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Various Impact of Polishing System on Roughness, Microhardness, Phase Transformation, Flexural Strength of Monolithic Zirconia

Monolitik Zirkonyada Farklı Polisaj Sistemlerinin Yüzey Pürüzlülüğü, Mikrosertlik, Faz Dönüşümü ve Eğilme Dayanımı Üzerindeki Etkileri

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of different surface polishing systems and autoclave aging process on the surface properties, phase transformation and flexural strength of monolithic zirconia.
Methods: Forty eight Ø15×1.2 ±0.01-mm disks of one monolithic zirconia materials (Prettau, Zirkonzahn) were prepared glazed, and assigned to 3 groups: A control group with no additional treatment after glaze; and 2 groups in which the glaze layer was removed and the surfaces polished using 2 different polishing systems (OptraFine and Meisinger). After the polishing, each group was divided into two and one of these was aged in an autoclave. The surface roughness of the samples was analyzed using a profilometer device, the surface microhardness was evaluated using the Vickers microhardness test. Both atomic force microscopy and scanning electron microscopy were used to image the surface properties. Phase transformation was analyzed using X-ray diffraction. Finally, the flexural strengths were evaluated.
Results: The effects of surface polishing applied on monolithic zirconia specimens were found to be statistically significant. The effect of autoclave aging on the phase transformation was significant, but the effect on surface roughness, microhardness, and flexural strength was not statistically significant.
Conclusion: It was determined that the surface polishing systems applied to the monolithic zirconia specimens affected the surface properties, phase transformation, and flexural strength. The autoclave aging process did not affect the surface properties and flexural strength but affected the phase transformation.
Keywords: Flexural strength, hardness, surface properties, zirconia

ÖZ

Amaç: Bu çalışmanın amacı, farklı yüzey parlatma sistemlerinin ve otoklav yaşlandırma işleminin monolitik zirkonyanın yüzey özellikleri, faz dönüşümü ve eğilme dayanımı üzerine etkisini değerlendirmektir.
Yöntemler: Tek bir monolitik zirkonya malzemesinden (Prettau, Zirkonzahn) hazırlanan kırk sekiz adet Ø15×1,2 ±0,01 mm disk glazelenmiş olarak hazırlanmış ve 3 gruba ayrılmıştır: Glazeden sonra herhangi bir ek işlem uygulanmayan bir kontrol grubu; ve glaze tabakasının kaldırılarak yüzeylerin 2 farklı parlatma sistemi (OptraFine ve Meisinger) kullanılarak parlatıldığı 2 grup. Parlatma işleminden sonra her grup ikiye ayrılmış ve bunlardan biri otoklavda yaşlandırılmıştır. Numunelerin yüzey pürüzlülüğü bir profilometre cihazı kullanılarak analiz edilmiş, yüzey mikrosertliği Vickers mikrosertlik testi kullanılarak değerlendirilmiştir. Yüzey özelliklerini görüntülemek için hem atomik kuvvet mikroskobu hem de taramalı elektron mikroskobu kullanılmıştır. Son olarak eğilme dayanımı ölçümleri gerçekleştirilmiştir.
Bulgular: Monolitik zirkonya numunelerine uygulanan yüzey parlatma işleminin etkilerinin istatistiksel olarak anlamlı olduğu bulunmuştur. Otoklav yaşlandırmanın faz dönüşümü üzerindeki etkisi anlamlı iken, yüzey pürüzlülüğü, mikro sertlik ve eğilme dayanımı üzerindeki etkisi istatistiksel olarak anlamlı bulunmamıştır.
Sonuç: Monolitik zirkonya numunelerine uygulanan yüzey parlatma sistemlerinin yüzey özelliklerini, faz dönüşümünü ve eğilme dayanımını etkilediği belirlenmiştir. Otoklav yaşlandırma işlemi yüzey özelliklerini ve eğilme dayanımını etkilememiş, ancak faz dönüşümünü etkilemiştir.
Anahtar Kelimeler: Eğilme dayanımı, sertlik, yüzey özellikleri, zirkonya

