

## Effect of Surface Finishing Methods and Corrosion on Surface Elemental Composition and Roughness of Dental Ceramics

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### ABSTRACT

**Aims:** The aim of this study was to compare the effect of surface finishing methods and chemical corrosion on surface elemental composition and roughness of ceramic materials.

**Material and Methods:** 20 ceramic disks were fabricated from A1 shade of Empress, Zirkozahn, Noritake Cerabien ZR, Finesse, Vita VM7 and divided into 2 groups of autoglaze and polishing (n=10). The surface roughness (Ra) and surface elemental analyses were evaluated before and after 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 at 80°C ± 2°C for 18 hours). The data were statistically analyzed by Univariate Analysis of Variance and Bonferroni Test. Multiple comparisons of Ra values between autoglaze and polishing groups for each brand of ceramic were computed by T-Test.

**Results:** Analyses showed that the surface was dominated by Na, Al, Si. Glazed ceramics showed less ion change than polished ceramics at high intensity corrosion. No significant difference was found between Ra values of ceramics for both surface finishing methods, except Empress, a glass ceramic.

**Conclusion:** The surface compositions of ceramics were found to differ according to type and brand of ceramic. Surface elemental composition did not make a difference on surface roughness or surface finishing technique. On the other hand surface elemental composition changes after chemical corrosion giving a sign of dissolution of ceramics.

**Key words:** Dental ceramics, Corrosion, Surface elements

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### INTRODUCTION

Dental ceramic materials are considered chemically most inert reconstructive materials. Different kinds of ceramics possess different chemical properties. Because of this, inertness of a specific dental ceramic material cannot be generalized. The ceramic structure is based on electric neutrality [1]. With respect to the chemical composition, ceramic materials can be classified as silica based oxide and non-oxide ceramics. Sintered dental ceramics are mainly silica based ceramics, characterized by a continuous glass matrix in which different

volume fractions of crystals and particles are interspersed [2]. The glass matrix in conventional ceramics is predominantly based on a potash feldspar or soda feldspar. In addition to feldspar, other oxides are frequently added. To enhance the esthetic appearance of the ceramic material, colorant oxides of Fe, Cu, Co, Mn, and opacifying oxides of Sn, Zn, Al, Zr and Ti are also added. Some of the low or ultra-low fusing dental ceramics demonstrate compositional and microstructural differences from traditional porcelain fused to metal ceramics. The composition, microstructure and physical properties of newly launched ceramic materials differ from those of traditional ones, which may affect the inertness and surface roughness of the material that influence plaque accumulation on exposed surfaces [3]. Most commercial dental

ceramic materials are partially crystallized feldspathic glasses reinforced with alumina particles which induce increased coarseness in the surface. Modern low-fusing ceramics use fine-grained Lucite crystals or mullite crystals as the reinforcing material. Recently introduced dental glasses or hydrothermal glasses are free from reinforcing alumina particles or crystals are thus smoother and softer than traditional dental ceramics [4].

The surface roughness of ceramic materials may be influenced by many factors, such as the composition and microstructure of the ceramic material, glaze temperature and surface finishing method used.

The aim of this in vitro study was to compare the effect of surface elemental composition and surface finishing method on roughness of dental ceramics.

#### MATERIALS AND METHOD

20 ceramic disks (8 mm in diameter and 2 mm in height) were fabricated from A1 shade of Empress II, Zirkozahn, Noritake Cerabien ZR, Vita VM7 and Finesse veneering ceramics. The ceramic specimen was fabricated in plexiglass mold. Ceramic powder was mixed with a brush on a glass plate using the mixing liquid recommended by producers of each ceramic material. The mold was filled carefully with the mixture and condensed. Excess liquid was removed by applying a piece of adsorbent paper to the surface of the specimen. After condensation the mold was removed leaving the non-sintered test specimen on firing tray. Each specimen was sintered in a calibrated oven according to manufacturer's instructions by the same experienced dental technician. Test specimens were divided into 2 groups of autoglaze and polishing (n=10). All test surfaces were ground using 1000 grit SiC paper on a rotating disc at 150 revolutions/min under water cooling. The test specimens were finally ultrasonically cleaned in distilled water. Then, autoglaze was applied according to the manufacturer's instructions of each material. Mechanical polishing was applied using Shofu All Ceramic Finishing and Polishing wheels using a slow-speed handpiece rotating at approximately 10,000 rpm, as recommended by the manufacturer. Polishing

