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Microleakage of Gradient Versus Translucent Monolithic Zirconia Crowns Bonded with Self-Adhesive Resin Cement

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ABSTRACT

Background: One of the most influencing factors on the clinical longevity of indirect restorations is marginal microleakage. the presence of a marginal gap results in exposure of the cement at the margins and its dissolution, followed by secondary caries, periodontal disease, and microleakage. Data regarding microleakage of gradient monolithic zirconia have been deficient. ***Aim of the study:*** To assess the microleakage of monolithic crowns made of gradient zirconia (IPS E-max ZirCAD Prime, Ivoclar) compared to crowns made of Translucent 5Y zirconia (UTML, Katana) bonded with self-adhesive resin cement (Theracem, Bisco). ***Materials and methods:*** Sixteen monolithic zirconia crowns were constructed using the CAD/CAM system and divided randomly into two equal groups according to the type of zirconia. Group I: ZirCAD Prime crowns (n=8), while group II: UTML zirconia single crowns (n=8). Both groups were bonded with Theracem self-adhesive resin cement (Bisco) to 16 extracted first premolar teeth. After cementation, the teeth were subjected to thermocycling for 5000 cycles at changing temperatures between 5°C and 55°C. The microleakage was measured by immersing the samples in 0.5% basic fuschin dye for 48 hours at 37°C. After the dye penetration, the crowns were sectioned vertically by Isomet Saw in a bucco-lingual direction to measure microleakage using stereomicroscope at 40X magnification. ***Results:*** There was no significant difference between the leakage scores of the two tested groups. ***Conclusion:*** Both Groups fell within microleakage scoring 1 &3, which indicated the extension of the dye only within the finish line area.

Keywords: Marginal fit, gradient zirconia, micro leakage**,** self-adhesive resin cement.

INTRODUCTION

For years, porcelain-fused-to-metal (PFM) restorations have occupied the "gold standard" in prosthetic dentistry due to their good mechanical properties and clinically satisfactory marginal and internal adaptation. However, the patients' need for natural-appearing restorations has led to the development of new ceramic materials with improved mechanical characteristics and suitable longevity.[1]

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Ceramic materials have become more and more popular over the past decade. Their development has produced materials with plenty of advantages, such as translucency, color stability, biocompatibility, low thermal conductivity, and high wear resistance.[2]

Generations of zirconia have shown different proportions of yttria. The mole % of yttria is an expression of the mechanical and physical properties of the zirconia. Zirconia containing 3 mole % yttria is the strongest but most opaque; while zirconia containing 5 mole % yttria gives a more translucent material. Yttria increases the zirconia grain size and lowers the coefficient of thermal expansion. [1,3]

The newest innovation of zirconia can be used as a monolithic restoration. It combines 3Y-TZP and 5Y-TZP together in one blank The top, or enamel zone, of these recently developed strength-graded zirconia blocks is composed of a more translucent zirconia 5Ywhich has a high cubic phase composition and is very translucent, while the bottom, or dentin zone, is composed of a stronger zirconia 3Y. which is a tougher dental ceramic used.[4]

Zirconia restorations are made by a subtractive process that involves milling a blank using CAD/CAM technology, which offers a number of benefits over conventional methods, including quality, speed, and ease of use. Compared to traditional impressions, digital scans are quicker and simpler since they do not require casting, wax-ups, investing, casting, or burning.[5]

The primary function of the luting agent is to retain the crown in place and fill the space between the tooth preparation and restoration, thus avoiding dislodgement of restoration during function.[6] Several resin cements are currently on the market for dental use. Its key benefits are low solubility, micromechanical and adhesive bonding, almost insoluble, and high resistance since, no matter how thin the restoration is, resistance is acquired when it is cemented because it receives a reinforcement structure and high resistance compression.[7] Surface pretreatment techniques had no detrimental effects on flexural strength according to Blatz et al.[8] in 2022. The bonding techniques used on 3Y-TZP zirconia also work well on 4Y-TZP and 5Y-TZP zirconia. Ceramic Primers have recently been introduced, showing high efficiency when used, especially those including phosphate monomers, e.g., MDP. Sandblasting and MDP work together to guarantee that both Zirconia dioxide and MDP are chemically and mechanically adhered to one another via micro-roughness.[9]

One of the most important factors that influence the clinical longevity of indirect restorations is microleakage. The presence of marginal gap results in exposure cement at the margins and its dissolution, followed by secondary caries, periodontal disease, and microleakage.[10] By reviewing the literature[11-13], many studies were concerned with the microleakage of the PFM, glass ceramics, and bi-layered zirconia restorations. However, there is a lack of knowledge regarding the comparison of microleakage between monolithic gradient and translucent zirconia. So, the aim of the present study was to evaluate the microleakage of gradient zirconia crowns compared to translucent zirconia crowns bonded with self-adhesive resin cement.

