Nobel Med 2022; 18(3): 194-200

EFFECT OF SURFACE FINISHING METHODS, pH AND TEMPERATURE ON ROUGHNESS AND ION RELEASE OF CERAMICS

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ABSTRACT

Objective: *There is a lack of information on dental ceramics long-term ion releases at different conditions and effect of surface finishing techniques on ion leaching. The difference between the amount of ions released from a polished ceramic surface and that released from glazed surfaces is unknown. This study aimed to investigate surface changes and ion leaching from dental ceramics after corrosion and to assess differences between two surface finishing methods.*

Material and Method: *20 ceramic disks (8×2 mm) (Empress II, Zirkonzahn, Noritake Cerabien ZR, Finesse, Vita VM7) were fabricated and categorized into autoglazed and polishing (n=10). The surface roughness (Ra) was evaluated before and after corrosion. After subjecting the specimens to in vitro corrosion (pH 4.5–pH 7–pH 9 in water at 37°C±2°C for 18 hours and at pH 2.4 in 4% acetic acid solution at 80°C±2°C for 18 hours), ion release was assessed. Data were analyzed using ANOVA and Bonferroni Test (α=0.05). Multiple comparisons were analyzed by Student t-test.*

Results: *Al, Na, Li, Si ions were leached from all ceramic groups. Ra was significantly changed between the groups at various pH ($p<0.05$). Post corrosion Ra values were lower than pre corrosion for polished groups.*

Conclusion: *Glazed surfaces are more resistant to corrosion for high sintering ceramics. Polishing can also be a reliable technique when restorations removal is avoided.*

Keywords: *Ceramics, corrosion, ion release, mass spectrophotometry, surface roughness.*

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DELIVERING DATE: 16 / 03 / 2021 • **ACCEPTED DATE:** 15 / 02 / 2022

YÜZEY BİTİRME YÖNTEMLERİ, pH VE SICAKLIĞIN SERAMİKLERİN İYON SALINIMI VE PÜRÜZLÜLÜĞÜNE ETKİSİ

ÖZET

Amaç: Dental seramiklerin farklı koşullar altında uzun süreli iyon salınımları ve yüzey bitirme tekniklerinin iyon salınımı üzerindeki etkisi hakkında literatürde yeterli bilgi bulunmamaktadır. Polisajlanmış bir seramik yüzeyden salınan iyon miktarı ile glazürlü yüzeylerden salınan iyon miktarı arasındaki fark bilinmemektedir. Bu çalışmada, dental seramiklerin korozyon sonrası yüzey pürüzlülüğü ve iyon salınımının miktarı ile iki yüzey bitirme yöntemi arasındaki farkların değerlendirilmesi amaçlanmıştır.

Materyal ve Metot: 20 adet seramik disk (8x2 mm) (Empress II, Zirkonzahn, Noritake Cerabien ZR, Finesse, Vita VM7) üretilerek otoglaze ve polisajlı olmak üzere 2 gruba ayrıldı (n=10). Yüzey pürüzlülüğü (Ra) korozyondan önce ve sonra ölçüldü. Örnekler in vitro korozyona (pH 4,5–pH 7–pH 9 suda 37°C±2°C'de 18 saat ve pH 2,4'te %4 asetik asit solüsyonunda 80°C±2°C'de 18 saat) tabi tutulduktan sonra iyon salınımı değerlendirildi. Elde edilen veriler ANOVA ve Bonferroni Testi (α=0,05) kullanılarak analiz edildi. Student t-testi ile çoklu karşılaştırmalar yapıldı.

Bulgular: Tüm seramik gruplarından Al, Na, Li, Si iyonlarının salındığı saptanmıştır. Ra ölçümlerinde, farklı pH değerlerinde gruplar arasındaki değişimler istatistiksel olarak anlamlıdır (p<0,05). Korozyon sonrası Ra değerleri poliajlı gruplar için korozyon öncesi değerlerden daha düşük olduğu görülmüştür.

