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Comparison of Microleakage of Monolithic Zirconia after Surface Treatment and Thermal Cycles Using Data Analysis Software

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ABSTRACT

Objective: The aim of this study was to investigate the microleakage of monolithic zirconia after different surface treatments using computer software.
Materials and Methods: Three different monolithic zirconia ML (Multilayered), STML (Super Translucent Multilayered), UTML (Ultra Translucent Multilayered) were prepared as discs with a diameter of 15 mm and a thickness of 1.2 mm. Four different surface treatments (Hydrofluoric acid, Tribochemical silica coating, Hydrofluoric acid application + Tribochemical silica coating, Milling + Tribochemical silica coating + Hydrofluoric acid application) were applied to the prepared samples according to their groups (n=8). Samples, Group C: Control group, Group HF: Hydrofluoric acid application, Group T: Tribochemical silica coating, Group HF+T: Hydrofluoric acid application + Tribochemical silica coating, Group F+HF+T: Milling + Tribochemical silica coating + Hydrofluoric acid application, then adhesive system was applied to all specimens and repaired with resin cement. The specimens were thermocycled for one year aging and then immersed in basic fuchsin solution to evaluate microleakage. The specimens were separated with a micro-cut device and evaluated under a stereomicroscope. The dimensions of the images were measured in Python program and the permeability and surface treatments of the zirconia samples were compared. Statistical analysis was performed by two-way ANOVA ($p<0.05$).
Results: UTML F+HF+T showed the lowest microleakage (12.15 ± 1.69), while ML C showed the highest microleakage (73.93 ± 1.59). Among the zirconia specimens, the highest adaptation was obtained in the UTML zirconia (37.59 ± 23.58).
Conclusion: According to the data obtained, milling + tribochemical silica coating + acid application surface treatments are recommended for the repair of monolithic zirconia restorations. The sintering temperature and Yttrium Oxide (Y_2O_3) content of the monolithic zirconia used are effective factors in microleakage after repair.

Monolitik Zirkonyaların Yüzey İşlemleri ve Termal Siklus Sonrasında Mikrosızıntılarının Veri Analizi Yazılımıyla Karşılaştırılması

Makale Bilgisi

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ÖZET

Amaç: Bu çalışmanın amacı; monolitik zirkonyalara uygulanan farklı yüzey işlemleri sonrasında mikrosızıntılarını bilgisayar yazılımıyla incelemektir.
Gereç ve Yöntemler: Çalışmada kullanmak amacıyla, üç farklı monolitik zirkonya ML (Multilayered), STML (Super Translusent Multilayered), UTML (Ultra Translusent Multilayered) 15 mm çapında 1,2 mm kalınlığında disk şeklinde hazırlandı. Hazırlanan örneklere gruplarına göre dört farklı yüzey işlemi (Hidroflorik asit, Tribokimyasal silika kaplama, Hidroflorik asit uygulama+ Tribokimyasal silika kaplama, Frez ile aşındırma + Tribokimyasal silika kaplama + Hidroflorik asit uygulaması) uygulandı (n=8). Örnekler, Grup C: Kontrol grubu, Grup HF: Hidroflorik asit uygulama, Grup T: Tribokimyasal silika kaplama, Grup HF+T: Hidroflorik asit uygulama+ Tribokimyasal silika kaplama, Grup F+HF+T: Frez ile aşındırma, + Tribokimyasal silika kaplama + Hidroflorik asit uygulaması şeklinde 5 gruba ayrıldı, daha sonra tüm örneklere adeziv sistem uygulanıp rezin siman ile tamir yapıldı. Örnekler bir yıllık yaşlandırma amacıyla termal-siklusa tabi tutulup daha sonrasında mikrosızıntıyı değerlendirmek amacıyla bazik fuksin solüsyonuna daldırıldı. Micro-cut cihazı ile ayrılan örnekler stereomikroskop altında değerlendirildi. Alınan görüntülerin ölçüleri Python programında ölçülerek zirkonya örneklerin geçirgenliği ve yüzey işlemleri karşılaştırıldı. İstatiksel analiz iki yönlü ANOVA ile yapıldı ($p<0,05$).
Bulgular: Örneklerden en düşük mikrosızıntıyı UTML F+HF+T gösterirken (12,15 ± 1,69), en yüksek mikrosızıntı ML C (73,93 ± 1,59) görüldü. Zirkonya örnekler arasında en yüksek adaptasyon UTML zirkonya örneklerde (37,59 ± 23,58) elde edildi.
Sonuç: Elde edilen veriler doğrultusunda frezleme+ tribokimyasal silika kaplama+asit uygulama yüzey işlemleri, monolitik zirkonya restorasyonların tamiri açısından önerilmektedir. Kullanılan monolitik zirkonyanın sinterleme sıcaklığı ve içeriğindeki İtriyum Oksit (Y_2O_3) tamir sonrası mikrosızıntıda etkili faktördür.

