Original Article / Özgün Araştırma

Effect of Surface Treatments on Leakage of Zirconium Oxide Ceramics

Farklı Yüzey İşlemlerinin Zirkonya Seramiklerin Sızıntısı Üzerine Etkisi

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Abstract

Objective: The aim of this pilot study was to compare the effects of pretreatments on leakage of zirconia ceramics.
Materials and Methods: The speciments divided into 6 groups that were subsequently treated as follows: group 1, no treatment (control); group 2, the ceramic surfaces were airborne-particle abraded with 110 µm aluminum-oxide (Al_2O_3) particles; group 3, after abrasion of the surfaces with 110 µm Al_2O_3 particles, silica coating using 30 µm (Al_2O_3) particles modified by silica (rocatec system) and application of the silane coupling agent (espe-sil); group 4, ceramic surfaces irritated with neodymium-doped yttrium aluminium garnet (Nd:YAG) laser [fidelis plus 3 foton (Ljubljana, Slovenia)] at 20 hz, 100 mj, 2 w, 100 µs; group 5, ceramic surfaces irritated with Nd:YAG laser at fidelis plus 3 fotona (Ljubljana, Slovenia) at 20 hz, 100 mj, 2 w, 100 µs; group 6; application of a zirconia primer (z-prime plus bisco, IL, USA) agent. And all ceramics tested for leakage.
Results: For marginal leakage, score 0 was found in all groups.
Conclusion: No significant differences were found in marginal leakage under all conditions.

Keywords
Leakage, resin cement, zirconia ceramics

Anahtar Kelimeler
Sızıntı, rezin siman, zirkonya seramikler

Öz

Amaç: Bu pilot çalışmanın amacı farklı yüzey işlemleri uygulanmış zirkonya yüzeyi ile rezin siman arasındaki sızıntının değerlendirilmesidir.
Gereç ve Yöntemler: Farklı yüzey hazırlıklarına tabi tutulmak üzere 6 gruba ayrılan örneklerden 1. grup yüzey işlemi uygulanmadan kontrol grubu (grup 1) olarak ayrıldı. Grup 2'deki örnekler 110 µm olan alüminyum oksit (Al_2O_3) kumu ile pürüzlendirildi. Grup 3'teki örnekler 110 µm olan Al_2O_3 kumu ile pürüzlendirildikten sonra silika kaplandı ve silan (espe-sil) uygulandı. Grup 4'teki seramikler, neodymium: yttrium-aluminum-garnet lazer cihazı [fidelis plus 3 fotona (Ljubljana, Slovenia)] kullanılarak pürüzlendirildiler. Cihazın paneli 20 hz, 100 mj, 2 w, 100 µs olacak şekilde ayarlandı. 5. gruptaki seramikler de lazerle pürüzlendirildi ve cihaz; 20 hz, 150 mj, 3 w, 100 µs olarak ayarlandı. Grup 6'daki örneklere ise çift kat zirconia primer (z-prime plus bisco, İL, ABD) uygulandı. Hava ile 10 sn kibarca kurutuldu 20 sn 800 mw/cm^2 ışıkla polimerize edildi ve tüm seramikler sızıntı testine tabi tutuldu.
Bulgular: Sızıntı testine tabi tutulmuş örneklerde marjinlerde ince bir çizgi şeklinde sızıntı izlenmiştir.
Sonuç: Farklı yüzey işlemleri uygulanan örneklerde sızıntı değerleri arasında fark bulunamamıştır.

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Introduction

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) were introduced in the biomaterials world many years ago (1) Indications for zirconia restorations have been extended in recent years due to their superior esthetic appearance and physical properties, such as high strength and toughness (2,3). Due to these improved mechanical properties, zirconia ceramic was introduced to restorative dentistry for the restoration of posterior teeth (1,4). Clinical applications of Y-TZP include full-coverage crowns, implant abutments, endodontic posts, and small bridges (5-9). Luting of a zirconia restoration can be done with conventional luting agents. However, the advantages of resin bonding, e.g. improvement of retention, marginal adaption, fracture resistance and inhibition of secondary caries, have made them more frequently used (10-13). A strong and long-term durable adhesive bond is key to the clinical survival of the restoration (10,14). The use of silanes and hydrofluoric (HF) acid is recommended for silica-based ceramics. However, this chemical reaction is not possible for zirconia-based ceramics because of their high crystalline content which makes them resistant to HF acid etching (9,14,15). Long-term stable ceramic-resin bonds require surface pretreatment such as acid etching, sand blasting, airborne-partical abrasion with aluminum oxide (Al_2O_3) particles, modified priming and/or resin composite luting agent that contains special adhesive monomers [e.g. 10-methacryloyloxydecyl dihydrogen phosphate (MDP)], treating with laser beam or combinations of these methods (9,14,15). Treating zirconia surface with laser beam is a quite new method but the obtained results are promising (16-18). The purpose of this study was to examine the null hypothesis that different surface treatments affect marginal leakage of zirconia ceramics.