Sonuç: Yüksek ısı seramiklerinde glazürlü yüzeyler, korozyona karşı daha dayanıklıdır. Ancak yine de polisaj işlemi, glazür uygulanabilmesi mümkün olmayan restorasyonların çıkarılmasının kaçınıldığı durumlarda güvenilir bir teknik olarak görülebilir.

Anahtar kelimeler: Seramikler, korozyon, iyon salınımı, kütle spektrofotometresi.

INTRODUCTION

Dental ceramic materials are considered the most chemically inert reconstructive materials and the chemical durability of ceramic materials is inherently reliable but is suggested to be influenced by several factors such as the composition and microstructure of the ceramic material, surface finishing methods, chemical characteristics of the corrosive medium, exposure time, and temperature.[1] Corrosion occurs via the diffusion of water molecules into the glass, and these molecules react with nonbridging oxygen atoms to produce negatively charged hydroxyl ions that migrate out with the positively charged alkali ions to maintain electrical neutrality. Extensive studies have shown that the corrosion of glass can be categorized into different levels depending on its composition and the chemical composition of the corrosive medium.[1,2] However, differences in composition, microstructure, and local corrosion conditions further complicates the corrosion process, possibly resulting in partial breakdown of the surface silicate structure.[1,3] The composition of ceramics strongly affects the surface dissolution. Glasses with high K_2O content have less chemical durability than those made using soda (Na_2O) as an added flux material.[4] Moreover, zirconia and alumina improve the chemical durability of glasses.[5,6] Following hydrolysis, ultra-low temperature sintering ceramics exhibit higher solubility than conventional high-temperature sintering ceramics.[7] However, repeated hydrolysis tests revealed inconsistency in the predicted reaction manner of high-and low-sintering ceramic materials. Following exposure to a corrosive environment, alumina, a very stable material, may also undergo compositional changes.[6]

Owing to the variations in microstructure and composition between dental ceramics, these interceramic differences have been suggested to influence ion leaching following the exposure of ceramics to aqueous media. Although dental ceramics are known as inert materials, there is not much information on their long-term ion releases at different conditions and effect of surface finishing techniques on ion leaching. There is a lack of information about polished surfaces long term ion releases and surface changes especially for restorations that require chairside adjustments leaving a polished surface rather than a glazed surface. In the present investigation, in vitro ion leaching and surface roughness (Ra) of different types of dental ceramics following exposure to high and low-intensity corrosion were assessed, and the results of surface finishing methods in the corrosive environments were compared. The first null hypothesis of this study is that surface finishing methods and pH changes does not affect the ion release and surface roughness of ceramics. The second null hypothesis is surface finishing methods and pH changes affect the ion release and surface roughness of ceramics in corrosive envorinment.

MATERIAL AND METHOD

For this study, 20 ceramic disks (8 mm in diameter and 2 mm in height) were fabricated from Zirkonzahn (ICE Zirkon, Zirkonzahn, Bruneck, Italy), Noritake Cerabien

Table. Firing parameters for ceramic samples.

Ceramics	**B (°C)**	**t↑**	**T**	**S**	**H**	**V_1**	**V_2**
Empress II	403	60	840	4	2	450	839
Vita VM7	500	55	910	6	1	600	910
Zirkonzahn	300	55	820	6	1	400	820
Noritake CZR	500	55	930	5	1	600	930
Finesse	450	35	760	5	0.5	450	760

B: Stand by temperature (OC), **T:** Firing temperature, **t↑:** Temperature incresase OC/minute, **S:** Closing time/minute; **H:** Holding time at last temperature (minutes), **V1:** Vacuum-on temperature (OC), **V2:** Vacuum-off temperature (OC).