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INTRODUCTION

Resin composites are commonly utilized in clinical dental practice due to their superior mechanical properties, excellent adhesion to tooth structures, ease of application, aesthetic appeal, and compatibility with minimally invasive dental techniques.[1,2] These materials offer the distinct advantage of reparability over time, as opposed to complete replacement of damaged restorations. Repair procedures mitigate the drawbacks of full replacement, which often involve extensive preparation and high costs.[3]

Clinically, crowns have been observed to experience issues such as crumbling and delamination after prolonged use, which leads to restoration failures.[4] Advances in materials science have introduced high-purity, highly translucent yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics, which address the typical limitations of zirconia ceramics, such as inadequate translucency and a monolayer appearance.[5] To establish a strong bond between tooth structures and porcelain restorations, various surface treatments are employed. Acid etching is one such method, although it is less effective with zirconia restorations due to their structural properties.[6-8] Various methods, such as etching, laser irradiation, and nano-grade aluminum coating, are used to create surface roughness that enhances the micromechanical bond between the zirconia and the resin cement.[9]

Despite these modifications, the micromechanical bond remains insufficient, thus necessitating the use of primers with resin cements. For optimal cementation of zirconia restorations, self-adhesive, resin-based agents and universal adhesives containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) are recommended.[10] Other surface treatments, such as tribochemical silica coating—which involves air-etching the ceramic surface with alumina particles coated with silica-have been developed to enhance the bonding between resin cement and zirconia.[11] Abrasion with diamond milling is a method frequently used on the fracture surface when repairing in the mouth. Abrasion removes contamination from the fracture surface. It also provides a mechanical connection by providing visibly rougher surfaces than other methods.[12]

Microleakage testing of dental materials is a generally accepted technique for the evaluation of margin integrity. Microleakage refers to the clinically undetectable passage of bacteria, fluids, molecules, or ions in the micro-gaps (10^{-6} µm) between a cavity wall and the restorative material applied over it. The evaluation of microleakage is conducted using the basic fuchsin stain methylene blue.[13]

While there is a relative of studies examining the durability of monolithic zirconia following repair, there is a significant gap in the literature about microleakage and surface treatments. This study aimed to evaluate the impact of different surface treatments on the repair of aged resin composites using monolithic zirconia. The null hypothesis was that there would be no significant difference between the microleakage of disc-shaped specimens repaired with various aged monolithic zirconia materials and surface treatments.

