

## A COMPARISON OF THE EFFECT OF ARTIFICIAL SALIVA WITH DIFFERENT PH VALUES ON SURFACE ROUGHNESS OF VENEERING CERAMIC TO METAL AND ZIRCONIA SUBSTRUCTURE (IN VITRO STUDY)

Inas Mohammed<sup>1</sup> and Dr. Zahraa Nazar Alwahab<sup>2\*</sup>

<sup>1</sup>B. Dent Tech, Ministry of Health, Baghdad, Iraq.

<sup>2</sup>Assistant Professor, BDS, MSc, Middle Technical University, College of Health and Medical Technologies. Baghdad, Iraq.

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### \*Corresponding Author

**Dr. Zahraa Nazar  
Alwahab**

Assistant Professor, BDS,  
MSc, Middle Technical  
University, College of  
Health and Medical  
Technologies. Baghdad,  
Iraq.

### ABSTRACT

**Background:** Dental ceramics are the restorative material used in dentistry because of their excellent aesthetic and biocompatibility.<sup>[1]</sup>

They have shown the most accurate reproduction of the appearance, color and texture of natural teeth,<sup>[2]</sup> so they are widely used for both anterior and posterior restoration such as inlays, onlays, veneers, metal- ceramic and all- ceramic restorations. **Objective:** The purpose of this in vitro study is to compare the effect of artificial saliva with different pH values (3.5, 7.0, and 10.0) on surface roughness of veneering ceramic to metal and zirconia substructure. **Materials and methods:** Sixty samples rectangular in shape (9mm length X 4mm width X 4mm height) were fabricated from metal and zirconia equally. The veneering ceramic was build up to (3mm height) on metal and zirconia samples. The thirty metal samples were from KERA NH, Ni –

Cr alloy then veneered with Vita VMK Master ceramic. The thirty zirconia samples were prepared from pre sintered IPS Emax Zir CAD, then after sintering, they were veneered with IPS Emax Ceram. Each group was divided randomly into three subgroups according to pH values of storage solutions (Neutral 7.0, acid 3.5, and alkaline 10.0). They were stored in incubator for 21 days at 37 C°. Surface roughness (Ra in µm) were recorded in before and after using roughness tester device (profilometer device). **Results:** The highest mean surface roughness values were for acidic group, followed by alkaline group and then neutral group for both metal and zirconia groups. There was a high statistical significant difference in t –

test between metal ceramic and zirconia group after storage. There was a high significant difference within metal ceramic group or zirconia ceramic group before and after storage.

**Conclusions:** Within the limitation of this *in vitro* study, it can be concluded that, the artificial saliva had significant effect on surface roughness for metal ceramic and zirconia ceramic groups.

**KEYWORDS:** Metal ceramic, zirconia ceramic, surface roughness.

## INTRODUCTION

Dental ceramics are the restorative material used in dentistry because of their excellent aesthetic and biocompatibility.<sup>[1]</sup> They have shown the most accurate reproduction of the appearance, color and texture of natural teeth,<sup>[2]</sup> so they are widely used for both anterior and posterior restoration such as inlays, onlays, veneers, metal- ceramic and all- ceramic restorations.<sup>[3]</sup> For more than 40years, metal ceramic system has been extensively used in fixed partial denture and still represents the gold standard.<sup>[4]</sup> This reliable choice can not only provide aesthetic characteristic, but also enjoys mechanical features such as high flexural and shear bond strength (SBS).<sup>[5]</sup>

However, metal ceramic restoration shows the problem of metal discoloration at the margin, allergic reaction and sensitivity to various metals. The metal substructure is opaque and does not duplicate the inherent translucency of natural teeth. Hence, the need for a restoration that mimics the natural tooth in esthetic and strength led to the development of Yttrium tetragonal zirconia poly crystal (Y- TZP) based material.<sup>[6]</sup> Zirconia- based ceramics are among the most popular and the most widely used ceramics in the modern dental practice. This can be attributed to their superior mechanical properties,<sup>[7]</sup> and excellent biocompatibility comparable to other dental ceramics.<sup>[8]</sup>