ZR (Noritake Dental Supply, Co., Nagoya, Japan), Empress II (Ivoclar Vivadent, Schaan, Liechtenstein), Vita VM7 (Vita Zahnfabrik, Bad Säckingen, Germany) and Finesse (Dentsply Ceramco, New York, US) veneering ceramics. These various ceramics were selected to represent different sintering temperatures and ceramic types (Table). The ceramic specimens were fabricated in a plexiglass mold according to the procedure described in a previous study.[6] The ceramic powder was mixed with a spatula on a glass plate by using the mixing liquid recommended by the manufacturers of each ceramic material. The cylindrical mold was filled carefully with the mixture and condensed. Using a piece of adsorbent paper, excess liquid was removed from the specimen surface. After condensation, the mold was removed, leaving the non-sintered test specimen on the firing tray. All specimens were sintered in a calibrated oven as per the manufacturer's protocol by the same experienced dental technician. All test surfaces were ground by using 1000 grit SiC paper on a rotating disc (Phoenix Beta, Buehler, USA) at 150 revolutions/minute. Finally, the test specimens were placed in distilled water and were ultrasonically cleaned. These were then divided into two groups of auto glaze and polishing (n=10). Next, the auto glaze was applied according to the manufacturer's instructions for each material. Mechanical polishing was applied with Shofu All Ceramic Finishing and Polishing wheels (Shofu Dental Corporation, US) by using a slow-speed handpiece rotating at approximately 10,000 rpm as recommended by the manufacturer.

The specimens were then ultrasonically cleaned with distilled water and dried with a blast of air for 30 seconds before surface analysis. The surface roughness was evaluated for all specimens using a profilometer (SJ-201 Mitutoyo Corp., Kawasaki, Japan). To measure the roughness profile value (µm), the diamond stylus was moved across the surface under a constant load of 3.9 mN and a speed of 0.100 mm/s during testing. This procedure was repeated ten times at a different location for each specimen to obtain the general surface characteristics of the specimens. The Ra values were considered to be the average values of these measurements.

After the initial surface roughness analysis, the ceramic specimens were subjected to different levels of corrosion intensity. In the water corrosion experiments, the specimens were placed in the shaking equipment (New Brunswick, Innova, 4080 Incubator Shaker, Herisau, Switzerland) and exposed to gentle rocking for 18 hours at 37°C in solutions with pH values of 4.5–7 and 9. In the acid corrosion experiments, we modified the conditions of ISO Standard 6872.[8] According to the standard, all specimens were momentarily exposed to 4% acetic acid at 80°C, and corrosion solution temperature was gradually increased to 80°C for reducing the risk of surface microcrack formation. A single ceramic specimen and 17 mL of corrosive medium were added to each of the polyethylene corrosion bottles, and the bottles were sealed with a screw cap. The bottles were then placed in an oven, and the temperature was increased until reaching the final temperatures of 37°C and 80°C. To avoid a steady-state situation at the surface of the test specimens, we subjected them to gentle rocking during the entire corrosion process in the oven.[6] After cooling to room temperature, the specimens were removed from the bottles, rinsed in distilled water, dried, and a new surface analysis was performed. Solutions were transferred to inductively coupled plasma mass spectroscopy for element analysis leached from the ceramics. To preserve the solutions during storage, we reduced the adsorption of metal ions to the surfaces of the polyethylene bottles by adding a small quantity of nitric acid to the solutions after corrosion. Calibration was performed by using matrix-matched standard solutions prepared from (MERC) stock standard solutions.

The mean blank values corrected the individual analytical values in parts per billion (ppb). We used an Inductively Coupled Plasma Mass Spectrophotometer (Agilent 7500ce Octopole Reaction System, Agilent Technologies, Japan) at low concentrations to obtain the required detection limits for all elements. Si, Al, Na, and Li were evaluated as those were the common elements for the types of ceramics used in this study.

All chemical experiments and measurements obtained in this study were performed at Izmir Institute of Technology Department of Chemical Engineering.

