

Effect of Surface Finishing Methods and Aging on Surface Roughness and Color Stability of Hybrid Ceramic

Yüzey Bitirme İşlemleri ve Yaşlandırmanın Hibrit Seramik Materyalde Yüzey Pürüzlülüğü ve Renk Stabilitesine Etkisi

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ÖZET

Amaç: Bu çalışmanın amacı, dört farklı yüzey bitirme metodunun ve yaşlandırma işleminin dental hibrit seramik materyalde yüzey pürüzlülüğü ve renk stabilitesi üzerine etkisinin değerlendirilmesidir.

Gereç ve Yöntem: Hibrit seramik bloklar 12x14x1 mm boyutlarında hazırlandı ve örnekler yüzey bitirme metodlarına göre; teknisyen kitiyle polisaj (ET), klinik kitiyle polisaj (EC), kumlanan yüzeye sealant ajanı uygulama (ES) ve asitlenen yüzeye sealant ajanı uygulama (EA) olmak üzere 4 alt gruba ayrıldı (n=10). Renk parametreleri ve yüzey pürüzlülük değerleri ölçüldü. Tüm örnekler yaşlandırma işlemi uygulandı. Renk ve yüzey pürüzlülük değerleri tekrar ölçüldü. Renk değişim (ΔE) değerleri hesaplandı ve verilerin analizi Kruskal Wallis, Bonferroni Dunn ve Wilcoxon signed ranks testleri kullanılarak yapıldı. Her gruptan 2 örnekte Taramalı Elektron Mikroskopuyla (SEM) yüzey analizi yapıldı.

Bulgular: Farklı yüzey bitirme işlemi uygulanan grupların ΔE değerleri arasında istatistiksel olarak anlamlı fark gözlenmedi. Yaşlandırma işlemi öncesi ve sonrasında, EA grubu en düşük yüzey pürüzlülük değerlerini gösterdi. Yaşlandırma işlemi sonrasında ES grubunun yüzey pürüzlülük değerleri yükseldi.

Sonuç: Hibrit seramik materyalin renk stabilitesi yüzey bitirme metodundan etkilenmedi ve en düşük yüzey pürüzlülük değerleri asitlenmiş yüzeye sealant ajanı uygulanan grupta görüldü.

Anahtar Kelimeler: Hibrit seramik; Pürüzlülük; Renk; Yaşlandırma; Yüzey işlemi

ABSTRACT

Aim: The aim of this study is to evaluate the effect of four different surface finishing methods and aging on the surface roughness and color stability of dental hybrid ceramic.

Material and Methods: Hybrid ceramic blocks were prepared with dimension of 12x14x1 mm and 4 groups were determined as; polishing with technical kit (ET), polishing with clinical kit (EC), applying sealant agent coupling on sandblasted surface (ES) and applying sealant agent coupling on etched surface (EA) (n=10). Color parameters and surface roughness values were measured. Thermocycling procedure was applied. Color and surface roughness values were re-measured. ΔE values were calculated and data was analyzed with Kruskal Wallis, Bonferroni Dunn and Wilcoxon signed ranks tests. Surface analysis were performed with scanning electron microscope to 2 specimens of each group.

Results: Significant difference was not observed among ΔE values of the groups with different types of surface finishing procedure. Before and after aging, EA group had lowest surface roughness values. After aging, surface roughness values increased for ES group.

Conclusion: The color stability of hybrid ceramic material was not affected by the surface finishing method, and the lowest surface roughness values were observed in the applying sealant agent coupling on sandblasted surface group.

Keywords: Color; Hybrid ceramic; Roughness; Surface treatment; Thermocycling

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INTRODUCTION

Ceramics and resin composites have been widely used to produce dental restorations.¹ Hybrid ceramics, which are used with computer aided design/computer aided manufacturing (CAD/CAM) systems, were developed to optimize the performance of these restorative materials.² Elastic modulus of hybrid ceramics is similar to the dentin and less wear are created on the opposing teeth surface.³ Besides, easy milling and intraoral reparability are other advantages of hybrid ceramic materials.⁴

The composition and microstructure of dental materials affects mechanical and physical properties.¹ Vita Enamic, which is a hybrid ceramic material, consists of sintered ceramic network (86 wt%) and polymer matrix (14 wt%) with both networks fully penetrating one another.⁵ After milling process, the surface of Vita Enamic restoration is rougher than feldspatic ceramic because the polymer matrix of Vita Enamic is easily separated from the ceramic network.¹ Rough surfaces may be caused plaque accumulation, discoloration of restoration and wear of opposing teeth.^{6,7} As a result, a reduction in the surface roughness of restorations is mandatory and reduction can be obtained by surface finishing procedures such as mechanical polishing or applying sealant agent coupling.⁸