was performed by the same investigator. The adjustment kit consisted of a 4-step process: Two green stones and 2 different polishers were used once for 20 seconds. The specimens were then ultrasonically cleaned with distilled water and dried with a blast of air for 30 seconds before surface analysis. Surface roughness of the specimens was evaluated using a profilometer (Mitutoyo Surf Test 402 Analyzer; Mitutoyo Corp, Kawasaki, Japan). To measure the roughness profile value in  $\mu\text{m}$ , the diamond stylus was moved across the surface under a constant load of 3.9 mN and a speed of 0.100 mm/s with a range of 600  $\mu\text{m}$  during testing. This procedure was repeated 10 times at a different location for each specimen to obtain the general surface characteristics of the specimens. The average values of these measurements were considered to be the "Roughness average" (Ra) values. Prior to elemental analysis, the samples were subjected to a standard cleaning procedure involving short exposure in isopropanol under ultrasonic stirring, followed by careful drying in air. This proved to be necessary to ensure that the amount of surface contamination of hydrocarbons that always appear on metallic and ceramic samples of all types was minimized. The analyses were conducted using Energy Dispersive X-Ray Spectrophotometer (EDX). The energy was fixed at 93.9 eV and the analyzed areas were about  $0.8 \times 2 \text{ mm}^2$ . For each sample, a survey spectrum of the binding energy range of 0-1350 eV was recorded for 44 mins on the horizontally mounted samples. The angle between the sample surface and the analyzer as well as between the incoming X-ray beams was then 45°. From the spectrum, the characteristic peaks of elements present in the surface were identified and the apparent elemental surface composition was derived from the peak intensities. Access evaluation software and the standard relative sensitivity given therein by the equipment manufacturer. Before doing this, carbon was excluded from the calculations as this element arises from external contaminants. A survey spectrum was taken from the central area of each ceramic specimen and was repeated for 5 times. The range and mean values (in atom %) were registered and mean average was calculated for each element. After the initial surface 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) 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 [5]. 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 [5]. After cooling to room temperature, the specimens were removed from the bottles, rinsed in distilled water, dried, and a new surface analysis was performed. The data were statistically analyzed by Univariate Analysis of Variance and Bonferroni Test. Multiple comparisons of Ra values between autoglaze and polishing groups for each brand of ceramic were computed by T-Test. For the determination of correlation, between surface elements and roughness average, Spearman correlation test were utilized. Level of significance was chosen as 0.05.

## RESULTS

From the spectrum of ceramic surface composition, strong characteristic peaks

representing Na, Al, Si were found. The intensity of Si also varied according to different types of ceramic that was determined highest for Noritake CZR and lowest for Zirkozahn veneering ceramics (Table 1).

Surface composition of polished ceramics was found to be dominated by silicon, for Noritake CZR at all pH values. The highest surface Na change was found for polished Zirkozahn at high intensity corrosion at pH 2.4 and no surface Na concentration was calculated for Empress (Table 2).

When comparing the different alkali ions it can be seen that, the surface content of Na was stronger even after low intensity corrosion with minor differences at percentages of different ceramic brands. Fe was found only on polished Zirkozahn surfaces while it was not existed on glazed ones. No significant difference was found for surface elemental Al change at various pH and temperatures for polished and glazed groups (Table 3). No systematic tendency in the deviations was noted for Na, Al, and Si. No reasonable correlation was found between the overall elemental surface composition and the surface roughness ( $p>0.05$ ).

For Empress II the difference for Ra values was significant between autoglaze and polishing groups ( $p<0.05$ ). Polishing group showed smoother surfaces than glazed group (Table 4). No significant difference was found between Ra values of the other four brands of ceramic material for both surface finishing methods ( $p>0.05$ ). The evaluation of two different surface finishing methods showed mechanical polishing created as smooth surfaces as the autoglazed specimens.

Table 1: Surface Si change ratio of glazed and polished groups at different pH values.

Si exchange ratio (%)		Noritake	Empress	Zirkozahn	Finesse	Vita
<b>Glazed ceramics</b>						
pH 2.4	Mean ± SD	8.85 ± 2.3	13.5 ± 30.6	1.72 ± 1.97	9.06 ± 3.07	0.89 ± 2.3
pH 4.5	Mean ± SD	1.52 ± 1.3	7.05 ± 0.9	4.38 ± 1.6	1.55 ± 2.2	2.28 ± 7.1
pH 7	Mean ± SD	6.81 ± 1.2	6.57 ± 3.3	7.13 ± 1.2	6.94 ± 2.9	2.08 ± 4.3
pH 9	Mean ± SD	2.55 ± 3.3	2.7 ± 0.7	4.12 ± 1.1	4.87 ± 2.09	0.85 ± 1.7
<b>Polished ceramics</b>						
pH 2.4	Mean ± SD	23.83 ± 2.6	7.6 ± 2.4	6.35 ± 3.1	6.95 ± 3.8	3.36 ± 1.1
pH 4.5	Mean ± SD	23.61 ± 2	8.37 ± 1.9	4.9 ± 9.9	0.49 ± 2.2	1.33 ± 2.9
pH 7	Mean ± SD	25.1 ± 1.97	12.23 ± 1.1	7.47 ± 3.4	0.52 ± 1.7	1.56 ± 3.03
pH 9	Mean ± SD	21.63 ± 0.7	7 ± 2.83	4.88 ± 3.2	7.31 ± 0.29	4.35 ± 3.2