MATERIAL and METHODS

Preparation of the Samples

Three types of monolithic zirconia blocks with different translucency properties were used: multilayered (ML), ultra super translucent multilayered (UTML), and super translucent multilayered (STML) (Table 1). Power and sample size analysis for 'f test - ANOVA: Fixed effects, special, main effects, and interactions' was conducted using G*Power v3.1.9.2. Sample size was determined by referencing the study according to the 95% confidence interval (CI; 1-α), 95% test power (1-β), effect size f=0.374 and analysis of variance (ANOVA) test.[14] A total of 120 samples were included in the study. All were of the same brand (Katana; Kuraray, Noritake Dental Inc, Tokyo, Japan) and were manufactured using a CAM (Yenadent D43, Yenadent Ltd, İstanbul, Türkiye) system. The sample was designed using computer software (Meshmixer, California, USA) and pre-sintered in a laboratory with a sintering furnace (Everest Therm; KaVo Dental GmbH, Biberach, Germany) following the manufacturer's instructions. According to the international standard ISO 6872, the final dimensions were in the form of a disk 15 mm in diameter and 1.2 mm in thickness, consistent with the methodology used in scientific studies on the durability of all-ceramic materials (Figure 1).[15] The thickness of the samples was checked using a digital caliper. The prepared ceramic samples were ultrasonically washed for two minutes, then air-dried and prepared for surface treatment.

Figure 1. Preparation of Specimens

Table 1. Materials used in the study, manufacturer, composition and flexural strength.

Material	Code	Manufacturer	Composition	Flexural Strength (MPa)
Multilayer	ML	Kuraray, Noritake Dental Inc., Tokyo, Japan	ZrO_2 + HfO_2 +Y_2O_3) >99%, (Y_2O_3) 4%, (HfO_2) ≤5%, other oxides ≤1%	1125
Supertranslucent	STML	Kuraray, Noritake Dental Inc., Tokyo, Japan	(ZrO_2 + HfO_2 +Y_2O_3) >99 %, (Y_2O_3) 5.3 %, (HfO_2) ≤5 %, other oxides ≤1 %	748
Ultratranslucent	UTML	Kuraray, Noritake Dental Inc., Tokyo, Japan	(ZrO_2 + HfO_2 +$Y2O_3$) >99 %, (Y_2O_3) 5.4 %, (HfO_2) ≤5 %, other oxides ≤1 %	557

Surface Treatment of the Samples

The prepared samples were randomly divided into five groups. For group C (the control group), no surface treatment was applied to the samples. For group HF (hydrofluoric acid), 9% HF (Ultradent Porcelain Etch; Ultradent Inc., South Jordan, USA) was applied to the samples and left for 60 seconds. After two minutes of washing, the HF was removed. Silane (Ultradent, Utah, USA) was then applied to the sample surfaces and allowed to dry for 60 seconds. For group T, tribochemical silica coating was applied. Samples were roughened with 30 µm silica-coated Al_2O_3 particles (3M ESPE, Seefeld, Germany) for 15 seconds under 3 bars of pressure.[16] A distance of 10 mm was left between the application tip of the blasting device and the sample. All operations were performed by a single user. After the procedure, the silica-coated samples were cleaned with 96% isopropyl alcohol using an ultrasonic device (Euronda, Sassuolo, Italy). For group HF+T, HF and tribochemical silica coating were applied. A 9% HF solution was applied to the samples and allowed to remain for 60 seconds. After two minutes of washing, the HF was removed. Silane was then applied to the sample surfaces and allowed to dry for 60 seconds. The surfaces were then roughened with 30 µm silica-coated Al_2O_3 particles for 15 seconds under 3 bars of pressure. A distance of 10 mm was left between the application tip of the silica coating and the sample. After the procedure, the silica-coated samples were cleaned with 96% isopropyl alcohol using an ultrasonic device.