Materials and Methods

In this study, the leakage of zirconia all-ceramics subject to different surface procedures with Panavia F 2.0 resin cement was investigated and the zirconia samples were obtained from prefabricated Zirkonzahn blocks. Cutting procedure was done using a precision cutter (Minitom, Struers, Denmark) with a special water-cooled diamond wheel (M0D10, Struers, Denmark) in dimensions of 13x13x2.5 mm. After flash pickling process at the corners, the samples were sintered according to the instructions of the manufacturer, in a Zirkonzahn sintering oven at 1500 °C for 8 hours. After sintering, samples' dimensions changed approximately 25%. The final dimensions of the samples were measured with a digital caliper as 10x10x2 mm. Then, the specimens were ultrasonically cleaned with distilled water for 3 minutes and carefully dried in air before surface treatment. The surface pretreatment protocols applied to zirconia surfaces are shown in Table 1.

Group 1: Control group, no treatment.

Group 2: Sand blasting (Rocatec Junior 3M ESPE, USA) with a size of 110 µ Al_2O_3 particles (Rocatec-Pre powder, 3M ESPE, Germany) was applied perpendicularly to the surface at a 10 mm distance and pressure of 2.8 psi for 13 seconds.

Group 3: Rocatec Plus powder (3M ESPE, USA) was blasted to the surface for 10 seconds using the same parameters as for samples in the group 2. Before cementation, the silane coupling agent ESPE Sil (3M ESPE, Germany), a component of the Rocatec system, was applied to the specimens and waited for 5 minutes according to the instructions of the manufacturer.

Groups 4-5 laser groups: The specimens in these groups were treated with Fidelis Plus 3 Fotona (Ljubljana, Slovenia) laser device. Since the reflectance of the ceramic samples is very low, the surfaces

Table 1. Surface pretreatment protocols applied to each ceramic

Group	Name	Pretreatments
1	Control	No treatment
2	Sand blasting	Rocatec-Pre powder with 110 µ Al_2O_3 particle size
3	Rocatec	Rocatec-Pre powder+Rocatec plus powder with 110 µ Al_2O_3 particle size+silane
4	Laser 1	20 Hz, 100 mJ, 2 w 100 µs-Nd-YAG laser
5	Laser 2	20 Hz, 150 mJ, 3 w 100 µs-Nd-YAG laser
6	Primer	Zirconia primer

Nd-YAG: Neodymium-doped yttrium aluminium garnet were washed with distilled water and stained with polymerized powdered carbon (graphite powder) before using laser.

Group 4: Ceramic surfaces were irritated with neodymium-doped yttrium aluminium garnet (Nd:YAG) laser. The Nd: YAG laser parameters were: 20 Hz , 100 mJ (energy in per pulse), 2 w (total pulse energy), 100 µs (pulse duration).

Group 5: For this group, the same laser system was employed at 20 Hz, 150 mJ, 3 w, 100 µs. In both laser etching groups, the ceramic surfaces were scanned with horizontal movements, perpendicular to the surface with a focused spot size of 1 mm fiber hand piece.

Group 6: After air rinsing, Zirconia Primer (Z-Prime Plus Bisco, IL, USA) was applied to the surfaces, gently dried for 10 seconds and light cured for 20 seconds at a light intensity of 800 mW/cm^2.

After surface pretreatments were performed; equal amounts of pastes A and B (Panavia F 2.0 -Kuraray Medical Inc., Japan) were mixed for 20 seconds on the mixing paper and applied on both two specimens. All ceramic samples subject to leakage test were cemented in the tension/compression device after the processed surfaces of the two samples in every group had been placed face-to-face. A weight of 750 g was applied for 10 minutes using the tension/compression device. After that loading excess luting agent, which exuded from the bonding surfaces, was cleaned with foam pellets and oxygen-blocking gel (Oxyguard, Kuraray, Osaka, Japan) was applied according to the manufacturer's instructions. Resin material was light polymerized from four directions for 40 seconds. Stain infiltration method (Figure 1) (45). Luted specimens were stored in 0.5% fuchsine solution at room temperature for 24 hours. The samples were cleansed from excess stains under running water for 5 minutes. The leakage scores of all samples were photographed.