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INTRODUCTION

Ceramic materials are widely used in prosthetic dentistry to meet the functional and aesthetic needs of patients. Numerous ceramic systems have been developed for dental restorations, and zirconia-based ceramics, in particular, have become increasingly popular as dental restorative materials.[1] The relatively high strength of monolithic zirconia compared to other dental ceramics and the ability to produce zirconia restorations with computer-aided design and computer-aided manufacturing (CAD-CAM) technologies offer a wide range of uses.[2]

Monolithic zirconia restorations fabricated with CAD/CAM technology, although precisely prepared, do not eliminate the need for intraoral fitting. With ceramic restorations, etching procedures can be performed before or after cementation to eliminate occlusal inconsistencies, correct contours and meet aesthetic requirements.[3] The resulting rough surfaces can cause many problems.[4] The texture of the surface is an important factor in the long-term success of a restoration.[5] Therefore, polishing reduces the surface roughness. By manually polishing the rough surfaces, stain formation, and plaque and tartar accumulation can be prevented. Biologic and esthetic continuity can be achieved.[4] Zirconia polishing systems have been developed that contain wheels of high-hardness particles (such as diamonds) that are applied at the chairside without the need for an additional appointment.[6]

The properties of dental materials exposed to different temperatures in the mouth are significantly affected. Since the evaluation of materials in vivo is time-consuming and difficult, different artificial aging methods can be applied in-vitro.[7] Artificial aging is an important method used to evaluate the duration of clinical use of different dental materials in in-vitro studies.[8] Many studies have been conducted on autoclave aging, which is one of the artificial aging methods.[9] In other study reported that autoclaving for one hour at 134 °C, in theory, is similar to 3–4 years in a clinical setting.[10] As a result, the current study aimed to investigate whether hydrothermal aging affects the phase transformation, surface properties, and flexural strenght of the examined materials.

Zirconia transforms between monoclinic, tetragonal, and cubic phases. After the applied surface treatments, the relative amounts of monoclinic and tetragonal phases are analyzed using X-ray diffraction analysis (XRD).[11] While evaluating the surface properties, devices such as scanning electron microscopes (SEM), atomic force microscopes (AFM), and profilometry are also used.[12] The Vickers test is widely used to evaluate the microhardness of dental materials.[13] Flexural strength tests are reliable in evaluating the strength of fragile materials. The three-ball piston technique, which is one of the biaxial flexural tests, is a frequently used method.[14]

The present study aims are to evaluate the effect of autoclave aging with different surface polishing systems on the surface properties, phase transformation, and flexural strength of monolithic zirconia.

The null hypothesis was that surface polishing systems applied to monolithic zirconia would increase the surface roughness and surface microhardness and decrease the phase transformation rate and flexural strength, and that autoclave aging would not affect the surface properties, phase transformation ratio, and flexural strength.

METHODS

The power analysis was calculated according to the previous article by considering the effect size = 2, beta error = 0.80, and alpha error = 0.95. Finally, it was decided that eight specimens for each group would be adequate for statistical evaluation.[15] A total of 48 pre-sintered specimens from a Prettau® Zirconia monolithic block were prepared using CAD/CAM (Yenadent D15, Yenadent Ltd., Turkey). Pre-sintered monolithic zirconia specimens were sintered in an Ivoclar Vivadent Programat P300 furnace (Ivoclar Vivadent AG, Germany) with final dimensions of 15 mm in diameter and 1.2 mm in thickness. A thin layer of glaze was applied to all specimens.

Formation of experimental groups

The specimens were divided into three groups. The surfaces of the specimens, except the control group, were abraded by the same user for 20 seconds at 20,000 rpm with a back-and-forth sweeping motion using blue band burs (881Z4, Meisinger, Neuss, Germany) containing 100-µm diamond particles recommended for monolithic zirconia restorations under water coolant with an aerotor. Two different surface polishing systems were applied to the monolithic zirconia specimens (32 specimens), which were etched.