MATERIALS AND METHODS

1. Sample size calculation:

Based upon the results of Korkut L et al.[14] in 2011, the mean and standard deviation values were 50.29 (5.19) µm for procera zirconia crowns and 43.02 (4) µm for cercon smart ceramics . The effect size (d) was 1.569. Using alpha (α) level of (5%) and Beta (β) level of (20%), i.e., power = 80%, the minimum estimated sample size was 8 specimens per group. Sample size calculation was performed using G*Power Version 3.1.9.2.

2. Sample selection:

This study received ethical approval from the Institutional Review Board of MIU Faculty of Dentistry (MIU-IRB-2122-170). A total of sixteen human maxillary first premolars, which had been extracted due to orthodontic reasons, were obtained from the extracted tooth bank at MIU. The selected teeth underwent a thorough visual and radiographic assessment to verify their absence of prior endodontic treatment, restorations, carious lesions, structural cracks, or signs of internal resorption. Handling extracted teeth was done following all the infection control measures in order to remove visible blood and gross debris using an ultrasonic scaler, followed by heat sterilization in an autoclave at 121 degrees Celsius for 30 minutes.[15] Then, teeth were stored in normal saline at room temperature.

3. Sample preparation:

Mounting the teeth in acrylic resin blocks:

To maintain the positioning of the long axis of the tooth perpendicular to the floor, a Customized 3-D Printed Mold with a Paralleling device was fabricated **(Figure 1)**. This device consists of 2 compartments; the upper compartment (Paralleling device) maintains the long axis of the tooth perpendicular to the floor by means of 4 screws, while the lower compartment (hexagon-shaped Mold) holds the acrylic resin around the roots of the teeth by 1 screw.

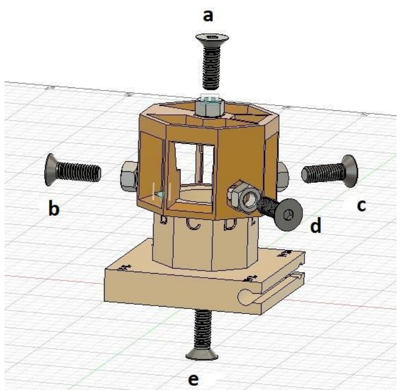


Figure (1): Diagram for Custom Mold with paralleling device.

Mounting was carried out by fixing the tooth to the upper compartment, then the acrylic resin was mixed according to the manufacturer's instructions and packed in the lower compartment. Then, finally, the upper compartment holding the tooth was lowered, in place guided with the hexagon-shaped walls of the device.

4. **Procedural steps:**

Tooth preparation:

To standardize tooth preparation, a CNC milling machine was used to prepare all teeth to receive full coverage of monolithic zirconia crowns. Before starting the tooth preparation, all teeth embedded in acrylic resin blocks were scanned using the MEDIT i700 intra-oral scanner, and the scans were exported as STL files. The files were imported into the dental CAD software to design the preparation using the "Egg-shell provisional" function according to the parameters for monolithic zirconia crown. The parameters were 6 degrees taper and a total occlusal convergence of 12 degrees. The axial reduction was 1 mm to maintain an axial height of 4 mm with a 0.8 mm wide smooth continuous chamfer finish line. The occlusal reduction of each tooth was 1.5 mm following the occlusal anatomy.[16] The preparation designs were imported into the CAM software to calculate the machining tool paths. The base of the Custom Mold Device was fixed to the base of the machine for proper teeth preparation. The milling machine spindle was loaded with TR 26 abrasive rounded-end diamond stone and positioned parallel to the long axis of the tooth in the custom mold. During the preparation, a water coolant line was directed at the tooth to keep the teeth hydrated and avoid overheating. Rounding off all line and point angles and finishing were carried out using an extra fine grit taper round-end stone (TR 13 EF) **(Figure 2).**

Zirconia crown construction:

Monolithic zirconia crowns were constructed using CAD/CAM; prepared teeth were first scanned then the crowns were designed, milled, and sintered.

Abutment scanning:

Scanning was carried out by MEDIT i700 intra-oral scanner according to the manufacturer's recommendations.