The oral cavity is a dynamic environment where changes constantly occur. It is also a warm and moist environment which represents many factors that would adversely affect the mechanical properties of ceramic restorations: (a) water from saliva; (b) masticatory stresses; (c) stresses due to differences in the coefficients of thermal expansion of restorative materials; (d) temperature variations; and pH variations.<sup>[9]</sup> The stability of dental ceramics in the oral environment is directly related to the high surface polish. Subcritical crack propagation and chemical inertness of these materials, which in turn enables them to resist degradation in the oral environment.<sup>[5]</sup>

A smooth restoration surface is important in three terms: function, esthetic and biologic compatibility that avoids dental complications such as plaque formation, gingivitis, periodontitis, and wear of the opposing dentition. It is also important for patient comfort.<sup>[10]</sup> In bilayer restoration, veneering ceramic may be cracked or delaminated during function.<sup>[11]</sup> Therefore the bond strength between the veneering ceramic and the substructure plays important role in the success of these restorations.<sup>[12]</sup>

## MATERIALS AND METHODS

Part I: Preparation of metal ceramic specimens.

Thirty rectangular shape samples were fabricated from metal, having dimensions (9mm length X 4mm width X 4mm height) according to the design used by Choi<sup>[13]</sup>, that associated to international organization for standardization 1999.<sup>[14]</sup>

Surface of the rectangular samples (4mm X 4mm) were veneered with Vita VMK Master® veneering according to manufacturer's instructions. Final thickness of veneered ceramic was 3mm. (Figure 1).

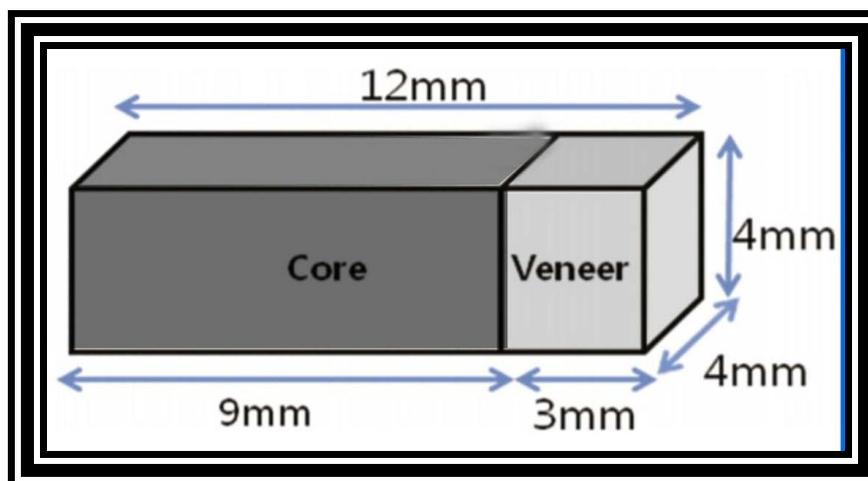
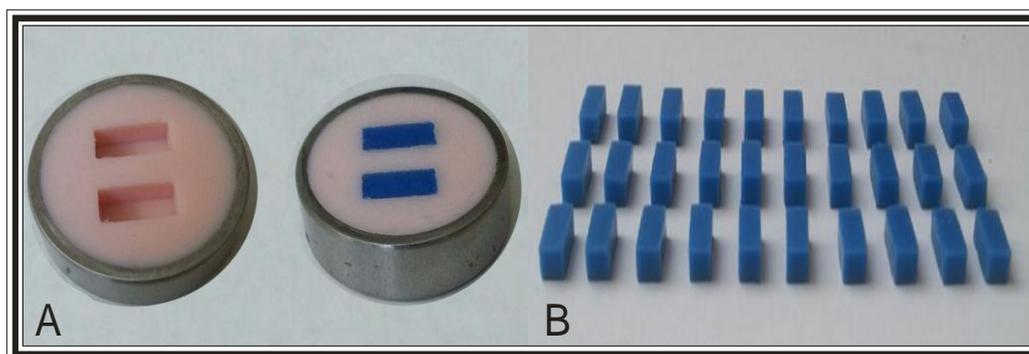


Figure 1: Final dimensions of the specimen.