Group F+HF+T involved the use of milling burs and HF as well as tribochemical silica coating applications. The samples were roughened by abrading in the same direction for ten seconds with finger pressure by the same operator using 125 µm green-banded diamond burs (Acurata, Thurmansbang, Germany) with a high-speed water-cooled clinical aerator (NSK, Nagaoka, Japan). The device was calibrated by a dental technician with professional assistance. Self-adhesive resin cement (Panavia SA Cement, Kuraray, Osaka, Japan) was bonded to the surfaces with special molds prepared for standardization. A mold with a diameter of 15 mm and a thickness of 2.5 mm was created using pink wax (Polywax, München, Germany). This mold was placed in silicone impression material (Zhermack, Badia Polesine, Italy). The samples, after the adhesive was applied, were then placed in the mold. The polymerization process was performed with an LED (light emitting-diode) light device (Bredent GmbH & Co KG, Senden, Germany) for 40 seconds. The measurements of luminous flux, luminous intensity, and energy density from the LED device were recorded with the Bluephase meter II radiometer (Ivoclar Vivadent, Schaan, Switzerland), and the accuracy was determined by comparison with the technical specifications and standards of the LED device itself. Next, the repair material was removed from the mold. After being bonded to each other with adhesive systems, the samples were soaked in 37°C distilled water for 24 hours and thermal-cycled to mimic aging. The samples were subjected to the aging procedure through a 10.000-cycle thermodynamic cycler (Gökçeler Makine, Sivas, Türkiye) at 5–55°C with a 30-second dwell time.[17]

Evaluation with a Microscope

Two coats of blue nail polish (Flormar, Kocaeli, Türkiye) were applied to all areas of the zirconia, except for 1 mm of the connection area. To evaluate marginal leakage, the samples were soaked in 0.5% basic fuchsin at 37°C for 24 hours. After staining, the prepared samples were cut in half to evaluate the microleakage. Using a linear precision saw (Isomet 1000 Linear Precision Saw; Beuhler, Illinois, USA), the specimens were cut in half at a speed of 600 rpm. The cutting process took into account the thickness of the water-cooled cutting blade, which is 0.3 mm. The prepared samples were kept in basic fuchsin solution for one day to evaluate the coloration of the microleakage areas. Then, images of the samples were taken under a stereomicroscope (SZx10 Olympus, Tokyo, Japan) at 25x magnification) (Figure 2). Calibration was performed by placing a ruler within the field of view of the microscope and utilizing the microscope's measurement capabilities. Surface images were captured once the ruler's measurements were aligned with the measurements provided by the microscope's software. The images were transferred to a computer program (Pycharm 3.12.3, Prague, Czech Republic), and the dimensional sizes of the images were obtained in square millimeters (mm^2) through the program (Figure 3).

Statistical Analysis

Statistical analyses were performed using the SPSS software (IMB SPSS Statistics for Windows version 14.0; IBM Corp., New York, USA). To assess the homogeneity of the composite and thickness variance distributions for each group ($n = 8$), the Shapiro-Wilk test was applied, and normal distributions were found. The measurement values for the monolithic zirconia types and the surface treatments were analyzed using a two-way ANOVA test, and the obtained values were compared using Tukey's test. The p-value's significance level was determined to be $p<0.05$.