Figure (2): Top view of prepared tooth in acrylic mold.

Crown designing:

The created STL files revealed no undercuts. Full contour crowns were designed using CAD software. The software generated the restoration proposals with minor adjustments when necessary, taking into consideration the insertion axis, margin placement, occlusal, wall thickness, and cement space. The cement space was decided to be 25 µm around the margin and an additional cement space of 80 µm starting 1mm above the finish lines of the teeth.[17] Each crown was designed to fit specifically to its corresponding prepared tooth. However, the outer crown shape was standard for all the crowns. This whole design for each sample was analyzed from all aspects prior to milling.

Crown milling:

The appropriate disc size (16 mm) was selected for the crown final size using CAM software. The software compensates for the sintering shrinkage of the material, which is 25%. Milling was performed by means of cylindrical burs under continuous coolant. After milling, the separation of the crowns from each other and from the disc was carried out by using a diamond stone held in a straight handpiece. The milling debris and the lubricant remnants in the fitting surface of the crowns were removed using a steamer.

Sintering:

The sintering process was done following the manufacturer's instructions for all the crowns in a special furnace at a sintering temperature of 1500°C for ZirCAD Prime crowns with holding time 2 hours and 1550°C for UTML crowns with a holding time of 2.5 hours according to manufacturer instructions.[18]

Randomization:

A colleague from another department created the random sequence list for the specimens using specialized software (Randomizer.com). Each specimen was numbered for each group using an Excel sheet. The random sequence list was kept by the colleague and concealed from the Principle Investigator. The colleague then informed the principal investigator about each specimen grouping at the time of the experiment.

Cementation:

After sintering, fit verification of the milled crowns over their corresponding prepared teeth was done and examined by stereomicroscope at several points to ensure proper marginal fit before cementation. For both materials, cementation was carried out using Theracem self-adhesive resin cement containing MDP. To improve the adhesion between the resin cement and the internal surface of the zirconia crown, the intaglio surface of the crowns was ultrasonically cleaned in alcohol for 5 mins, then Sandblasted with 50 µm alumina at 2 bar pressure. According to the manufacturer's instructions, no Surface treatment was used.[8] The self-adhesive cement is supplied as an automix tube. A proper amount was injected into the fitting surface of the crown and placed over the prepared tooth. The cementation procedure was carried out using a specially designed cementation device[19]; with 5 kg of static load on the prepared tooth. **(Figure 3).** A 5 sec Tack curing was done where the excess cement was removed using an excavator. The samples were then subjected to curing for 40s from each side using LED curing unit and left for 6 minutes under the load.

Figure (3): A sample crown being cemented under 5 kg load.

Thermocycling:

All the cemented crowns underwent thermocycling using a thermocycling unit in distilled water for 5000 cycles at changing temperatures between a cold water bath at 5°C for 30 seconds and a hot water bath at 55°C for 30 seconds with a dwell time of 10 seconds. This is equivalent to 6 months in vivo.[20]

Die immersion:

In order to assess microleakage, all teeth were covered with two layers of nail varnish (essence shine last and go, gel nail polish) within 1 mm of the bonded interface (to prevent any dye penetration except at the tooth/crown interface) and left undisturbed for one day to dry. Then, all bonded crowns were immersed in 0.5% basic fuschin dye for 48 hours at 37°C. All the coronal parts of the study samples (crowns cemented on the prepared teeth) were embedded in acrylic resin and poured in polyethylene tubes to avoid their fracture during sectioning. Using IsoMet (4000 Buehler), the microleakage tested assembly was mounted and sectioned vertically in a bucco-lingual direction with a low-speed cutting saw of 0.7 mm thickness abrasive disc at a speed of 2500 rpm, under continuous water-Coolant. The sections were then separated, and the tooth restoration interface was examined at the cervical margins under a stereomicroscope at magnification 40X.

Dye scoring:

The dye penetration depths were scored according to Gu and Kern[21] as follows:

0: no leakage

1: 1/3 of the chamfer finish line preparation

2: 2/3 of the chamfer finish line preparation

3: all of the chamfer finish line preparation

4: more than 1/3 of the axial wall

5: more than 2/3 of the axial wall

6: all of the axial walls, including the occlusal edge

7: exceeding the occlusal edge

All generated data were collected, tabulated, and statistically analysed.