**Table 2: Surface Na change ratio of glazed and polished groups at different pH values.**

Na exchange ratio (%)		Noritake	Empress	Zirkonzahn	Finesse	Vita
<b>Glazed ceramics</b>						
pH 2.4	Mean ± SD	0.47 ± 2.1	-	2.24 ± 3	0.006 ± 1.3	1.69 ± 1.6
pH 4.5	Mean ± SD	1.83 ± 1.9	-	0.9 ± 1.9	6.85 ± 4.7	1.21 ± 2.2
pH 7	Mean ± SD	0.75 ± 2.1	-	2.94 ± 4.2	8.06 ± 2.9	1.65 ± 3.1
pH 9	Mean ± SD	1.95 ± 2.5	-	1.5 ± 1.6	10.7 ± 0.9	2.28 ± 0.8
<b>Polished ceramics</b>						
pH 2.4	Mean ± SD	0.33 ± 0.7	-	10.06 ± 3.2	2.56 ± 2.7	0.21 ± 0.69
pH 4.5	Mean ± SD	4.27 ± 1.1	-	6.87 ± 0.9	6.46 ± 2.6	4.11 ± 0.8
pH 7	Mean ± SD	7.51 ± 0.9	-	4.51 ± 2.5	2.9 ± 1.6	4.61 ± 0.6
pH 9	Mean ± SD	1.36 ± 2.2	-	5.02 ± 0.9	7.84 ± 4.6	3.8 ± 0.4

**Table 3: Surface Al change ratio of glazed and polished groups at different pH values.**

Al exchange ratio (%)		Noritake	Empress	Zirkonzahn	Finesse	Vita
<b>Glazed ceramics</b>						
pH 2.4	Mean ± SD	0.78 ± 0.3	2.72 ± 38	1.69 ± 1.5	0.65 ± 1.7	0.27 ± 0.8
pH 4.5	Mean ± SD	0.13 ± 0.4	1.31 ± 3.1	1.31 ± 0.67	0.21 ± 2.6	1.65 ± 5.6
pH 7	Mean ± SD	0.28 ± 3.4	0.25 ± 0.49	1 ± 1.6	2.7 ± 2.3	1.18 ± 3.7
pH 9	Mean ± SD	0.53 ± 1.9	0.61 ± 1.3	0.15 ± 2.1	2.63 ± 3.7	0.45 ± 3.3
<b>Polished ceramics</b>						
pH 2.4	Mean ± SD	1.36 ± 2.7	0.12 ± 0.9	0.91 ± 1.7	1.46 ± 1.9	1.6 ± 0.2
pH 4.5	Mean ± SD	0.56 ± 1.5	3.51 ± 1.8	3.38 ± 2.7	2.15 ± 0.8	0.76 ± 0.62
pH 7	Mean ± SD	0.76 ± 2.3	1.02 ± 2.5	4.5 ± 1.4	0.98 ± 1.7	1.57 ± 3.2
pH 9	Mean ± SD	2.69 ± 1.1	4.48 ± 2.9	0.7 ± 5.4	4.03 ± 2.9	3.21 ± 2.9