Blue and pink rubbers were applied to 16 specimens by the same researcher for 30 seconds with Meisinger Luster LUS91 intraoral polish kit at 10,000 rpm under water coolant. On the other 16 specimens, F (coarse grit) and P (fine grit) rubbers in the OptraFine (Ivoclar Vivadent) polish kit were applied by the same researcher for 30 seconds under water coolant at 10,000 rpm. Finally, polish paste was applied with nylon brushes without water coolant.

The specimens in all groups were divided into two groups (n=8). One of the groups was aged in an autoclave (Getinge, Getinge Group) at 134°C under 0.2 MPa pressure for 5 hours. And the other group (control) was not aged.

Examining the surface properties of the specimens

Surface roughness was measured using a profilometer (Surtronic 25, Taylor Hobson Leicester, UK).

The surface microhardness values of the specimens were determined by measuring the diagonals of the traces under 200 g of load for 15 s using a Vickers microhardness device (TMTeck HV-1000B, TMTeck Manufacturing Limited, Beijing, China).

The surface of a randomly selected specimen from each group (20 μm^2) was scanned using an AFM (Hitachi 5100N, Tokyo, Japan) with a speed of 1.6 Hz and a vibration frequency of 10 KHz, and three-dimensional images were obtained with a resolution of 256×256 pixels.

A randomly selected monolithic zirconia specimen was gold plated in a gold plating device (SC7620, Quorum Technologies, Newhaven, UK) because its surface was non-conductive. Afterward, the surfaces of the specimens were evaluated using a SEM (JSM-6610, Jeol, Peabody, USA).

Examining the phase transformations of specimens

Three randomly selected specimens from each group were analyzed by scanning with an XRD device (Rigaku Smart-Lab Diffractometer, Tokyo, Japan) at 40 KV voltage and 30 mA current at 20-40 degrees using monochromatic CuK alpha rays (2ϴ). The amount of phase-changed monoclinic and tetragonal phases of the specimens was evaluated using the formula of Garvie and Nicholson:[16]

Equation: Xm=[Im(-111)+Im(111)]/[Im(-111)+Im(111)+It(101)]

I: The highest value of the phase density

Im (-111): Plane showing the crystal geometry of the monoclinic phase (-111)

Im (111): Plane showing the crystal geometry of the monoclinic phase (111)

It (101): Plane showing the crystal geometry of the tetragonal phase (101)

Evaluation of flexural strength data of specimens

To evaluate the flexural strength of monolithic zirconia specimens, a three-ball piston technique was used in the universal test device (Instron 3340, Wycombe, UK). Three steel balls with a diameter of 3.2 mm were placed on a metallic platform with a diameter of 10 mm, equidistant from each other. A force was applied to the treated surfaces of the specimens with a cylindrical apparatus with a diameter of 1.4 mm at the tip, at a loading speed of 0.15 mm per minute. The breaking force of each specimen was recorded in Newtons and the flexural strengths were converted to MPa in accordance with the piston test on three balls. σ is the biaxial flexural strength (MPa), P is the total load up to flexural strength in Newtons, and d is the specimen thickness in mm.

$$\sigma = -0.2387P\,(X - Y) / d^2$$

X and Y were obtained as follows:

$$X = (1 + \nu) \ln (r2 / r3)^2 + [(1 - \nu) / 2]\,(r2 / r3)^2$$

$$Y = (1 + \nu)\,[1 + \ln (r1 / r3)^2] + (1 - \nu)\,(r1 / r3)^2$$

ν Poisson's ratio (=0.23); r1 is the radius of the support circle, r2 is the radius of the charged area, r3 is the radius of the specimen; all are expressed in millimeters.[15]

Statistical analysis

The normality of the data was detected with Shapiro-Wilk test. Two-way ANOVA and Tukey's post hoc tests were used for comparing the differences at a significance level of *P*<.05 (SPSS v29.0, Chicago, IL, USA).