Analysis of variance was used to analyze the data statistically. The descriptive statistics were used as an overview and to determine if there were any patterns on a group level. Multiple comparisons (type of ceramic, polishing/auto glaze, and pH) were conducted separately using Tukey HSD test between the glazed and polished groups for each element. Ra comparisons were evaluated using a Student t-test. Because the ion leaching values were very low, logarithms of the

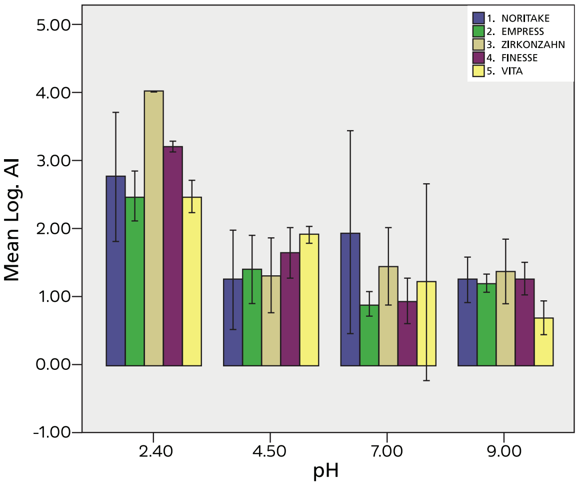

Figure 1A. Al leaching amounts of glazed ceramics in 10^4 ppb, $p<0.05$.

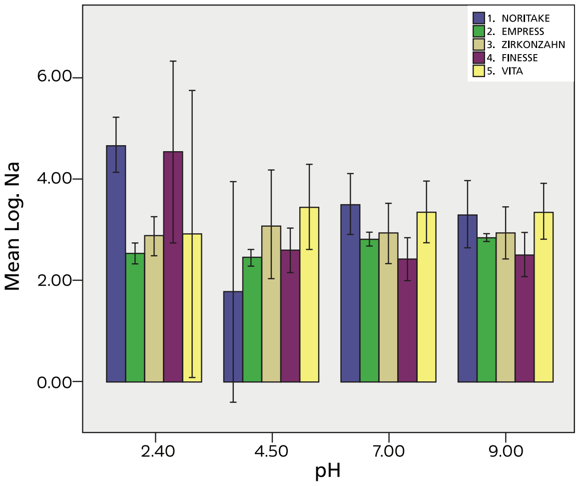

Figure 2A. Na leaching amounts of glazed ceramics in 10^4 ppb, $p<0.05$.

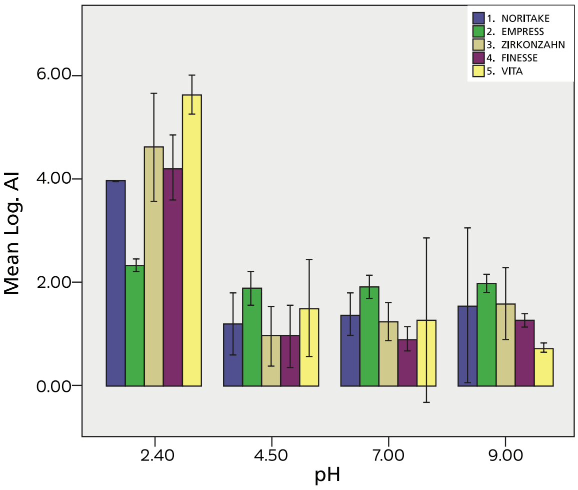

Figure 1B. Al leaching amounts of polished ceramics in 10^4 ppb, $p<0.05$.

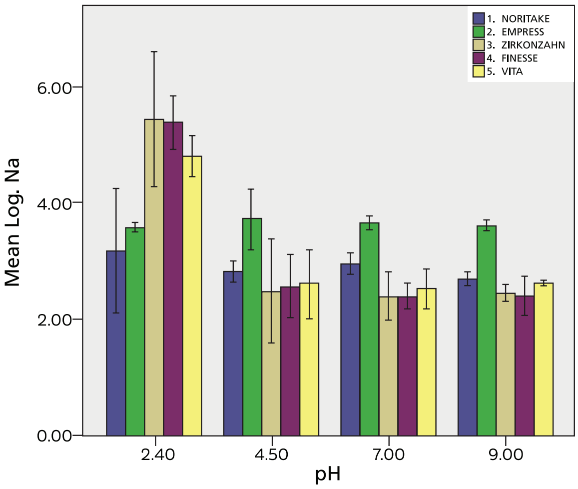

Figure 2B. Na leaching amounts of glazed ceramics in 10^4 ppb, $p<0.05$.

numerical values were taken for comparative tests to show on the graphics, that is why some data were shown on negative y axis.