Color stability throughout the functional lifetime of a restoration is as crucial as mechanical and physical properties. Discoloration of hybrid ceramic restoration can be caused by extrinsic or intrinsic factors. Extrinsic discoloration can be occurred because of staining beverages, foods and smoking habits.^{9,10} Intrinsic discolorations are related to mode and duration of polymerization, initiator system, the resin matrix composition, conversion of the matrix monomers, particle size, and oxidation of the unreacted carbon double bonds.^{11,12} Discolorations affect negatively the quality and survival rate of restorations during clinical usage¹³, thus color stability of restoration materials must be ensured. It was reported that both color stability of dental restorative materials can be provided due to well-finished surfaces, and technical and aesthetical problems of restorations can be decreased.^{14,15}

Manufacturers recommend different surface finishing procedures such as polishing combined with dif-

ferent sets and applying sealant agent coupling for hybrid ceramic. But it is uncertain whether applying sealant agent coupling material after sandblasting/etching or polishing with different sets provide more appropriate color stability and surface roughness. This study aimed to evaluate the effects of four different surface finishing methods and aging on the surface roughness and color stability of hybrid-ceramic. The first null hypothesis was that the type of surface finishing method and artificial aging would not affect surface roughness of hybrid ceramic material. The second null hypothesis was that type of surface finishing method and artificial aging would not affect color stability of hybrid ceramic material.

MATERIALS AND METHOD

The materials used in this study are presented Table 1. Hybrid ceramic CAD/CAM blocks (Vita Enamic, VITA Zahnfabrik, Bad Säckingen, Germany), shade 2M2-HT, were prepared by using low-speed precision cutting device (Micracut 125 Low Speed Precision Cutter; Metkon) at 12x14x1 mm size and all surfaces were smoothed with 400-, 600-, 1200-grit silicon carbide paper respectively. The specimens were divided into four groups according to different surface polishing and finishing protocols (n=10).

Group I (ET): Specimens were polished with technical polishing set (Vita Enamic Polishing Set Technical, VITA Zahnfabrik, Bad Säckingen, Germany) that were used pink pre-polishing instrument with 7000 rpm hand piece and grey high-gloss polishing instrument with 5000 rpm hand piece respectively.

Group II (EC): Specimens were polished with clinical polishing set (Vita Enamic Polishing Set Clinical, VITA Zahnfabrik, Bad Säckingen, Germany) that were used pink pre-polishing instrument with a speed of 7000 min⁻¹ and grey high-gloss polishing instrument with a speed of 5000 min⁻¹ respectively.

Group III (ES): Specimens were subjected to sandblasting with 50 µm Al₂O₃ particles at a 1 bar pressure.

Group IV (EA): Specimens were etched with 5% hydrofluoric acid gel (IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein) for 60 seconds. The specimens were rinsed with distilled water for 2 minutes and air-dried.

Table 1. Manufacturers and compositions of the materials used in this study

Material	Composition	Lot Number	Manufacturer
Vita Enamic Hybrid ceramic	Polymer-infiltrated ceramic network material (UDMA, TEGDMA) with 86 wt% ceramic	67300	Vita Zahnfabrik, Bad Säckingen, Germany
Vita Enamic Polishing Set Technical	Silicon carbide, diamond	E54470	Vita Zahnfabrik, Bad Säckingen, Germany
Vita Enamic Polishing Set Clinical	Silicon carbide, diamond	E65220	Vita Zahnfabrik, Bad Säckingen, Germany
IPS Ceramic Etching Gel	5% hydrofluoric acid	W99196	Ivoclar Vivadent, Schaan, Liechtenstein
Vitasil	3-trimethoxysilylpropyl methacrylate, ethanol	45040	Vita Zahnfabrik, Bad Säckingen, Germany
Vita Enamic Glaze	Methyl methacrylate, 2-propenoic acid, diphenylphosphinoxide	73680	Vita Zahnfabrik, Bad Säckingen, Germany

Table 2. The mean and standard deviations of surface roughness values of groups

		Before aging Mean±SD	After aging Mean±SD	^c P
ET		0.23 ± 0.05	0.19 ± 0.04	0.092
EC		0.27 ± 0.07	0.27 ± 0.06	0.386
EA		0.06 ± 0.03	0.05 ± 0.02	0.575
ES		0.13 ± 0.05	0.2 ± 0.08	0.005***
	^a P	0.001**	0.001**	
Pairwise Comparisons; ^b P	ET-EC	1	0.415	
	ET-EA	0.001*	0.007*	
	ET-ES	0.109	1	
	EC-EA	0.001*	0.001*	
	EC-ES	0.007*	0.335	
	EA-ES	0.649	0.010*	
^a Kruskal Wallis Test	^b Bonferroni Dunn Test	^c Wilcoxon Signed Ranks Test		
** P < 0.01	* P < 0.05	*** P < 0.05		

All of the specimens were placed in distilled water in ultrasonic bath for 5 minutes.