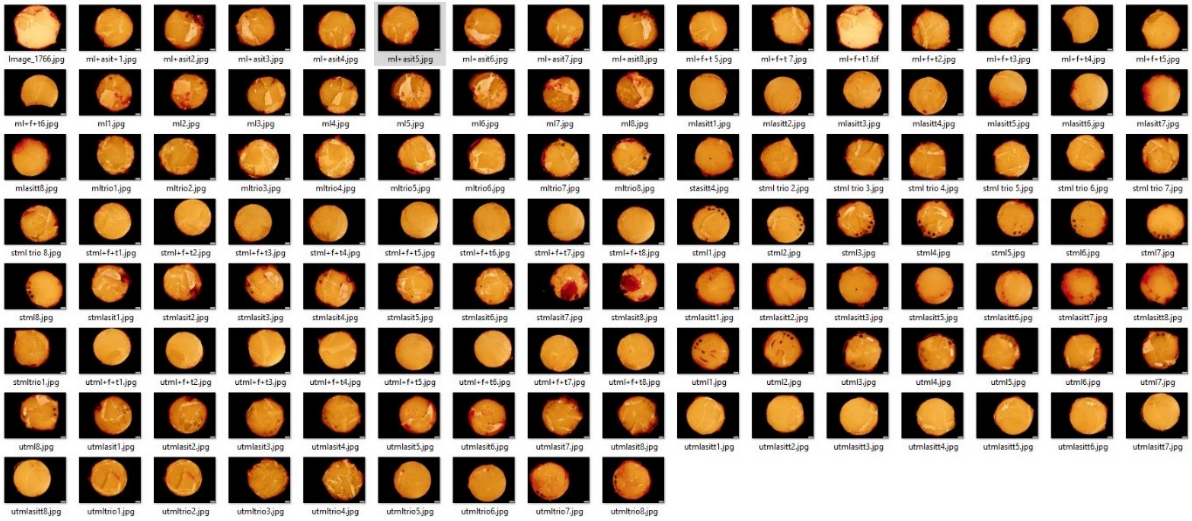

Figure 2: Images of samples under the microscope

```
    # Toplam alanı mm² cinsine dönüştür ve daha az küsürlü ş
    toplam_alan_mm2 = round(toplam_alan * piksel_mm_orani**2


    # Alanı ve dosya adını alanlar listesine ekle
   # alanlar.append({"Dosya Adı": resim_dizini, "Alan (mm²)"

   # Verileri bir veri çerçevesine aktar
   #df = pd.DataFrame(alanlar)

   # Verileri Excel dosyasına yaz
   #df.to_excel("resim_alanlari1.xlsx", index=False)
    # Sonucu görselleştir
    kucuk_mask = cv2.resize(mask, (0, 0), fx=0.50, fy=0.50)
    cv2.imshow('Kirmizi Alanlar', kucuk_mask)
    cv2.waitKey(0)
    cv2.destroyAllWindows()
```

```
C:\Users\user\PycharmProjects\untitled1\venv\Scripts\python.exe
ml3.jpg - Toplam Alan (mm²): 7.70282
```

Figure 3. Measurement of microleakage areas in the Pycharm program

RESULTS

A two-way ANOVA test showed a significant difference in the microleakage values of the monolithic zirconia types and the surface treatments ($p<0.001$) (Table 2). Table 3 presents the mean microleakage values and standard deviations (SD) of the monolithic zirconia types and the surface treatments. Group F+HF+T exhibited lower microleakage values compared to other surface treatments ($p<0.05$). When compared with other surface treatments, significant differences were observed among all groups ($p < 0.05$). The lowest microleakage was observed in the control group. A significant difference was found between zirconia grades for control and HF-treated surfaces ($p < 0.001$). However, no significant difference was observed between UTML and STML on tribochemical silica-coated surfaces ($p\geq0.05$). The lowest microleakage value was obtained for UTML zirconia in the F+HF+T group (12.15±1.69), while the highest microleakage value was found for ML zirconia in the control group (73.93±1.59). No significant difference was observed between zirconia types in the control group ($p\geq0.05$), but significant differences were found between zirconia types when surface treatments were applied ($p<0.05$). Additional multiple comparison results are presented in Table 3 and Figure 4.

Table 2: Two-way ANOVA Test for the Effect of Monolithic Zirconia Types and Surface Treatments on Microleakage

Microleakage	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	66892.136^{a}	14	4778.010	1106.864	<0.001	0.993
Intercept	202517.827	1	202517.827	46914.874	<0.001	0.998
Material	1347.706	2	673.853	156.103	<0.001	0.748
Surface Treatment	64967.154	4	16241.789	3762.540	<0.001	0.993
Material * Surface Treatment	577.276	8	72.160	16.716	<0.001	0.560
Error	453.254	105	4.317			
Total	269863.218	120				
Corrected Total	67345.391	119				

a. R Squared = .993 (Adjusted R Squared = .992)

