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EFFECT OF CORROSION AND FINISHING TECHNIQUE ON MICRO-HARDNESS AND TRANSLUCENCY OF DENTAL CERAMICS

Moustafa S. Mohammed* *and* Mohammed G. Hussein**

ABSTRACT

Purpose: This research aimed to compare microhardness and translucency of lithium disilicate glass ceramic and high translucent monolithic zirconia before and after being exposed to a corrosive medium and using different finishing techniques for translucency measurement.

Materials and Methods: Two main ceramic materials, Lithium Di-silicate (IPS e.max CAD LT A3, C14) and High Translucent Zirconia (HTZ) from DD BioZx2 A3-HT, along with 4% acetic acid as a corrosive agent were employed in this study. Eighty ceramic discs(10x12mm) were classified into two groups (40 discs each) according to the type of ceramic used then each group was subdivided into two subgroups (20 discs each) according to the performed test (VHN and TP). Then the VHN subgroup was divided into two classes according to the corrosion procedure (10 corroded and 10 not corroded) and the TP subgroup was divided into two classes according to the finishing procedure (10 glazed and 10 polished) and all the 20 TP sample discs were immersed in 4% acetic acid as a corrosive agent.

Results: In the microhardness test (VHN), there was a statistically significant difference in the Emax subgroup samples before and after correction (p= 0.027), while the difference in the HTZ subgroup samples was not statistically significant (p= 0.075). When comparing the samples of both materials, a statistically significant difference was observed, with the HTZ samples showing a higher VHN either before or after corrosion (p=0.001). For the translucency parameter (TP), both glazed and polished corroded E-max samples showed no statistically significant difference (p = 0.149). HTZ samples followed the same pattern (p = 0.853). Comparing glazed samples of E-max and HTZ revealed no significant difference (p = 0.067), while polished samples showed a statistically significant difference, with polished E-max having superior translucency over HTZ (p = 0.029).

Conclusions: HTZ was not significantly impacted by either corrosion or the finishing technique. In contrast, Emax showed a significant reduction in VHN values due to corrosion, though the finishing technique had no significant effect. HTZ consistently exhibited higher microhardness values both before and after corrosion, while polished Emax samples had the highest translucency parameter (TP) values.

KEYWORDS: Corrosion, microhardness, zirconia and translucency

* Lecturer, Fixed Prosthodontics Department, Faculty of Dentistry Assuit University,

INTRODUCTION

The demand for ceramic restorations has increased due to the search for clinical materials that closely mimic the optical properties of dental parts. Ceramics have been the material of choice in the field of restorative dentistry because of their biocompatibility, clinical endurance, and capacity to replicate the color, translucency, and surface texture of dental enamel. [1]

For both anterior and posterior restorations, including veneers, inlays, onlays, metal–ceramic, and all-ceramic restorations, dental ceramics are frequently employed. Their exceptional aesthetic qualities, biocompatibility, and wear resistance are what primarily drive their uses. Furthermore, dental ceramics are regarded as restorative materials that are chemically inert. Nonetheless, the chemical makeup and microstructures of ceramics vary greatly from one another. The chemical characteristics of various ceramic types vary. As a result, it is impossible to generalize about the inertness of a particular dental ceramic. [2]

The growing adoption of CAD/CAM systems in dentistry has led to the development of diverse monolithic ceramic blocks, each possessing distinct flexural strength and esthetic characteristics. Alongside monochromatic blocks, the industry has introduced multi-colored CAD/CAM blocks to address esthetic disparities between the restoration and the natural tooth. These blocks aim to replicate the natural dentine and enamel by offering multiple chroma and translucency variations across different regions, extending from the cervical to incisal thirds. It's worth noting, however, that not all CAD/CAM blocks come with multicolor options. (3) In the clinical setting, adaptation procedures performed during the delivery of a patient's restoration result in the formation of a rough surface, necessitating intraoral finishing and polishing. These treatments are essential to minimize wear on opposing teeth by reducing the restoration's abrasiveness and ensuring hygiene by preventing bacterial adherence. Previous research indicates that finishing treatments contribute to increased color stability in restorations. Despite optimal color selection, materials may undergo clinically noticeable color changes in the oral environment. Various surface finishing treatments apply to esthetic CAD/CAM restorations, with glazing in a porcelain furnace being the most commonly preferred treatment before cementation. Recent studies have also demonstrated that a smooth and lustrous surface can be achieved through manual polishing in addition to glazing. [3]