For the sealant agent coupling groups (ES, EA), the silane (Vitasil, Vita Zahnfabrik, Bad Säckingen, Germany) was applied to the roughened surface before applying sealant agent coupling procedure. A small drop of sealant agent coupling solution (Vita Enamic Glaze, VITA Zahnfabrik, Bad Säckingen, Germany) was placed on the ceramic with disposable applicator to create a thin and even layer. Light Emitting Diodes (LED) polymerization unit (Valo Cordless, Ultradent Products Inc, South Jordan, UT, USA) was used for 30 seconds with 395-480 nm wavelength for the polymerization of sealant agent coupling solution.

Color parameters L* (brightness), a* (red-green axis) and b* (yellow-blue axis) were measured three times with CIELab color system using a clinical spectrophotometer (Vita Easyshade Advance, Vita Zahnfabrik, Bad Säckingen, Germany) on grey background and surface roughness values were recorded by taking the average of six measurements with profilometer (Perthometer M2, Mahr Federal, Germany) of each specimen. Thermocycling procedure was applied for 5000 cycles from 5°C to 50°C temperatures with 60 seconds waiting time (SD Mechatronic Thermocycler, Westerham, Germany). After artificial aging, color and surface roughness values were re-measured. ΔE values were calculated according to the formula: $\Delta E^* = [(L^*_1 - L^*_0)^2 + (a^*_1 - a^*_0)^2 + (b^*_1 - b^*_0)^2]^{1/2}$

Surface analysis were performed with Scanning Electron Microscope (SEM) to 2 specimens of each group as before and after thermocycling procedure.

Statistical Analysis

The mean ΔE and surface roughness values were calculated with SPSS statistical software package program (SPSS version 24.0 software, SPSS; Chicago, IL, USA). The results were analyzed by using Kruskal Wallis test to study the difference among different surface finishing protocols both before and after aging procedure. Bonferroni Dunn Test was used for pairwise comparisons. Wilcoxon signed ranks test was performed to determine the effect of aging procedure on surface roughness of different surface treatments. Color differences of groups were evaluated by using Kruskal Wallis and Bonferroni Dunn tests. Level of significance was set at $p < 0.05$.

RESULTS

Surface Roughness

The means and standard deviations of groups for before and after aging were shown at Table 2 and Fig 1. Before aging procedure, it was observed that there was a significant difference between groups ($p = 0.001$, $p < 0.01$). EC group showed higher surface roughness values than EA group and ES group. Also surface roughness values of ET group was higher than EA group ($p = 0.001$; $p < 0.01$). No statistically significant difference was determined between other groups ($p > 0.05$).

After aging procedure, a significant difference was found between groups ($p = 0.001$; $p < 0.01$). EA group has lower surface roughness values than ET, EC and ES groups. There was no significant difference between other groups ($p > 0.05$).

The analysis of results, which was compared surface roughness of different surface treatments values of before and after aging procedure, was revealed that after aging surface roughness value of ES was significantly higher than before aging surface roughness value ($p < 0.05$). For other groups (ET, EC, EA), there was no significant difference between before and after aging values ($p > 0.05$).