RESULTS

Ceramic samples had the highest elution values for aluminum although they were present at much smaller amounts compared to Si ions. Polished samples of Zirkonzahn, Finesse, and Vita VM7 leached more Al ions than glazed ones (Figure 1A, Figure 1B) at 80°C at pH 2.4 compared to the water corrosion tests performed at 37°C ($p<0.05$). Na ion elution is shown in Figure 2A, Figure 2B. Polished samples had higher values for all ceramic groups and were more prominent at pH 2.4 (Figure 2B). A slight difference was calculated for Li leaching amounts (Figure 3A) for glazed samples at pH 2.4. The highest Li ion leaching values (Figure 3B) were obtained at pH 2.4 in the polished groups. Glazed samples showed the lowest Si leaching amounts at pH 7 (Figure 4A). The highest Si ion release was obtained at pH 2.4 particularly in the polished samples of all groups (Figure 4B).

Post-corrosion Ra values (Figure 5A) of glazed groups of Noritake, Zirkonzahn and Vita VM7 were lower than the pre-corrosion values. We obtained lower post-corrosion values from all the polished samples except for Noritake, especially at a pH of 2.4 (Figure 5B). Both the polished and glazed Vita and Zirkonzahn groups showed smoother surfaces after the corrosion tests.

The pre-corrosion SEM image of polished Finesse exhibits a dense surface, with very few open porosities. After corrosion the surface is covered in numerous microporosities, in between seemingly intact areas (Figure 6A, Figure 6B).

DISCUSSION

Ion leaching results and surface roughness measurements revealed that surface finishing methods affects the surface roughness and amount of ions that released from ceramics. Therefore, the first null hypothesis of the study is rejected. Decreased surface roughness values calculated from polished surfaces after corrosion at low pH values may be a sign of dissolution of ceramics. The second null hypothesis is accepted

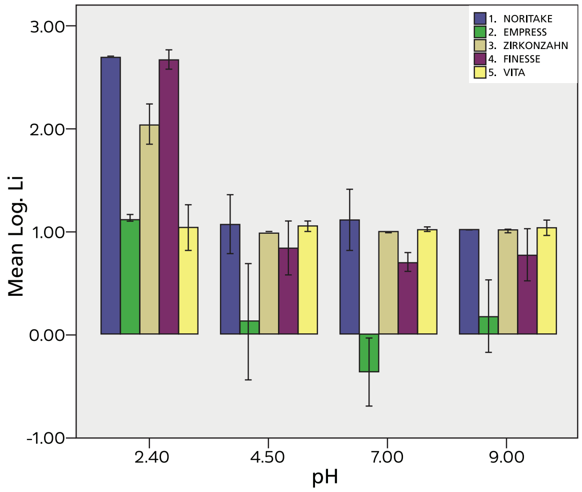

Figure 3A. Li leaching amounts of glazed ceramics in 10^4 ppb, $p<0.05$.

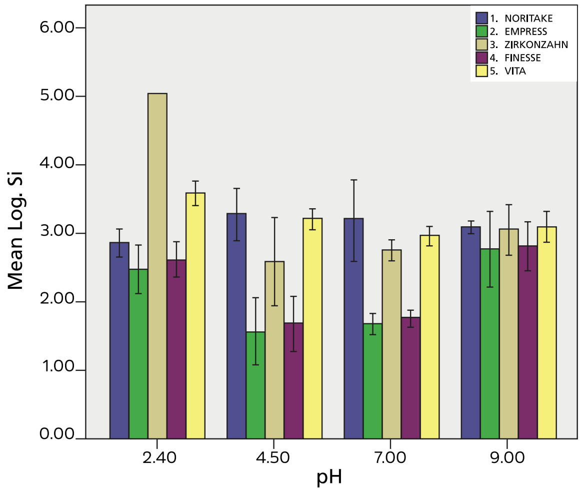

Figure 4A. Si leaching amounts of glazed ceramics in 10^4 ppb, $p<0.05$.