During the clinical adaptation process, ceramic restorations should ideally undergo as little modification as possible to preserve the brightness and surface smoothness that are usually attained by glaze application. However, at this point, certain practical and aesthetic changes are typical, and the thin glaze coating may chip off and expose a rough surface. [4]

Thermal aging and exposure to acidic solutions cause dental ceramics to deteriorate, changing their surface roughness and microhardness. [5]

The null hypothesis of this study postulates that the **corrosion process will not affect the microhardness, and the finishing technique will not affect the TP.**

MATERIALS AND METHODS

Sample Grouping:

Based on the type of ceramic used, eighty 10x12mm ceramic discs were divided into two groups (40 discs each) and then further divided into two subgroups (20 discs each) based on the test that was conducted (VHN and TP). Next, all 20 of the TP sample discs were submerged in 4% acetic acid as a corrosive agent. The VHN subgroup was divided into two classes based on the corrosion procedure (10 corroded and 10 not corroded), and the TP subgroup was divided into two classes based on the finishing procedure (10 glazed and 10 polished).

Disc fabrication

A ceramic cylinder was designed, 10mm diameter and 12mm in length the aid of CAD system software (exocad GmbH, Darmstadt, Germany). The size of zirconia cylinders was made 12.5 ×15mm to compensate for the shrinkage that will occur after sintering, as the Shrinkage Ratio of the used blocks was 0.25. The shape of the cylinder was confirmed and exported to the CAM system. After sintering, discs with a 2 mm thickness and 10 mm diameter were created by cutting cylinders with an IsoMet 4000 micro saw (Buehler Germany precision cutting, Germany). A digital caliper (Mitutoyo, Mitutoyo America Corporation, California) was then used to measure the thickness of each disc.

Finishing of the Samples (subgroups)

Glazing:

Glazing of E-max and HTZ samples: The glazing procedure was carried out using an oven (Programat P310 G2; IvoclarVivadent, Schaan, Liechtenstein) with IPS Ivocolor Glaze Paste (IvoclarVivadent, Schaan, Liechtenstein).

Polishing:

E-max CAD polishing: The specimens underwent finishing and polishing procedures using Eve Diapol, EvE Ernst Vetter GmbH (Rastatter Str, Pforzheim, Germany). According to manufacturer's instructions, initially, finishing with the green discs (medium) with approximately 35 microns particle 6 size was done. Subsequently, the grey wheel (fine) with a particle size of 4–8 microns was used for pre-polishing and smoothing. Finally, for high-luster polishing, the pink wheel (extra fine) with a particle size of about 1-2 microns was used. Every step of the process took one minute, and the recommended speed was 7000 rpm. Polishing was executed using a straight handpiece (NSK EX-6B, Japan), mounted to a specialized device (Fig.1) to ensure standardization of grinding pressure, direction, and rate applied to the samples. [6]

HTZ Polishing: The polishing process involved utilizing the OptraFine ceramic polishing kit (Ivoclar Vivadent) in the following sequence (according to manufacturer instructions): light blue silicone points for initial finishing at 15,000 rpm, followed by dark blue silicone points for polishing at 15,000 rpm. The final step included using a nylon brush along with diamond polishing paste for ultimate polishing at 10,000 rpm. Each polishing point was applied for a duration of 40 seconds. [7]

Aging procedure

Initially, the specimens underwent a thorough cleaning process involving three washes with ethyl alcohol, followed by drying with face tissue (Fine, Egypt). Subsequently, they were immersed in a 4% acetic acid solution at a temperature of 80°C, adhering to the ISO 6872 (8) standards for evaluating the hydrolytic resistance of dental ceramic materials. This immersion was sustained for a duration of 16 hours. Afterward, the specimens were allowed to cool to room temperature and then extracted from the solution. Following this, the samples underwent rinsing with distilled water and alcohol before being dried. [8]

Microhardness Test

Surface microhardness was measured in a digital microhardness tester (Model HVS-50, Laizhou Huayin Co., China). Vickers diamond indenter of the device performed three indentations on working surface of each sa mple with 200 grams load for 20 seconds to determine Vickers hardness number (VHN). The length of the indentation lines was measured at40× through the built-in scale microscope. Fig. **(1)** The measurements were converted into a micro-hardness value (VHN) using the following equation:

$$HV = 1.854\ P/d2$$ [9]

Where HV is micro-hardness in kg/mm2, P is the load in kgf and d is the average length of the diagonals in mm.

Three indentations were applied for each specimen at three different locations (left, right, and central regions).