RESULTS

The mean (Ra) and standard deviation results of the surface roughness data of monolithic zirconia specimens are shown in Table 1. It was determined that the difference between the applied surface polishing systems was significant (*P*<.05), and autoclave aging was not significant. The highest value was found in the Meisinger polish kit group.

The mean and standard deviation results of the surface microhardness data of the specimens applied with different surface polishing systems are shown in Table 2. It was determined that the different surface polishing processes applied were significant (*P*<.05), and the autoclave aging process was not significant.

When the Meisinger polish kit group was compared with the OptraFine polish kit group, it was determined that the Meisinger polish kit increased the surface microhardness, but this increase was not statistically significant.

Three-dimensional images of the monolithic zirconia specimens, on which surface polishing systems were applied, were taken using the AFM device before and after the aging process in the autoclave (Figure 1). When the images obtained from the specimens in the control group were evaluated, the surface was smooth and free of small bumps before and after autoclave aging.

Table 1. Mean and standard deviation test results of surface roughness data (Ra) (µm)

Surface Polishing Systems	Autoclave Aging Process	
	Non-aged*	Aged*
Control	0.31 (0.03) ^{a,A}	0.37 (0.04)^{a,A}
Meisinger Polishing Kit	0.60 (0.26)^{b,A}	0.76 (0.32)^{b,A}
OptraFine Polishing Kit	0.52 (0.27)^{b,A}	0.66 (0.7)^{b,A}

**: Vertical significant differences are shown in lower case letters, horizontal significant differences are shown in capital letters.*

Table 2. Mean and standard deviation results of surface microhardness values (HV)

Surface Polishing Systems	Autoclave Aging Process	
	Non-aged*	Aged*
Control	540.72 (77.93)^{a,A}	532.75 (81.19)^{a,A}
Meisinger Polishing Kit	1168.30 (141.87)^{b,A}	1110.60 (80.89)^{b,A}
OptraFine Polishing Kit	1059.31 (163.67)^{b,A}	1043.30 (168.56)^{b,A}

**: Vertical significant differences are shown in lower case letters, horizontal significant differences are shown in capital letters.*

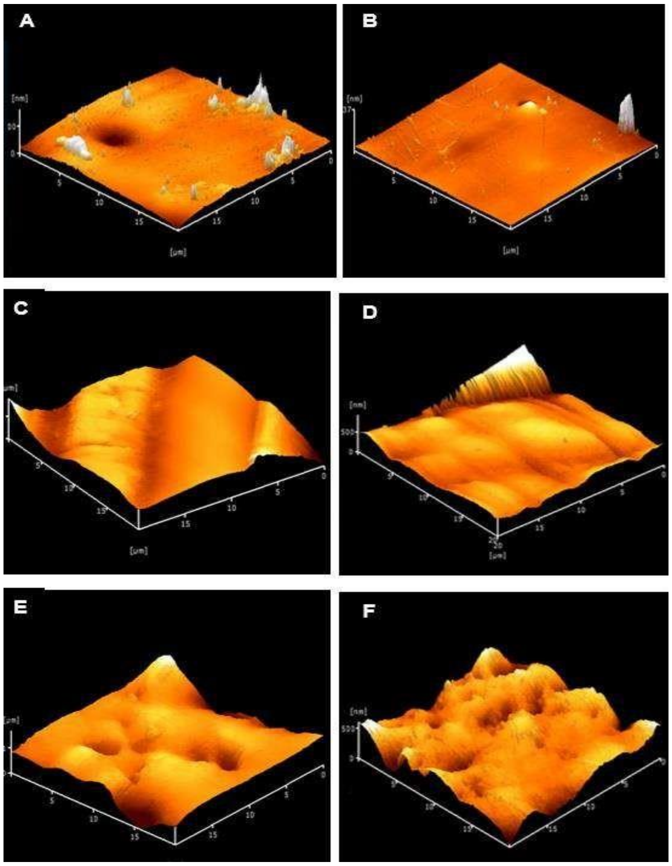

Figure 1. (A) AFM image of the control group **(B)** AFM image of the control group after autoclave aging **(C)** AFM image of the Meisinger polishing kit group **(D)** AFM image of the Meisinger polishing kit group after autoclave aging **(E)** AFM image of the OptraFine polishing kit and paste group **(F)** AFM image of the OptraFine polishing kit and paste group after autoclave aging

In the group in which the Meisinger polishing kit was applied, bur traces were evident and fluctuations were found before aging. The surface roughness increased after aging in the autoclave and irregular pitting was observed.