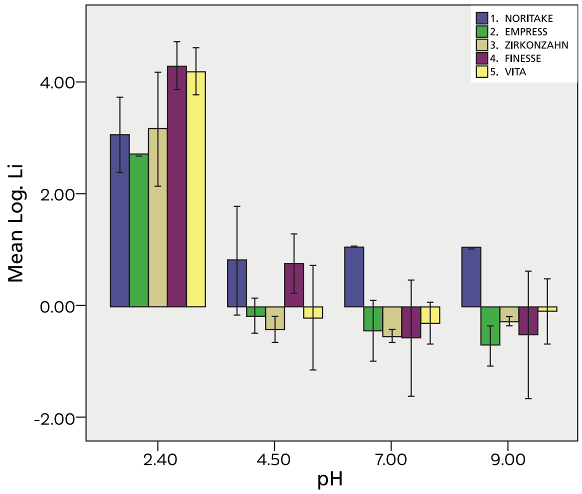

Figure 3B. Li leaching amounts of polished ceramics in 10^4 ppb $p<0.05$.

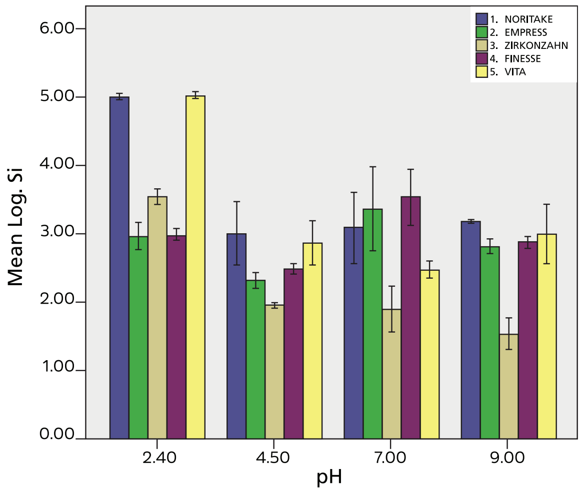

Figure 4B. Si leaching amounts of polished ceramics in 10^4 ppb, $p<0.05$.

since both pH and surface finishing methods affected the ion leaching amounts and surface roughness.

In this investigation, the steps were followed disclosed in ISO Standard 6872.[8] The amount of weight loss due to corrosion was not measured, the number of released elements was assessed following immersion in water at different pH values. Measurement of specific ion release can more precisely determine the process of dental ceramic degradation compared to the measurement of the total weight loss of specimens as mentioned by Marco Jakovac *et al*.[9] We observed significant changes in the leaching pattern for the tested ceramics, ranging from very low to high values of ion release. As might be expected, differences in dissolution were observed between the corrosive solutions. With acid corrosion, significantly higher values for all the examined elements were found. This situation may be explained by differences in the thermal energy, which was too low at 37°C to overcome the activation barrier of a number of elements. As in dental ceramics, compositional heterogeneity will probably create local differences in multicomponent glasses and ceramics.[6,10]