Evaluation of translucency parameter:

Translucency is the property of a substance that permits the passage of light but disperses the light so that an object cannot be observed clearly through the material, i.e., a state between complete opacity and transparency. Based on the CIE L*a*b* system, the translucency of a material is usually determined with the translucency parameter (TP). TP refers to the color difference of a uniform thickness of a material over black and white backgrounds, which corresponds directly to the visual assessments of translucency. The greater the TP value, the higher the actual translucency of a material. If the material is opaque the TP value is zero and if the material is totally transparent TP=100. The TP of the corroded samples was obtained by calculating the color difference between the specimen over the white background and that over the black background by the following equation: TP = [(Lb – Lw) 2 + (ab – aw) 2 + (bb – bw) 2] ½ [10]

Where 'b' refers to the color coordinates over the black background and 'w' refers to those over the white. The color parameters L*a*b* were obtained by measuring the samples using EasyShade spectrophotometer. **Fig (2)**

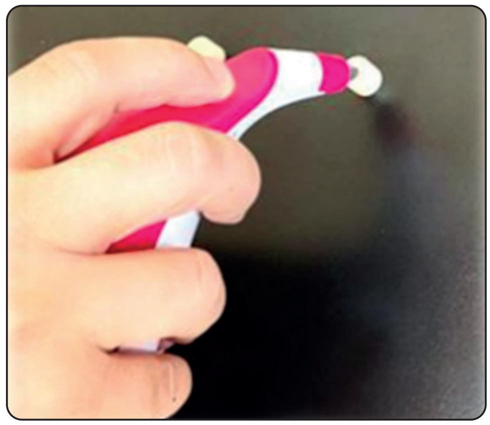

Fig. (2) Easy shade spectrophotometer

Statistical Analysis:

The collected data were systematically tabulated and subjected to statistical analysis. Mean and standard deviation values were computed for each group in every test. To compare the mean differences over time, a one-way ANOVA was conducted. A p-value less than 0.05 was considered significant.

RESULT

Micro hardness results:

Relation between before and after corrosion test:

The Micro hardness mean values obtained in (VHN) are shown in Table (1) & figure (1). The statistical test showed:

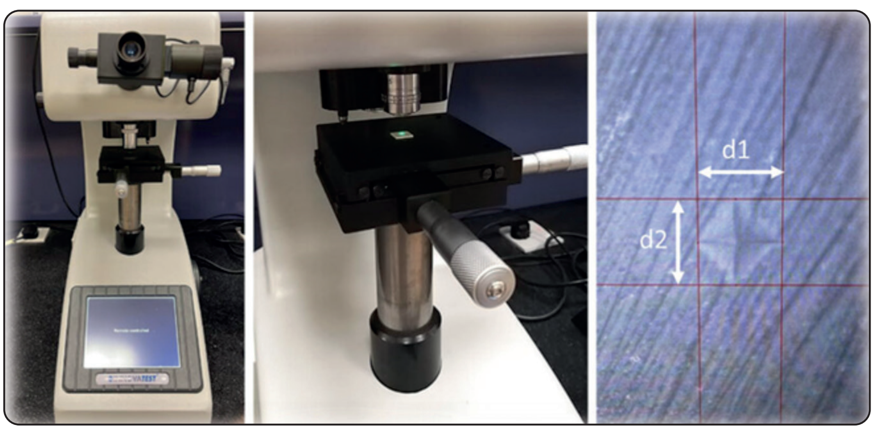

Fig (1) Microscopic image of indentation crack showing crack extension

E-Max group:

There was a statistically significant difference between (Before) and (After) where (p= 0.027). The highest mean value was found in (Before) while the least mean value was found in (After).

HTZ:

There was no statistically significant difference between (Before) and (After) where (p= 0.075).

The highest mean value was found in (Before) while the least mean value was found in (After).

Relation between groups:

There was a statistically significant difference between (E-Max) and (HTZ) where (p=0.001) before and after corrosion as shown in Table (1) & figure (3).