In the specimens where the OptraFine polishing kit and paste were applied, clear pits were observed on the surface before aging in the autoclave, and a rough surface was observed with the increase in the depth of these pits after aging in the autoclave.

When the SEM images of the control group were examined, a homogeneous image and indistinct point residues were determined on the surface of the specimens that did not undergo any treatment after glazing. It was determined that autoclave aging did not cause any significant surface difference in all groups.

After the etching process, inhomogeneous pits and lines were obtained in the group in which the Meisinger polish kit was applied. Inhomogeneous pits and fluctuations due to etching were detected in the group to which the OptraFine polish kit was applied (Figure 2).

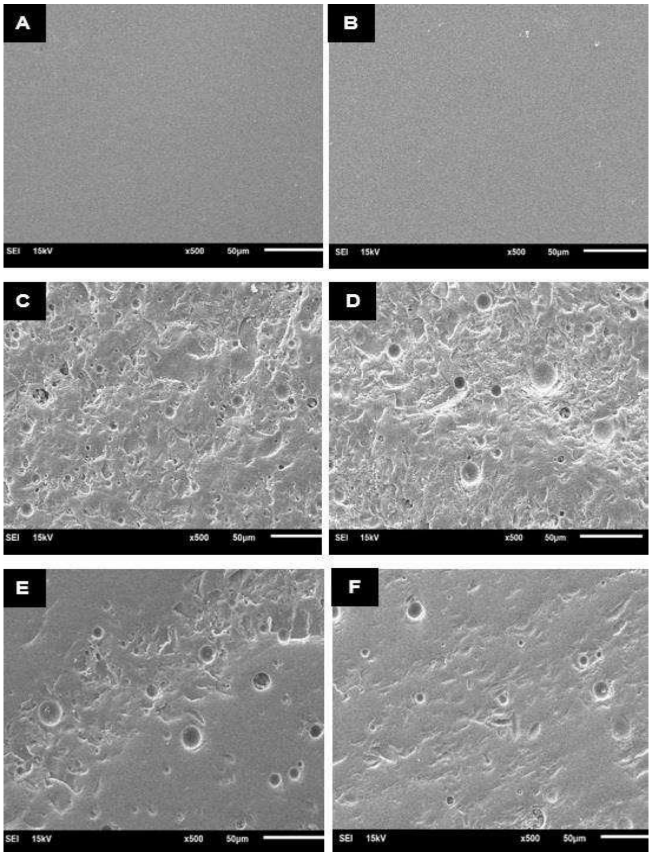

Figure 2. (A) SEM image of the control group **(B)** SEM image of the control group after autoclave aging **(C)** SEM image of Meisinger polishing kit group **(D)** SEM image of Meisinger polishing kit group after autoclave aging **(E)** SEM image of the Optrafine polishing kit and paste group **(F)** SEM image of the Optrafine polishing kit and paste group after autoclave aging

The mean and standard deviation results of the phase transformation amounts of the specimens are shown in Table 3. It was determined that the effect of the surface polishing systems applied to the specimens and the autoclave aging process on the amount of phase transformation was statistically significant (*P*<.05).