RESULTS

I-Microleakage measurement (μm)

There was no statistically significant difference between the two materials **(Table 1), (Figure 4).**

II-Microleakage scores

There was no statistically significant difference between the two materials **(Table 2), (Figure 5).**

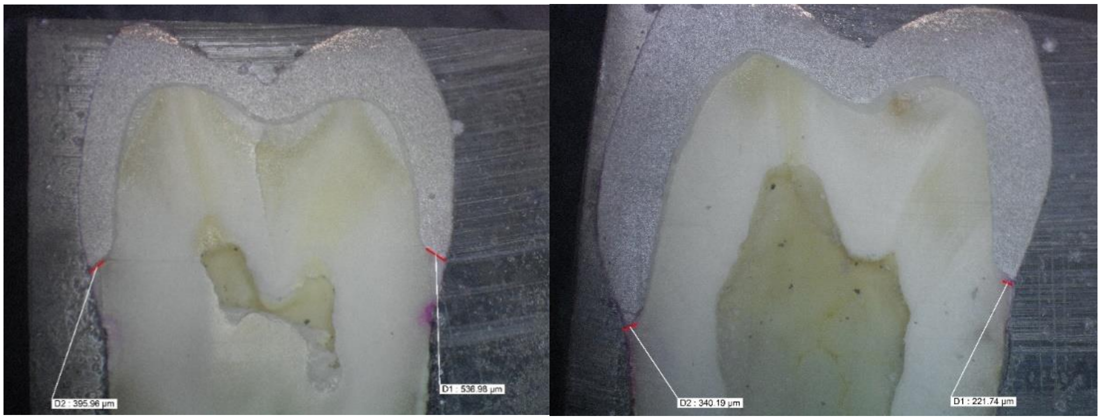
Table (1): Descriptive statistics and results of Mann-Whitney U test for comparison between microleakage measurements (μm) of the two materials.

IPS e.max ZirCAD Prime		UTML Katana		*P*-value	*Effect size (d)*
Median (Range)	Mean (SD)	Median (Range)	Mean (SD)		
534.9 (466.1-802.8)	557.5 ±112.5	440.2 (282.5-1129.3)	530.1 ±274.2	0.248^{NS}	0.603

Significant level at P ≤ 0.05
NS; Non-significant

DISCUSSION

Microleakage is the term used to describe the spread of bacteria, oral fluids, chemicals, and/or ions into a fluid-filled area or a structural gap that forms between dental structure and restorative materials.[22] Microleakage is one of the main causes of failure that impacts the clinical durability of indirect restorations.[11] The most common re-



(a) **(b)**

Figure (4): Dye penetration of sectioned tooth/zirconia crown.
(a) E-max ZirCAD Prime Crown.
(b) UTML Crown.

Table (2): Descriptive statistics and results of Mann-Whitney U test for comparison between microleakage scores of the two materials.

IPS e.max ZirCAD Prime		Katana		*P*-value	*Effect size (d)*
Median (Range)	Mean (SD)	Median (Range)	Mean (SD)		
2 (1.5- 3)	2.19 ±0.75	2 (1-3)	2 ±0.71	0.629^{NS}	0.238

Significant level at P ≤ 0.05
NS; Non-significant

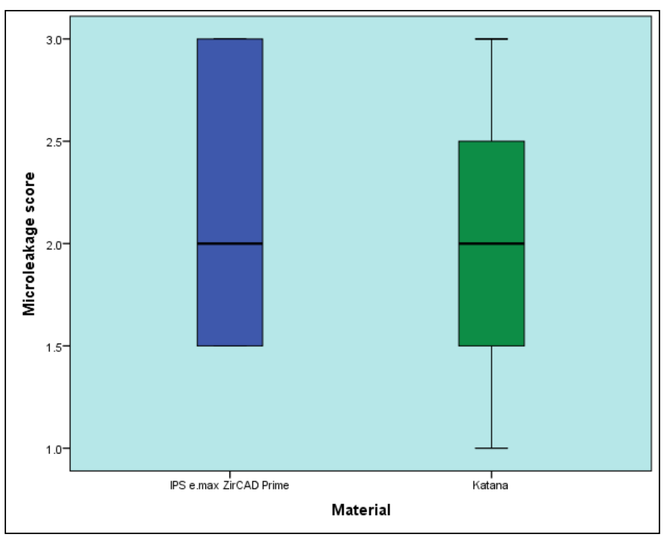


Figure (5): Box plot representing median and range values for microleakage scores of the two materials.

ason for microleakage in restorations is when the gingival margin is positioned below the cementoenamel junction. This is because bonding to dentin is less predictable than bonding to enamel due to the complex pattern and lower mineral content of dentin.[23] It's extremely important to assess the microleakage of zirconia restorations.