In our study, two surface finishing methods were tested against corrosion resistance. Polishing created as smooth surfaces as with the autoglazed surfaces, but the polished surfaces, not all but to a large extent, appeared to be more prone to corrosion, especially at low pH and high temperature. The pH value of acetic acid (2.4) does not represent a clinically unrealistic value because low pH values have been recorded in subplaque areas and in common beverages such as carbonated soft drinks (pH 2.5), citrus juice (pH 3.3), and coffee (pH 4.8).[11] When changing to more acidic conditions and higher temperatures, the corrosion process was found to influence the surface properties of glass-phase (Empress) and leucite reinforced veneering ceramics (Finesse) to a greater extent. Technically, the aqueous corrosion of dental ceramics has been associated with crack propagation and reduced strength. Surface finishing methods sufficiency to create a resistant outer shield against acidic envorinment is of great importance for long term success. Glazed surfaces seem to be more resistant to corrosion especially for high sintering ceramics (Vita VM7 and Noritake Cerabien ZR). Polishing can also be accepted as a reliable

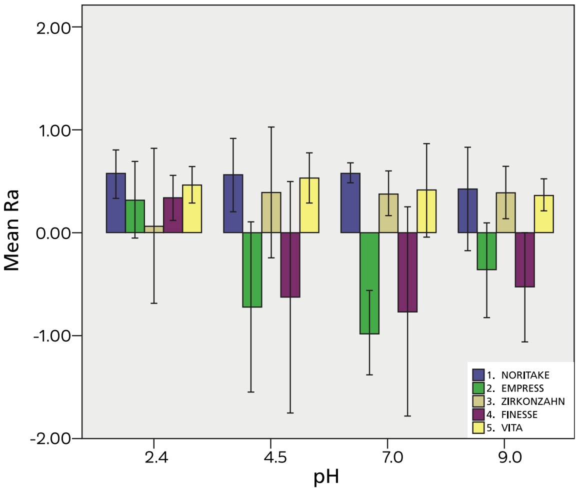

Figure 5A. Ra changes (Ra initial–Ra final) of glazed surfaces in µm, $p<0.05$.

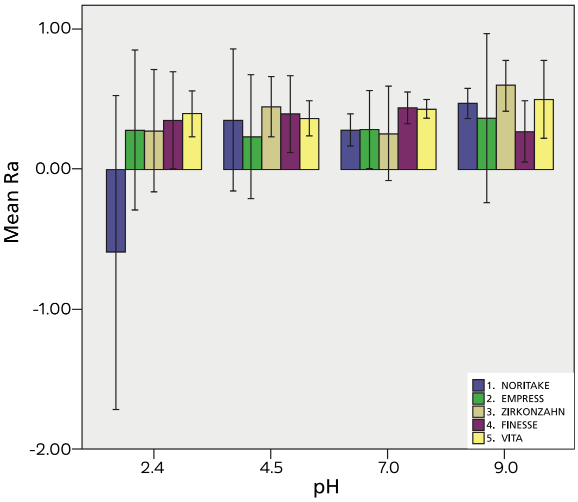

Figure 5B. Ra changes (Ra initial–Ra final) of polished surfaces in µm, $p<0.05$.

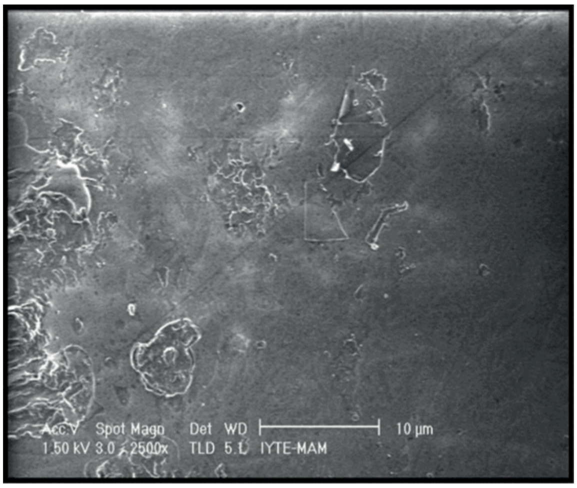

Figure 6A. SEM image of glazed Empress before corrosion.