Table 3. Mean and standard deviation results of phase transformation data

Surface Polishing Systems	Autoclave Aging Process	
	Non-aged*	Aged*
Control	21.08 (4.01)^{a,A}	20.09 (6.35)^{a,B}
Meisinger Polishing Kit	20.10 (4.34)^{b,A}	18.97 (6.02)^{b,B}
OptraFine Polishing Kit	19.37 (2.28)^{b,A}	17.76 (5.76)^{b,B}

**: Vertical significant differences are shown in lower case letters, horizontal significant differences are shown in capital letters.*

Table 4. Mean and standard deviation results of flexural strength values (MPa)

Surface Polishing Systems	Autoclave Aging Process	
	Non-aged*	Aged*
Control	674.50 (130.87) ^{a,A}	630.38 (108.87)^{a,A}
Meisinger Polishing Kit	605.25 (85.30)^{b,A}	555.50 (130.88)^{b,A}
OptraFine Polishing Kit	663.00 (90.46)^{b,A}	582.12 (120.20)^{b,A}

**: Vertical significant differences are shown in lower case letters, horizontal significant differences are shown in capital letters.*

Although the phase transformation values obtained in the Meisinger polishing kit and the OptraFine polishing kit groups were lower than those obtained in the control group, no significant difference was found between the two groups.

The mean and standard deviation results of the flexural strength amounts of the specimens are shown in Table 4. It was determined that the surface polishing systems were statistically significant (*P*<.05) and the autoclave aging process was not statistically significant.

Although the flexural strength values obtained in the Meisinger polishing kit and OptraFine polishing kit groups were lower than those of the control group, the difference between the Meisinger polishing kit and OptrafFine polishing kit groups was not statistically significant.

DISCUSSION

In the present study, the effect of autoclave aging with different surface polishing systems on the surface properties, phase transformation, and flexural strength of monolithic zirconia was investigated. Surface polishing systems applied to monolithic zirconia specimens increased the surface roughness and surface microhardness and decreased the phase transformation and flexural strength. However, the effect of autoclave aging on the amount of phase transformation was significant, surface roughness and surface microhardness and flexural strength were no significant. Accordingly, some of the null hypotheses were accepted and some were rejected.

Occlusal adjustments of monolithic zirconia restorations are routinely performed in clinics by dentists. The goal is obtain the smooth surface after occlusal adjustments. For this reason restorations are regulated reglaze or polishing in the chairside. For monolithic zirconia, polishing instead of glazing has been reported to reduce surface roughness.[17]

Steiner et al.[18] applied a mechanical polishing process on five different ceramics with five different polishing systems (Komet, Optra-Fine, Shofu, Zenostarm and Diatech) and found the smoothest surface in the glazed specimens. They also determined that the OptraFine polishing kit made the surfaces of the specimens smoother due to the paste content. Although it has been reported that the roughness of conformalized surfaces can be reduced by polishing, there are studies showing that the surface obtained is not as smooth as a glazed surface.[19] In the present study, after the polishing systems were applied to the monolithic zirconia specimens, the surface roughness was evaluated using a profilometer device and the lowest surface roughness values (0.31 Ra) were found in the specimens in the control group, which were non-aged and glazed. After applying the polishing rubbers included in the OptraFine polishing kit, a polishing paste was used. Thanks to this paste, lower surface roughness values (0.52 Ra) were obtained than those obtained with the Meisinger polishing kit (0.60 Ra). Polishing kits made with polishing paste containing diamond particles provide smooth surfaces.[20] However, the most smoothest surface is obtained with glaze.

In the present study, a 5-hour aging process was applied to simulate 15-20 years of aging. There was no statistically significant difference between the surface roughness values before and after the autoclave aging process. Pereira et al.[21] reported that aging (20 hours in an autoclave at 134°C at 2 bar pressure) did not significantly increase the roughness of monolithic zirconia.

The microstructure of the material is affected by the properties of the crystal phase, and yttria content. The resistance of the material is measured by the surface microhardness test.[21] The increase in microhardness in the OptraFine polishing kit and Meisinger polishing kit groups could be attributed to the transformation t→m. Pittayachawan et al.[22] reported a phase transformation t→m induced by HV (Vickers) testing. In the present study, the highest surface microhardness value was found in the non-aged OptraFine polishing kit group (1059.31 HV). Meisinger and OptraFine polishing kit statistically significantly increased the surface microhardness compared to the control group. Roy et al.[23] compared the surface microhardness values of aged and non-treated Y-TZP ceramics and stated that non-aged Y-TZPs had higher microhardness values. It may be due to non-aged specimens' crystal phase.