Table 3: Microleakage Descriptive Statistics

	Zirconia Types			
Microleakage	ML	STML	UTML	Total
Group C	73.93 ± 1.59^{ab}	71.00 ± 1.77^{a}	72.56 ± 1.82^{ab}	72.56±1.82^{A}
Group HF	67.16 ± 0.58^{bc}	59.33 ± 2.49^{c}	65.51 ± 4.92^{d}	65.51 ± 4.92^{B}
Group T	35.25 ± 0.69^{e}	26.67 ± 1.52^{f}	24.77 ± 3.22^{f}	28.90 ± 5.07^{C}
Group HF+T	25.77 ± 2.12^{f}	19.38 ± 1.85^{h}	20.68 ± 2.40^{gh}	21.94 ± 3.48^{D}
Group F+HF+T	24.21 ± 4.43^{fg}	13.12 ± 0.38^{ı}	12.15 ± 1.69^{ı}	16.49 ± 6.17^{E}
Total	45.60 ± 21.80	40.05 ± 25.71	37.59 ± 23.58	41.08 ± 23.79

A-E: No difference between surface treatment with the same letter. a-ı: No difference between zirconia types and surface treatment interactions with the same letter.

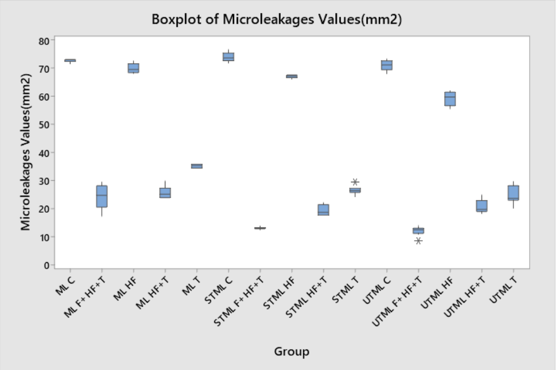

Figure 4. Boxplot of Microleakage values according to zirconium

DISCUSSION

The results of this study revealed that the surface treatment techniques significantly affected the microleakage values after the repair procedures were performed on the monolithic zirconia ($p<0.05$). The highest mean marginal compliance values were observed in the burs+HF+ tribochemical silica coating treatments. Therefore, the null hypothesis tested in this study was rejected because our findings showed that there was a significant difference between the microleakage values of the monolithic zirconia that was repaired using different surface treatments. In this study, a diamond bur of silica-coated aluminum oxide was used for mechanical surface roughening. The use of bonding agents increases the bond strength of the repair bonds.

Most clinicians prefer to use the bonding system that they already have in their practice rather than acquire a specialized bonding system for composite repair procedures.[18-20] However, the bonding potential of zirconia restorations is low, and there is no standard repair procedure. Different resin cements have been proposed for the repair of these restorations.[21,22]

The silica coating has been observed to produce microcracks on the surface of zirconia ceramics, increasing their strength.[23] The porcelain and the silane form a chemical connection when the silica creates a glassy coating on the ceramic surface. The results of our study corroborated this finding, as tribochemical silica coating demonstrated a higher level of agreement compared to the other groups. In study, the marginal compatibility with the tribochemical silica coating was significantly increased compared to the control and HF treatments. This is due to the fact that the tribochemical silica coating provides chemical retention with the silica-coated zirconia surface because it binds to the silane more effectively than silica coating.[24,25] According to a recent study, the use of HF etching on both glass matrix and crystal surfaces resulted in the highest bond strength.[26] Moreover, the application of silane and HF to the ceramic surface prior to cementation has been documented to significantly enhance the bonding efficacy of silica-based ceramics.[27,28] However, it has been shown that the lack of a glassy phase or high crystal content causes HF etching to fail in ceramics reinforced with zirconia and alumina.[29] Ural et all.[30] found that HF application did not cause any changes in zirconia surface morphology. According to the results of the present study, the HF-treated groups exhibited reduced microleakage in surface marginal areas compared to the control group.

In HF applications, the protocols can vary considerably, particularly in terms of etching time and acid concentration.[31,32] These variations complicate the assessment of the definitive advantages of this surface treatment, making it challenging to establish a standardized approach for optimal bonding outcomes. In a systematic review, it was concluded that surface treatments with tribochemical silica particles and HF acid resulted in lower coupling than etching with Al_2O_3 or diamond bur abrasives.[33] The current study contradict is incompatible with that systematic review. However, in this study, tribochemical silica coating and diamond milling were used together to reduce microleakage. In clinics, the combination of the two surface treatments may be preferred as a surface treatment for monolithic zirconia repairs.