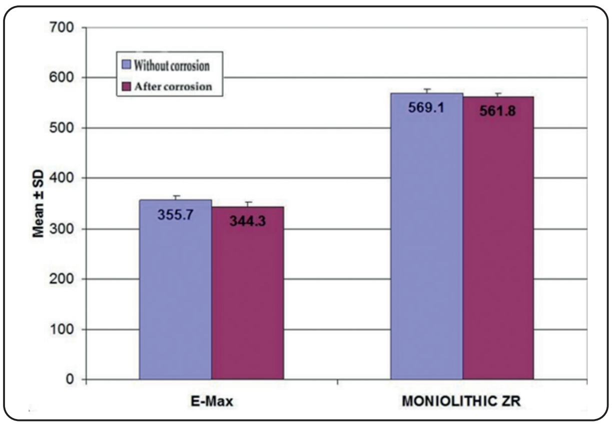

Fig. (3) Comparison between the micro hardness of tested ceramics

Translucency Parameter results

The mean and standard deviation of the Translucency Parameter (TP) were measured for both material groups, lithium disilicate (IPS E-max CAD) and high translucency zirconia (HTZ) and among the subgroups according to finishing technique (glazing and polishing). The results are measured and presented in tables: (2,3) and figure (4)

1. TP of (E-max samples) Results showed that there was no statistically significant difference between the subgroups (glazed/polished) p-value=0.149 (table, 1 fig, 3).
2. TP of (HTZ samples) Results showed that there was no statistically significant difference between the subgroups (glazed/polished) p-value=0.853. (table,1 fig,3)
3. TP of the (glazed samples) Results showed that there was no statistically significant difference between the glazed samples of both materials (E-max/HTZ) p-value=0.067 (table, 2 fig, 3)
4. TP of the (polished samples) Results showed that there was statistically significant difference between the polished samples of both materials (E-max/HTZ) p-value=0.029. Where the polished E-max group shows more translucency than the polished HTZ group. (Table, 2 fig 3)

TABLE (1) Means, standard deviations (SD), and statistical analysis results of micro hardness (VHN)

Groups	Micro hardness				P-value
	Without corrosion		After corrosion		
	Mean	SD	Mean	SD	
E-ma.x	355.71	9.53	344.28	8.32	0.027·
MONIOLITIDC ZR	569.12	7.57	561.77	7.70	0.075
p-value	<0.001*		<0.001*		

***** *Means statistically significance difference, significant (p<0.05) ; non-significant (p>0.05)*

TABLE (2) TP of (E-max and HTZ groups)

TP Mean±SD	Method		P-value
	Glazed	Polished	
E-max group	14.67 ± 1.27	15.79 ± 1.68	0.149
HTZ group	13.76 ± 0.21	13.85±1.4	0.853

TABLE (3) TP of (Glazed / polished samples of both material groups)

ΔE Mean±SD	Material		P-value
	E-max	HTZ	
Glazed samples	14.67 ± 1.27	13.76 ± 0.21	0.067
Polished samples	15.79 ± 1.68	13.85 ± 1.40	0.029*

The * symbol indicates statistically significance difference
****; significant (p<0.05) ns; non-significant (p>O.05)***

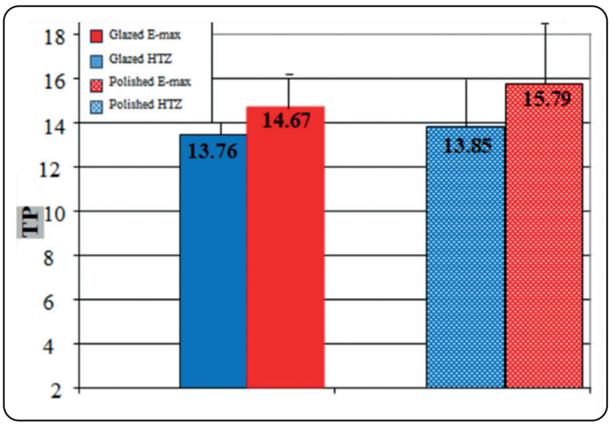

Fig. (2) Easy shade spectrophotometer

DISCUSSION

This study investigated whether there is a significant change in micro hardness and translucency occurring after corrosion by acetic acid and after different finishing techniques. In the present study, the different ceramic materials were kept in a 4% acetic acid solution at 80ºC for a shorter period of time to permit the detection of early surface changes. Compared with the ISO standard for hydrolytic stability tests of ceramic materials [11], the time factor was increased from 16 to 18 h to compensate for the time taken to reach the recommended temperature level.

Acetic acid was selected for its pH value (pH 2.4), which closely resembles the pH values of certain beverages, juices, and those encountered in dental plaque. [12] Additionally, acetic acid is a commonly utilized acid for domestic applications. [13] The current ISO 6872:1995(E) standard employs 4% acetic acid as the chemical agent for assessing the chemical solubility of ceramic materials through an 18-hour reflux process. [8] Aging of the tested ceramics has been done after finishing the ceramic samples to resemble the effect of oral environment on the ceramic restorations.