Results

For all the pretreated specimens, score 0 was found (Figure 2).

Discussion

The purpose of the prosthetic restorations is to regain the aesthetic appearance with the proper functional structure. Studies focused on this objective, regarding the fixed restorations, facilitated the development of all-ceramic systems using durable core structures instead of metal substructures. It is reported that cement and cementation process are important in the clinical success of all-ceramic

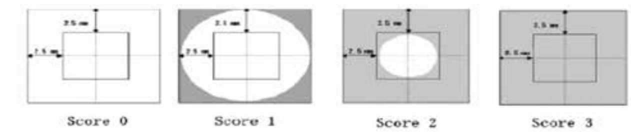

Figure 1. Stain infiltration method (45)
0: No stain infiltration, 1: Stain infiltrated in less than ½, 2: Stain infiltrated in more than ½, 3: Stain infiltrated in whole

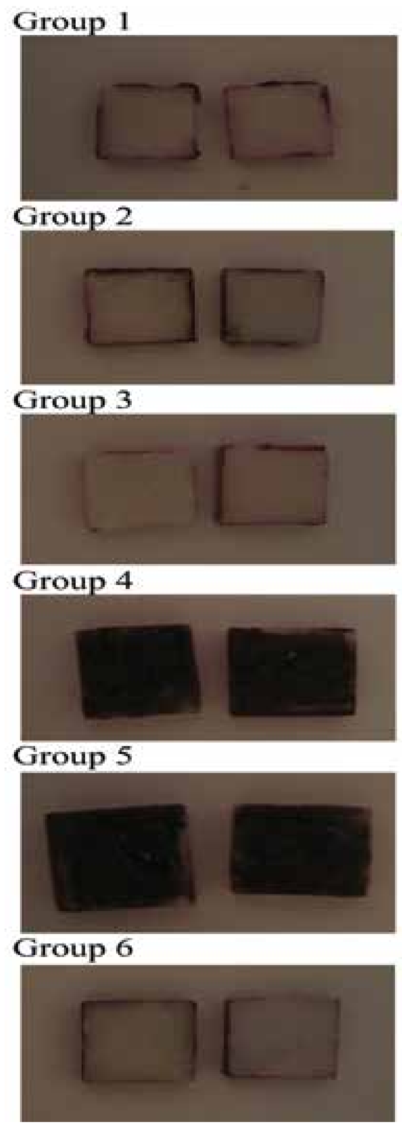

Figure 2. Leakage surfaces

restorations. The cement should cover the entire restoration-tooth interface with a mechanic and chemical mechanism or with a combination of these and prevent microbial leakage (19,20). Until recently, although the usage of all-ceramic systems was limited to anterior teeth, further development of the ceramic systems facilitated their implementation also for the posterior teeth. Today, there are various ceramic systems, which provide high-quality esthetics for many different clinical practices (21). After the clinical success of alumina-based ceramics, zirconia ceramics came in use in the dentistry. Zirconium oxide as a substructure material is preferred due to its advantages like high bending stiffness, biocompatibility, satisfying optical features and making multiple-unit fixed restorations possible. As zirconium is a very durable material, it has a wide range use in dental practice (15,21-23). Conventional cement can be used for bonding of all-ceramic restorations with high durability like zirconia oxide. As the surface of zirconia all-ceramic restorations does not contain a glass phase, it cannot be etched with HF acid like conventional ceramics. This prevents the formation of micromechanical adhesion. The physical strength of conventional cement depends on micromechanical adhesion, as it does not possess adhesive features (24,25). We put the mechanical adhesion aside, currently, the only way of an effective chemical bonding to zirconia is the use of phosphate ester monomers (10-MDP). This monomer, resistant to hydrolyzation, provides a stable adhesion during different ageing methods. Lots of studies have reported that the highest stability in the shear-bond was found in samples cemented with Panavia F 2.0 and carried out with a priming agent containing 10-MDP (26-29). As MDP monomer is water resistant and it is believed that the resin-based cement has an effect of “treating” the microcracks on the ceramic surfaces and of strengthening the ceramic, resin cement containing MDP are recommended (29,30). In addition to that, in resin cementation, the possibility of fracture of the restoration and the supportive tooth is decreased compared to the conventional bisphenol-A-glycidyl dimethacrylate (Bis-GMA) containing cements. Therefore, many investigators recommended the usage of adhesive resin-based cement in the regions where adhesion is important because of the strong occlusal forces (10,31-34). In their study which focused on alumina disks infiltrated with glass, Kern and Thompson (35), have reported that the adhesive resistance of samples cemented with Bis-GMA composite resin had lower bond strength than the samples bonded with MDP-containing composite resin. They suggested that the reason for this was the direct bonding of phosphate ester groups in the monomer to the metal oxides, hence, to the alumina and zirconia ceramics (14,31,36,37).