Color Difference

ΔE values of different surface treatments of dental

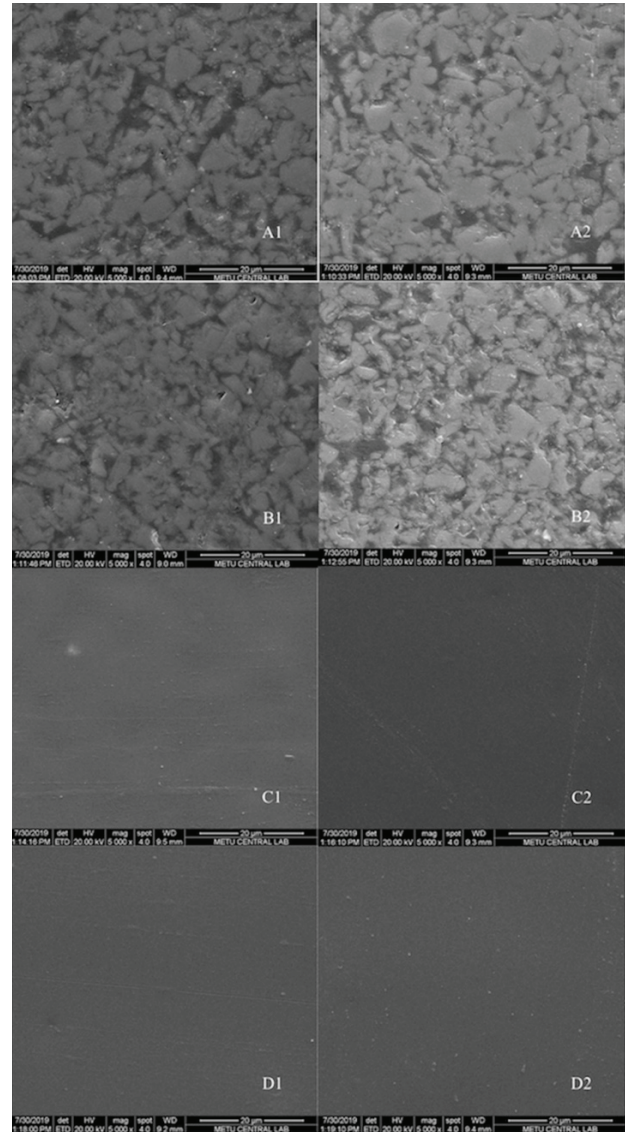


Figure 1. Scanning electron microscope images (x5000 magnification) of groups before and after aging respectively. A1-A2, polishing with technical kit; B1-B2, polishing with clinical kit; C1-C2, applying sealant agent after etching; D1-D2, applying sealant agent after sandblasting.

hybrid ceramic material were presented in Table 3 and Fig 2. There was no statistically significant difference between groups ($p > 0.05$) and ΔE values of each group were lower than clinically acceptable threshold ($\Delta E < 2.7$).¹⁶ Nevertheless, the highest color differences were observed at specimens of ES group (1.23 ± 0.39) and the lowest color differences were determined at specimens of EA group (0.98 ± 0.4).

Scanning Electron Microscope Examination

SEM images revealed that both of the applied sealant agent surfaces were smoother than the polished

surfaces before aging. After aging, the porous surface was seen on the specimen of applied sealant agent coupling on sandblasting group. Scratches were detected on the surfaces of the technical polishing and clinical polishing groups (Figure 1).

Table 3. Mean and standard deviation of ΔE values of groups

Group	Mean \pm SD	^a P
ET	1.17 \pm 0.64	0.701
EC	1.11 \pm 0.47	
EA	0.98 \pm 0.4	
ES	1.23 \pm 0.39	

^aOne way ANOVA

DISCUSSION

In this *in vitro* study, significantly different surface roughness values were found between four different surface finishing methods before and after aging procedure. These results showed that the first null hypothesis, that the type of surface finishing method and artificial aging would not affect surface roughness of hybrid ceramic material, was rejected. The color differences of the materials were clinically acceptable threshold and there was no significant difference between surface finishing methods of hybrid ceramic. Therefore, the second null hypothesis was confirmed.

Hybrid ceramic materials were developed as alternatives to the ceramic materials to create tooth-like esthetic restorations. One of the most important advantages of hybrid ceramic materials is that the restorations can be fabricated in a single milling stage without heat treatment.¹⁷ However, the milling process is increased the roughness of CAD/CAM restorative materials.⁸ Rougher surfaces of dental restorations may be caused wear of the opposing teeth, retention of microbial biofilm, inflammation of the periodontal tissue and discoloration of the restoration.^{6,7} Mota et al.⁸ evaluated the surface roughness of CAD/CAM ceramic (Mark II, Suprinity LS, IPS Empress CAD, IPS e.max CAD) and hybrid ceramic (Vita Enamic, Lava Ultimate) materials after milling and after surface finishing procedures. It

was concluded that the surface roughness values of the tested materials were increased after milling and the polishing procedure must be applied to reduce roughness. In another study, surface roughness of CAD/CAM restorative materials and direct composite resins was compared after 3 different surface treatments (polishing, roughing with SiC P1200 and roughing with SiC P500). It was reported that the polishing procedure was decreased the surface roughness values.¹⁸

Hybrid ceramic restorations can be completed with manual polishing techniques or applying light-polymerized sealant agent coupling material.¹⁷ The manufacturer of Vita Enamic recommends four different surface finishing methods such as polishing with technical kit, polishing with clinical kit, applying sealant agent after 5% hydrofluoric acid application or applying sealant agent after sandblasting with 50 μm Al_2O_3 particles. In the present study, the all of the surface finishing procedures of Vita Enamic were performed.