Dental computer-assisted design and computer-assisted manufacturing (CADCAM) technology are extensively utilized to streamline the production of ceramic restorations, with the goal of reducing the number of clinical visits as well as the amount of production time needed. Because ceramic restorations are more biocompatible and have greater chemical stability than traditional metal-ceramic restorations, clinicians prefer using them. [14,15,16]

Hardness is an essential property to compare restorative materials' resistance to surface indentation through a combined effect of brittle fracture as well as plastic flow. It marks out materials' wear resistance as well as their abrasiveness to the opposing material. It directly affects materials' ability for finishing and polishing [17,18]. A pyramidal diamond indenter was compressed into a polished surface under known load and conditions to measure micro hardness to correlate indentation size to the materials' hardness. [19,20,21].

Among a variety of indentor geometries used in hardness testing, the Vickers indentor is one of most widespread use. The Vicker Hardness Test was selected because it is suitable for determining the hardness of small areas as used by previous investigator [22,23].

The type of surface finishing is an important factor in translucency. [24] Several ceramic materials and polishing systems are now available. In the current study, polishing was performed for the specimens according to the manufacturer's instructions, and regarding the evidence proving that surface luster and translucency are improved with properly polished surfaces. [25] Several studies have confirmed that various chair-side ceramic polishing systems yield surfaces as smooth as those achieved through glazing. [26,27] A spectrophotometer was used in this in vitro investigation because of its ability to produce objective measurements without the subjective influence of color. [28]

The null hypothesis of this study was partially rejected, as elucidated in the subsequent discussion.

The current study results came in partial agreement with Assad R and Salem S (2021) [29] who recorded no significant effect of aging on micro hardness of lithium di silicate and zirconia reinforced lithium silicate ceramics, which is in contrast with our results regarding only lithium di silicate and in agreement with our results regarding zirconia reinforced lithium silicate ceramics.

Also, our study was in contrast with Vasiliu et al (2020) [30] who recorded no

Significant effect of thermo-cycling on micro hardness of lithium disilicate and zirconia reinforced lithium silicate ceramics, while they found no significant difference in micro hardness of both ceramics. They related this to the regular scattering crystals within ceramic structure of both materials [30]. Also, hardness value of any material is a function of test type and loading conditions [31].

The present results agree with El-Sharkawy (2020) [32], who reported that the staining drinks and coffee have a negative effect on translucency of lithium silicate and disilicate materials. Also, they showed that the mechanical polishing showed better translucency results for both materials; This could be attributed to the glaze layer may reflect part of the incident light, so the amount of light passing through the materials is decreased. [32] The results of TP evaluation also agreed with Demirel (2022) [33] who showed that the lithium disilicate samples showed the highest values of TP along the study. They explained that chemical composition and crystalline structure have a greater influence on translucency. [33] The result of current study was in contradiction with Alp G et al (2018) [34] who found that the glazed e-max group presented higher translucency than polished surfaces. Difference between the current study and those in the study by Alp G et al may be due to the differences between the evaluated thicknesses of the tested material. Finally, the present study was in contrast with Roxana et al (2023) [35] as they showed that after aging; an increase in TP values was reported for lithium disilicate and zirconium samples. An increase in translucency, explained by the reduction of light scattering from the boundaries of the cubic phase particles during the aging process. Also, they stated a lower TP value of polished Ceramill Zolid fx; An explanation for the decrease in TP of polished CeZ would be that the high roughness of the material negatively affects the appearance of the surface. [35] Given the limitations inherent in this in vitro study, it is crucial to emphasize the need for further research into the optical and various mechanical properties of monolithic CAD-CAM restorative materials. Specifically, studies should aim to simulate the diverse variables present in the intraoral environment to provide definitive clinical recommendations.

CONCLUSIONS

1. Aging had no significant impact on the micro-hardness of E.max or monolithic zirconia. However, monolithic zirconia showed higher hardness than E.max, especially before corrosion.
2. Commonly the finishing technique (mechanical polishing/glazing) has statistically no significant effect on the translucency parameter (TP) of the tested materials.
3. Mechanical polishing showed better translucency results than glazing of the tested samples.
4. Corroded E-max has a better translucency than corroded HTZ.

RECOMMENDATIONS

With the limitation of this study (in vitro) further research of other mechanical and optical properties of monolithic CAD-CAM restorative material is needed, especially by simulating the variables of the intraoral environment to make definitive clinical recommendations. Additional in vivo investigations assessing clinical complications, biocompatibility, wear, micro-leakage, color stability, adhesion of these materials to tooth structures, dental cements, and the overall survival rate are essential for the validation of their clinical utility.

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