Considering all these features, we used dual-polymerized Panavia F 2.0 containing MDP monomer in our study. However, in dual-polymerized cement, the completion of the polymerization lasts approximately 24 hours in regions with limited exposure to light (14,38,39). Therefore, in our study, we kept our samples in water for 24 hours after cementation. Regarding the comparison of the methods used with silica-based ceramics, only a few methods are recommended in the literature for increasing the adhesion force of the zirconium oxide ceramics to resin cement. These studies focused rather on the usage of agents containing special adhesive monomers like MDP or micro chiseling of the surface with the Al_2O_3 salts with different particle sizes (21,36,40). In addition to these methods, procedures aiming the increase of silica content of the surface in zirconia-based ceramics were also applied. For this purpose, manufacturers recommend various products (rocatec, cojet, pyrosil Pen). There are also studies showing that adhesion force may be enhanced with the plasma spray method. Treating ceramic surfaces with laser energy is a relatively newer method (21,41-43). Sand particles with particle sizes of 50, 100, 110 or 250 µ can be used for treating the surfaces before the cementation of the porcelain surfaces. On the other hand, in several previous studies, mostly Al_2O_3 particles with a size of 110 µ was used (21,25,29). Therefore, in our study, we also preferred sand blasting with a particle size of 110 µ Al_2O_3 for surface treatment. Some in vitro studies have showed that air abrasion damages the integrity of high durable ceramics. However, in few studies, it has been reported that air abrasion is necessary for long-term resin bonding of the high durable ceramics (43,44). As zirconium is a highly reflective material, it cannot interact directly with the Nd-YAG laser beam. In both laser groups, ceramic surfaces were painted with polymerized powdered carbon (graphite powder) in order to increase the efficacy of the laser beams on the material (16). We preferred powdered carbon because of its dark color, easy-cleaning after the procedure and its common usage in similar studies.

However, as it can be seen on the photographs (Figure 2), it caused grey-black staining on the sample surfaces and could not be removed with ultrasonic bathing. In order to evaluate the effect of this observation on bondings and hence on infiltration, further studies are needed. Evaluation of leakage in ceramics treated with different surface procedures was performed only in a limited number of studies. Tsukakoshi et al. (45) investigated the leakage in their study and found that the samples covered with silica with Rocatec system displayed highest adhesion forces in all groups with different cement. However, all resin-based cement groups revealed rather different adhesion forces compared to the glass ionomer cement reinforced with resin. Regarding the leakage evaluation, almost in all groups with resin cement-surface procedure, scores were registered as "0". Rominu et al. (46), in their study on microleakage, have reported that there was always microleakage in the metal-resin interface independent from the resin used and it could not be prevented completely with chemical bonding systems. In line with the studies in the literature, in our study, we also obtained similar results after keeping the samples in basic fuchsin for one day and after a one-week follow-up, the leakage values were registered as "0". However, we observed leakage as a thin line at the corners of the restoration. There was no difference between the groups. The null hypothesis is accepted that different surface treatments did not affect the leakage of ceramic surfaces (p>0.05).

Conclusion

Within the limitations of this in vitro study; no significant differences were found in marginal leakage under all conditions.

Ethics

Ethics Committee Approval: A pilot study, Informed Consent: A pilot study.

Peer-review: Internal peer-reviewed.

Authorship Contributions

Concept: Mustafa Zortuk, Design: Mustafa Zortuk, Data Collection or Processing: Göknil Alkan Demetoğlu, Mustafa Zortuk, Analysis or Interpretation: Göknil Alkan Demetoğlu, Mustafa Zortuk, Literature Search: Göknil Alkan Demetoğlu, Writing: Göknil Alkan Demetoğlu.

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