Prolonged exposure of Y-TZP zirconia to low temperatures may cause different disadvantages. One of these is surface roughness. In addition, reduced durability results in bending force resistance that is sufficient to withstand chewing forces.[34] The addition of a stabilizer containing Y_2O_3 as a component to the zirconia material can significantly improve the mechanical properties of zirconia and enhance its biological properties.[35] In the results of the current study, UTML (5.4% Y_2O_3), with the highest Y_2O_3 content, showed the least microleakage (12.15±1.69), while ML (4% Y_2O_3), with the lowest stabilizer content, showed the highest microleakage values among all of the surface treatments. The results suggest that increasing the Y_2O_3 ratio may enhance the marginal compatibility of the material.

In addition, different sintering temperatures are likely to change the edge fit due to shrinkage as ceramic materials cool to room temperature.[36] This shrinkage depends on several factors, including material composition, density, and the sintering procedure.[37] Ersoy et all.[38] found that increasing the sintering temperature and decreasing the sintering time improve the mechanical properties of the zirconia structure. The sintering temperature of ML monolithic zirconia used in the current study was 1500°C, while the sintering temperature of the STML and UTML monolithic zirconia was 1550°C, as specified by the manufacturer. The differences in the microleakage values of the different experimental groups in this study may have been due to the stability of the zirconia samples and structural differences. With an increase in the sintering temperature, the zirconia samples were found to be completely sintered until the tetragonal stage, and no transformation was observed until the monoclinic stage.[38,39] New generation zirconia types include 4Y-PSZ (Katana ML), 5Y-PSZ (Katana STML), and 6Y-PSZ (Katana UTML). In modern dentistry, the content of Y_2O_3, the proportion of tetragonal or cubic phases, and the material's fracture toughness are crucial factors for clinical applications. The addition of Y_2O_3 to ZrO_2 powder significantly increases the cubic ZrO_2 phase. While this improves certain properties, it can reduce both flexural strength and fracture toughness.[40]

In this study, thermal cycling was applied for 10,000 cycles, which is equivalent to one year. However, D'Amario et all.[41] reported that thermal cycling significantly reduced the bond strength between zirconia and resin cement. In another study, thermal cycling with 10,000 cycles had no effect on bond strength, and even veneer ceramics showed higher bond strength after thermal cycling. In the present study, applying too many thermal cycles was found because it reduced bond strength.[42]

Additionally, the dye penetration method is often preferred in microscope studies due to its cost and ease of application.[43] In the current study, microleakage values were compared using the PyCharm 3.12.3 software to ensure objectivity, rather than relying on traditional scoring methods. Although AutoCAD software was used in previous studies, the data obtained with this program were automatically calculated numerically.[14,44,45]

The chief limitation of this study is that the oral environment cannot be replicated using different surface treatments and monolithic zirconia with different components. However, this study can guide future in-vivo and in-vitro studies. It will also inform clinicians about the microleakage that may occur after the preferred surface treatment for monolithic restoration repair.

CONCLUSIONS

Conclusions obtained as a result of the limitations of the study; Mechanical and chemical treatments applied to the surface during the repair of monolithic restorations help reduce the risk of microleakage. The components of the monolithic material influence the microleakage values. For minimizing the risk of microleakage, it is recommended that clinicians use monolithic zirconia with high Y_2O_3 content with processes such as tribochemical silica coating and milling.

Ethical Approval

This in-vitro study does not require ethics committee approval.

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The authors declare that this study received no financial support.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

Author Contributions

Design: EA, HS, Data collection or data entry: EA, HS, Analysis and interpretation: EA, HS, Literature review: EA, Writing: EA, HNÖ, HS.

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