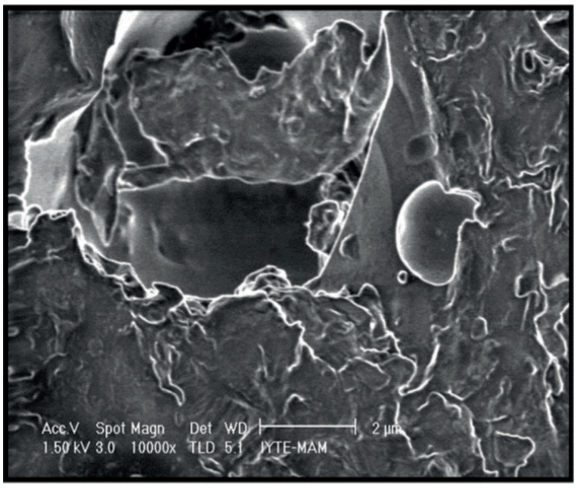

Figure 6B. SEM image of glazed Empress after corrosion at pH 4.5.

technique when restorations removal must be avoided after permanent cementation for minor corrections or in case of chipping etc.

Some investigators recommend glazed rather than polished surfaces to obtain smoother surfaces. But inlay, onlay, and veneer restorations require chairside adjustments and finishing using polishing wheels. Polished and glazed surfaces showed approximate Ra values in the present study. Polished specimens present smoother surfaces than glazed ones after corrosion tests. This may be a sign of higher ion leaching values of polished surfaces and glazed ones may thought to be more stable in the corrosive medium but leaching amounts of the ions are at low levels. Besides the surface roughness, under more severe conditions, the Si–O–Si bonds may be broken and degradation processes will also occur. Significant changes in the leaching pattern were observed for the tested ceramics, ranging from very low to high values of ion release. Primarily the alkali ions were leached, which is in accordance with earlier findings.[1,3] However, in some of the ceramics (Noritake, Zirkonzahn, and Vita VM7), aluminum, sodium and silicon also leached in higher concentrations than expected. Although the amounts of the leaching elements vary according to the surface finishing technique and the type of ceramic, polished surfaces leached significantly more ions than glazed surfaces. However, this does not mean that a direct correlation exists between post-corrosion surface appearance and degree of surface structural changes.

No toxic effects or allergic reactions are associated with any of the major elements in bioceramics.[12,13] But our knowledge about the existence of these substances at low amounts is limited. Granuloma formations are reported related to tissue accumulation of zirconia and alumina particles in animal experiments.[14,15] Similar observations have also been made in humans in association with zirconia ceramics and glass phase ceramics for oral restorations.[16,17] Apart from ion dissolution, the mechanical wear of ceramic materials resulting in microfine particles may rise to local and possibly systemic effects by accumulating in target tissues and organs.[18,19] The corrosion process may facilitate the mechanical degradation and thereby increase the release of inorganic ions and particles. Because of their resistance to degradation, a concentration gradient of particles can be built up within the tissues, which may constitute a potential problem. Nonbioactive ceramic materials are permanent, and biodegradation proceeds very slowly under normal conditions. However, a different situation develops under more aggressive environmental conditions, such as parafunctional activity, frequent contact with chelating agents, more acidic or alkaline pH, higher temperature levels, and acidulated fluoride gels, especially in combination with heavy loading. The method used for the in vitro corrosion may, therefore, be considered a simplified model for the study of basic corrosion reactions in comparison with in vivo conditions. However, the aqueous corrosive conditions seem to be the most crucial factor in ceramic and metal corrosion. Under such conditions, the bioinert concept associated with bioceramics may require re-evaluation, and a relative result may be considered.[20]

CONCLUSION

The findings of this study revealed the following:

1. The surface changes induced by the corrosion process were influenced by the pH and temperature changes at different degrees according to ceramic type.
2. Polished specimens present smoother surfaces than glazed ones after corrosion tests.
3. Polishing can also be accepted as a reliable technique for surface finishing of ceramics.
4. Glazed surfaces seem to be more resistant to corrosion especially for high sintering ceramics.

*The authors declare that there are no conflicts of interest.

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