Since zirconia is a metal-free restoration, it was found widespread as an alternative for improving the aesthetics of fixed dental prostheses (FDPs) supported by teeth and implants. It displays high mechanical properties, biocompatibility, white coloration, and opacity. Although veneered zirconia has a high success rate, cohesive fractures and chipping of the veneering porcelain have been a major setback in its clinical performance. The introduction of zirconia in its monolithic form prompted efforts to enhance the material's aesthetic properties in order to reduce the potential of chipping with a reduced ceramic thickness. A variety of compositional and processing changes were made with the goal of reducing light scattering within the material.[2,24]

In this study, the Multi zirconium oxide IPS e.max ZirCAD Prime was used. Maharishi et al.[4]**,** in 2024, have concluded that the ZirCAD Prime strength and color-graded zirconia material offers advantages in both strength and translucency. Ahmed et al.[25] in 2024**,** also claimed that ZirCAD prime zirconia have shown a weaker bond stability in comparison with IPS e.max CAD ceramic. However, this material was used in our study due to scarce research on its marginal performance. Another zirconia was used in our study, which was the Ultra Translucent Multi-Layered zirconia (UTML), as it contains 5% yttria, and offers high esthetics, and can be used as a monolithic without the need for veneering .[25] Its crystal structure is primarily cubic and has a light transmittance of 43%, comparable to lithium disilicate LT ingot. Zhang et al.[26]**,** in 2019, claimed that due to high yttria content in the UTML zirconia, it has higher cubic content and larger grain size, which result in lower strength but higher translucency.

The teeth selected for the study were extracted for orthodontic reasons to ensure that all teeth are from young patients and that the dentin available for bonding is not aged. Nawrocka and Szymańska[27] in 2019**,** in their study claimed that these human teeth that have been extracted are used as models in dental research to simulate the in-vivo operations as they are preferred for assessing bond strength.

To stabilize and standardize the preparation of teeth, the roots of each tooth were embedded with vertical centralization in a clear acrylic resin block with the help of a specially customized mold and paralleling device. A standardized, consistent tooth preparation was carried out using CNC technology. Kasem et al.[28] in 2020, in his study he used CADCAM technology to prepare the samples and claimed that the development of CAD/CAM technology provides a reproducible method that enables the standardized preparation of extracted natural teeth for in vitro testing. This can decrease the bias resulting from sample production and enable comparisons across research investigations.

To date, Y-TZP is shaped by subtractive milling. Using CAD/CAM, intraoral scanning, design, and milling of the crown were made possible. In our study, the tooth/ resin assembly was scanned by MEDIT i700 intra-oral scanner according to the manufacturer's recommendations. Jivanscu et al.[29] in 2021, claimed that Medit i700 IOSs generated digital impressions with a very high accuracy compared to the investigated IOSs.

To design full contour crowns, ExoCAD software was then used. It was determined that there should be 25 µm of cement space surrounding the margin and an additional 80 µm of cement space beginning 1 mm above the tooth finish lines. Hammoud and Ibraheem[17] explained that increasing the luting material escape pathway and decreasing the hydraulic pressure generated within the crown avoided inadequate seating for the restoration. These results came in accordance with Nayyef and Ibraheem[30] in 2021, which claimed that 25 µm cement space at the margin reduced the microleakage of zirconia restorations.

Zirconia is often machined in a pre-sintered porous condition rather than a densely sintered state as it is easier to mill, and that's why they were selected for our study. Nevertheless, these Y-TZP ceramics must be sintered after milling to reach their maximal strength, with consideration prior to milling the workpieces that a 25–30% shrinkage is accompanied.[31]

It was strongly agreed that the combination of mechanical and chemical pretreatment can provide long-lasting, durable resin bonding on surface-treated Y-TZP and recommended the application of primers and MDP-containing resin cements.[8,32,33]. According to a systematic review by Ammar and Blatz[34] in 2022, bonding to new generations of zirconia. is the same as 3Y-TZP, and the mentioned surface pretreatment techniques had no detrimental effects on its flexural strength. Gerdzhikov et al.[35] in 2023, have concluded that sandblasting the ZirCAD Prime zirconia ceramics increased the strength of the bond with resin cements. The self-adhesive resin used was Thera-cem. It is a dual-cured, self-adhesive resin luting cement containing MDP, calcium, and fluoride; provided as a paste/paste automix tube to enhance bonding to zirconia. It doesn't require any surface treatment before cementation. According to Blatz et al.[36] in 2018, self-adhesive bonding technologies offer a marginal seal, improved compressive strength, and stronger shear bond strength. Berkman et al.[37] in 2020, in their study underwent 5,000 thermocycling and proved that TheraCem showed a high micro-tensile bond strength value.