Özarslan et al.¹⁹ performed polishing (technical kit, clinical kit) and applying sealant agent procedures on Vita Enamic translucent and high translucent specimens to determine the effect of surface finishing methods on surface roughness and color differences. Unlike present study, before applying sealant agent procedure, any surface treatment was not applied on specimens. It was observed that the lowest surface roughness and color difference values were in technical kit groups and the highest surface roughness values were observed in sealant agent groups. In the present study, before aging the lowest surface roughness values were determined on applying sealant agent after acid application group and applying sealant agent after sandblasting group. However, the surface roughness values of applying sealant agent after sandblasting group were increased dramatically after aging. It might be resulted that the ultrasonic bath with 5 min period were not cleaned the sandblast particles on surfaces of specimens. Hence, the bonding between hybrid ceramic material and sealant agent coupling liquid was weak. Also, applying sealant agent after etching group were showed lower surface roughness values than 0.2 μm , which is critical threshold for microbial retention²⁰, both before and after aging.

SEM analysis was performed to compare the differences on surfaces of Vita Enamic specimens, which were finished different surface finishing methods. SEM images showed that the applied sealant agent surfaces were smoother than the polished surfaces. However, the porous surface was detected on specimen of applied sealant agent on sandblasting group. Besides, scratches were seen on the surfaces of the technical polishing and clinical polishing groups. As a result, SEM images corroborated the surface roughness values.

Color stability of a restoration is important parameter in terms of the esthetic outcome and clinical usage. As well as surface roughness, the composition and microstructure¹ of dental restorative materials affect the optical properties.²¹ Vita Enamic may be susceptible to discoloration due to high water absorption of hydrophilic triethylene glycol dimethacrylate (TEGDMA), which is a component of polymer network.²² Saba et al. compared color stability of hybrid ceramic (Vita Enamic) and feldspatic ceramic (Vitablocks Mark II) after 28-days of immersion. It was stated that hybrid ceramic showed significantly higher color change values than feldspatic ceramic in distilled water because of resin matrix.

Several studies reported that sealant agent coupling was more effective than polishing techniques in terms of color stability and surface properties.²⁴⁻²⁶ However, hybrid ceramic materials are polished without furnace and sealant agent coupling liquid is polymerized by LED curing device. There was no consensus about the quality of surface finishing procedures of Vita Enamic hybrid ceramic material in the literature. Sagsoz et al.⁷ reported that the color change values of polished specimens were lower than applied sealant agent specimens for Vita Enamic material. On the contrary, Kilinc et al.²⁴ compared the color differences of CAD/CAM restorative materials (Lava Ultimate, Cerasmart, Vita Enamic, Vita Suprinity, Vita Mark II) after different surface finishing procedures and UV artificial aging, which was equivalent 1 year of clinical use. They found that applying sealant agent procedure could be recommended for Vita Enamic in terms of color stability but the color difference was higher than clinically unacceptable threshold ($\Delta E=4.64\pm 1.56$). In this study, thermocycling procedure was performed for 5000 cycles, which is imitation of oral conditions for

6 months²⁷ and it was observed that there was no significant difference between color change values of different surface finishing procedures. And also, the color change values of each group were under the clinically unacceptable threshold ($\Delta E < 2.7$).¹⁶

There are some limitations of this *in vitro* study. The effect of surface finishing methods on other esthetic parameters such as translucency and gloss were not considered. Thermocycling procedure was applied to imitate the oral conditions but chewing forces can be affected the surface of hybrid ceramic material for long time period. Consequently, further *in vivo* and *in vitro* studies are necessary to determine the optimum surface finishing method for Vita Enamic hybrid ceramic material.

CONCLUSION

Within the limitation of this *in vitro* study, the lowest surface roughness values were determined at applied sealant agent after etching group before and after aging. The surface roughness values of applied sealant agent after sandblasting group were increased after aging. After artificial aging, there was no significant difference between color difference values of surface finishing procedures. The most effective surface finishing procedure was applying sealant agent after hydrofluoric acid gel application in terms of surface roughness and color change.

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