**Table 4: Surface Ra change of glazed and polished groups at different pH values.**

Ra Difference Mean ± SD		Noritake	Empress	Zirkonzahn	Finesse	Vita
<b>Surface Method</b>		<b>Glazed</b>	<b>Glazed</b>	<b>Glazed</b>	<b>Glazed</b>	<b>Glazed</b>
pH 2.4	Ra	0.8 ± 0.03	0.8 ± 0.03	0.7 ± 0.06	0.7 ± 0.05	0.7 ± 0.07
	Ra final	0.2 ± 0.09	0.5 ± 0.1	0.6 ± 0.2	0.4 ± 0.05	0.2 ± 0.01
pH 4.5	Ra	0.8 ± 0.08	0.7 ± 0.09	0.8 ± 0.03	0.8 ± 0.05	0.7 ± 0.1
	Ra final	0.25 ± 0.1	1.5 ± 0.3	0.4 ± 0.2	1.4 ± 0.4	0.2 ± 0.06
pH 7	Ra	0.8 ± 0.04	0.8 ± 0.03	0.7 ± 0.02	0.7 ± 0.08	0.7 ± 0.09
	Ra final	0.2 ± 0.07	1.8 ± 0.1	0.3 ± 0.06	1.5 ± 0.3	0.3 ± 0.2
pH 9	Ra	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.05	0.7 ± 0.06	0.8 ± 0.03
	Ra final	0.3 ± 0.1	1.1 ± 0.3	0.3 ± 0.04	1.2 ± 0.2	0.4 ± 0.1
<b>Surface Method</b>		<b>Polished</b>	<b>Polished</b>	<b>Polished</b>	<b>Polished</b>	<b>Polished</b>
pH 2.4	Ra	0.7 ± 0.06	0.6 ± 0.08	0.6 ± 0.02	0.7 ± 0.09	0.6 ± 0.02
	Ra final	1.3 ± 0.5	0.4 ± 0.1	0.4 ± 0.1	0.4 ± 0.04	0.2 ± 0.04
pH 4.5	Ra	0.63 ± 0.1	0.6 ± 0.09	0.7 ± 0.06	0.7 ± 0.04	0.7 ± 0.05
	Ra final	0.2 ± 0.08	0.4 ± 0.1	0.3 ± 0.08	0.3 ± 0.08	0.3 ± 0.06
pH 7	Ra	0.5 ± 0.07	0.7 ± 0.06	0.8 ± 0.02	0.7 ± 0.06	0.7 ± 0.02
	Ra final	0.3 ± 0.05	0.4 ± 0.05	0.5 ± 0.1	0.2 ± 0.06	0.3 ± 0.04
pH 9	Ra	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.07	0.7 ± 0.1	0.7 ± 0.1
	Ra final	0.2 ± 0.08	0.3 ± 0.1	0.18 ± 0.1	0.47 ± 0.1	0.2 ± 0.03

**DISCUSSION**

Ceramic materials have multiple flaws because of the inhomogeneous distribution of crystals in a glassy matrix. Additional defects formed during ceramic processing reduce the strength and increase the wear of enamel [6-8]. Sealing these irregularities may improve the strength

and reduce the abrasiveness of dental ceramics. Surface glazing produces a glassy and hygienic surface when performed at a relatively low temperature and increases the strength of the ceramic system. Surface glazing reduces the wear of opposing teeth; however, the glazed layer is easily removed by an occlusal adjustment at chairside or after a short period in function. If the

exposed surface is not adequately polished, the ground surface may lead to accelerated abrasive wear of the opposing dentition, increased plaque accumulation, and reduced strength of the ceramic material [9,10]. From this point of view, this study aimed to compare the results of glazed and polished ceramics after in vitro corrosion to simulate oral conditions that occurs over years.

A number of studies were made to compare finishing and polishing techniques that would create surfaces as smooth as glazed ceramics [11-13]. The results were inconsistent. Some authors found the initial smoothness of a glazed surface superior to the polished surface, some found no significant difference between the glazed and polished surfaces [12,14]. Some concluded that surface polishing could equal the smoothness performed by surface glazing [15-17]. More recent studies suggest that a polished surface is smoother and fosters less plaque adhesion than a glazed surface [17,18].

This study hypothesized that the different ceramic finishing techniques would have different effects on the surface roughness and surface elemental composition would affect the surface roughness of feldspathic porcelain. The surface roughness of glazed and polished ceramic surfaces has been investigated, and it was reported that polishing created as smooth surfaces as glazed ones. According to results of the study, surface elemental composition also changed due to the intensity of corrosion and surface finishing methods.

Superficial layer of the materials are largely controlled by the chemical composition and microstructure of the surface. As expected, the surface composition of the different ceramics was dominated by oxygen, silicon, aluminum, potassium and sodium, the elements which form feldspar. The microstructure can be defined as partially crystallized feldspar or glass ceramic with additions of opacifying and strength-promoting oxides. Although qualitatively similar, the different native glass-phased ceramics displayed quantitative differences [19]. Compared with information available from the manufacturers, the surface elemental composition determined by EDX was found to correspond but differs in detail. Porcelain surface elemental composition did not make a difference on surface roughness or surface finishing technique. The evaluation of

two different surface finishing methods showed mechanical polishing created as smooth surfaces as the auto glazed specimens.

### CONCLUSION

When comparing the spectra in surface composition, Ca, Sn, Fe and Ti were found at small levels on the surface, and not involved in all ceramic types. These elements are apparently added to ceramic composition as colorant and opacifying oxides since they are not found in all types. Most probably this difference can be attributed to the fact that, this element arised from the polishing wheels and could not be removed by ultrasonic cleaning.

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