For microleakage testing, the dye penetration approach, 0.5% basic fuschin dye was used, which colors the areas of microleakage with a contrasting dye used as an immersion solution. Our study agreed with Abo elenein et al[38] in 2020**,** who measured the microleakage for zirconia crowns by immersing them in 0.5 % fuschin dye. It is advantageous because of the lack of radiation or reactive materials. This method is highly convenient, repeatable and dye solutions are easily accessible.[23]

Referring g to studies by Morresi et al.[20] in 2014, and Ebadian et al.[39] in 2021, samples were exposed to thermocycling in which 5000 cycles were employed, simulating a six-month aging In-Vivo. This was done to simulate the oral environment. Strains larger than the cohesive and adhesive strength of a material can be produced when it contracts during thermocycling, leading to microleakage and the development of microcracks.

The null hypothesis of the present study was accepted as there were no significant differences between the two tested groups regarding the microleakage. Also, the microleakage scoring of zirconia crowns was all within the finish line area.

Our study came in accordance with Beuer et al.[40], who evaluated the effect of preparation angles on the accuracy of zirconia copings and resulted that the tapering degree of 12 achieved the best overall score in microleakage compared with tapering degrees of 4 and 8.

Although Ebadian et al.[38] in 2021**,** suggest that self-etching resin cement has a better marginal sealing compared to self-adhesive resin cement; however, the difference between the total-etch and self-adhesive cement was not statistically significant. Also, Yang et al.[41] in their study claimed that MDP-containing primers, adhesives, and composite cement create bonds to zirconia with acceptable strength after long-term aging. As mentioned by Dometeglu and Zortuk[13] study, MDP monomer is believed to treat the microcracks on the ceramic surfaces and strengthen the ceramic, and therefore, resin cement containing MDP is recommended for adhesion to zirconia. In their study, the leakage values were registered as “0” but a thin line of leakage at the corners of the restoration was observed. Our study is also in agreement with Geerts et al.[12], claiming that small leakage scores can be result of the high efficiency of self-adhesive resin cement used in the cementation of zirconia crowns.

Al-Kataan and Al Naimi[42] in 2023, also supported our study. In addition to other factors that influence the connection with zirconia, such as filler particle size and viscosity, the MDP bond demonstrated a strong affinity for the oxide layer on the zirconia surface. The wettability of the resin on the zirconia surface affects the bond strength. TheraCem, has a high substrate content, low viscosity, and a high penetrating ability, which makes it mechanically strong.

Our study came in contrast to Abo-elenein et al.[38]**,** who claimed that microleakage is increased by thermocycling. The thermocycling compromises the restoration's marginal integrity and raises the risk of staining, marginal leakage, hypersensitivity, and pulpal pathology, whereas in our study, microleakage scores were within the finish line only. Mahrous et al.[43] in 2018 in their study, claimed that TheraCem positively influences micro-shear bond strength and modifies the bond strength with dentin. TheraCem resin cement contains specific active components (calcium and fluoride) that can interact ionically with hydroxyapatite, as per the adhesion-decalcification idea. These molecules have the ability to etch or penetrate dentin and react with hydroxyapatite to produce calcium ions that have a lower binding energy. Chemical contact with the composite is made possible by the electron-accepting properties of these ions. In this sense, it is believed that the chemical bonding and micromechanical interlocking with hydroxyapatite might work to give the material its maximum adherence.

This study had limitations. As with any in-vitro study, the degree of variation in the microleakage of multilayered zirconia crowns is yet unknown. The intraoral environment, which includes factors like humidity and the teeth's ongoing function within the oral cavity, might cause changes in the clinical setting. Only 1 cement (TheraCem Self-adhesive resin cement) was used. There was no handling of the cement, for example, heating to change its viscosity, which may affect the results. ZirCAD Prime multilayered zirconia was compared to 5Y zirconia only in our study; other studies may be needed for other yttria % in zirconia.

Within the limitations of this study, it was concluded that:

Thermocycling didn't negatively affect the microleakage of the bonded monolithic gradient (3y and 5y) and translucent (5y) crown, which extended only within the finish line area.

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CONFLICTS OF INTEREST: There was no conflict of interest.

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