Zirconia restorations are completely crystalline therefore phase change within the material could be assessed by XRD analysis.[24] In the present study, it was determined that the effect of different polishing systems and autoclave aging on phase change was statistically significant. Although it was determined that the surface roughness of the specimens increased after aging in the autoclave, the difference was not statistically significant. Kohorst et al.[25] applied autoclave aging for 16, 32, 64, and 128 hours on zirconia specimens, except for the control group. In the analyses performed after aging in the autoclave, 3Y-TZP surfaces became rough due to the increased monoclinic phase content. They stated that this was due to the significant elevation of the phase-transformed zirconia crystals.

In the present study, the flexural strength of the specimens was evaluated after the application of etching and polishing kits and a decrease in flexural strength was observed in all groups. Although autoclave aging was statistically not significant, Meisinger and OptraFine polishing kits significantly reduced the flexural strength. After 5 h ageing process, monoclinic-phase transformation is reported to be induced and low-temperature degradation may occur due to the steam. For the polished specimens, the initial ageing for 5 h must have relieved the mechanically induced surface stress, resulting in a reduction in biaxial flexural strength.[26]

Moon et al.[27] reported that surface polishing processes caused phase transformation of zirconia, reducing its transformation capacity and was thus less resistant to applied forces. The t→m transformation start from the surface of the specimen and proceed with microcracks. The microcracks progress to cause major cracks. The flexural strength of the monoclinic phase is lower than that of the tetragonal or cubic phases.[28] More clinical studies should be conducted to test the longevity of repolished zirconia restorations.

There are limitations in the present study. A single type of zirconia block is used in the present study. Since there is no comparison with different brands of blocks, a general opinion cannot be reached. The content and features of each polishing kits are vary. Therefore, when using another polishing kit, the same results may not be found.

CONCLUSION

The lowest surface roughness values were found in the specimens in the control (glazed) group. When polishing systems were compared, a smoother surface was obtained in the OptraFine polish kit and the highest surface microhardness was found in the group to which the Meisinger polish kit was applied. The effect of the applied polishing systems and the autoclave aging process on the phase transformation was found to be statistically significant. It was determined that the effect of surface polishing systems on flexural strength was significant, while the effect of autoclave aging was not significant.

Ethics Committee Approval: Ethical approval was not required because this study was conducted in vitro and did not involve human or animal subjects.

Peer-review: Externally peer-reviewed.

Author Contributions: Concept –GA, ZY; Design -GA, ZY; Supervision -GA, ZY; Resources -GA, ZY; Materials – GA, Data Collection and Processing –GA, ZY; Analysis and Interpretation -GA, ZY; Literature Search -GA, ZY; Writing Manuscript -GA; Critical Review -GA, ZY.

Conflict of Interest: The authors have no conflicts of interest to declare.

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Hakem Değerlendirmesi: Dış bağımsız.

Yazar Katkıları: Fikir -GA, ZY; Tasarım – GA, ZY; Denetim – GA, ZY; Kaynaklar – GA, ZY; Materyaller – GA; Veri Toplama ve İşleme – GA, ZY; Analiz ve Yorum – GA, ZY; Literatür Taraması – GA, ZY; Makaleyi Yazan – GA; Eleştirel İnceleme – GA, ZY.

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Yapay Zeka Kullanımı: Bu çalışmanın hazırlanması sırasında yapay zekâ destekli herhangi bir araç, model veya teknoloji bilimsel içerik üretmek amacıyla kullanılmamıştır. Tüm analizler, yorumlar ve yazım süreçleri yazar(lar) tarafından gerçekleştirilmiştir.

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