhibited significantly lower microleakage values compared to crowns with no cement gap [39]. Additionally, a study in the literature comparing microleakage values between prefabricated and custom-made zirconia crowns found no significant difference between the two types of crowns, except for the groups cemented with RMGIC [8]. Another limitation is that the study results may have been influenced by the time elapsed between tooth extraction and restorative procedures, as well as by the differences in application under clinical conditions. The small sample size is another noteworthy limitation. This situation was caused by the difficulty in collecting primary anterior teeth due to rapid root resorption and excessive tissue loss. Conducting studies with larger sample sizes would provide more reliable data for clinicians. Moreover, in addition to the materials used in our study, we believe that incorporating bioactive cement could enhance the findings and serve as a valuable direction for future research.

5. Conclusions

Within the limitations of the present study,

- SARC demonstrated lower microleakage values compared to RMGICs, although no significant differences were found among the groups.
- SARCs may be considered a promising alternative to RMGICs in PZC cementation.
- The outcomes of this study may provide valuable guidance to clinicians in the selection of luting cement for PZCs and should be supported by further *in vitro* and *in vivo* studies to increase the potential for clinical success.

AVAILABILITY OF DATA AND MATERIALS

The datasets generated and analyzed during the current study are not publicly available due to privacy or ethical restrictions but are available from the corresponding author on reasonable request.

AUTHOR CONTRIBUTIONS

BEAE and AAO—designed the research study. BEAE—performed the research, analysed the data and wrote the manuscript. AAO—contributed to acquisition and analysis of the data. All authors contributed to editorial changes in the manuscript. All authors read and approved the final manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This *in vitro* study protocol was approved by the Ethics Committee of Kırıkkale University (Decision no: 2020.04.01; Date: 20 May 2020). Written informed consent was obtained from each legal guardian before tooth extraction.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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