### Wax pattern preparation

Thirty samples of wax pattern rectangular in shape with dimensions (9mm length × 4mm width × 4mm height) were constructed using custom made rubber mold from silicon material (Figure 2).



**Figure 2: Wax pattern preparation.**

A. Custom made rubber mold B. Wax patterns of samples.

### **Burnout Procedure**

Following the investing and bench set procedure, the burnout was done according to the manufacturer instructions.

### **Casting Procedure**

Casting Procedure was done by the use of centrifuge machine for casting the nickel chromium alloys) (Kera@NH, Germany).

**Oxidation Procedure:** The oxide layer on each metal sample was formed by heating inside a ceramic furnace at 980°C for 5 minutes with vacuum according to the manufacturer instruction.

**Sandblasting Procedure:** Sandblasting was done with 250µm aluminum oxide under 3- 4 bar pressure according to the manufacturer's instructions for 10 seconds.

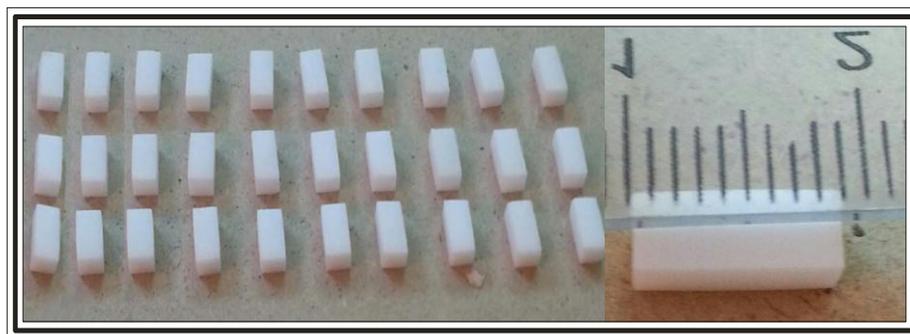
**Veneering Ceramic application:** Opaque layer was applied, and then followed Dentin and enamel. After complete firing, checking of the dimensions of veneering ceramic was done by digital vernia, and then adjustment was done by using straight hand piece (Marathon, Taegu, Korea). The metal ceramic samples were ground flat by using the grinder/ polisher device (Mopao, 160E, China). With silicone carbide paper 1200 grit under water cooling<sup>[15]</sup> to obtain standardized smooth surface. Finally, glazing was done for all samples in the conventional method.

### **Preparation of zirconia samples**

Zirconia disc pre- sintered Y-TZP zirconium (IPs e.maxZirCAD, shade MO1, Ivoclarvivadent, Schaan, Liechtenstein) have dimensions (9.5cm diameter x 1.4cm height)

was used in this study. The zirconia disc was cut into many rectangles using cutting saw. Each rectangle was glued into the fitting pin that was placed to a milling machine<sup>[14]</sup> to be milled to the desired dimensions (11.23mm length x 5mm width x 5mm height). The rectangular block of zirconia was divided by using diamond disc with 0.20mm thickness and straight hand piece without water. The straight hand piece was operated at high speed and fixed by holder which moved in horizontal direction toward the zirconia block to make the blade of diamond disc exactly perpendicular on the line drawn on the zirconia block to determine the thickness of the sample which was (5mm).

The samples were sintered in the furnace with high temperature (infire HTC speed sintering furnace, Sirona) at 1500°C 1273°F for 8 hours according to the manufacturer instructions. The dimensions of each sample was (9mm length, 4mm width and 4mm height) due to shrinkage was occurred in zirconia samples during sintering (about 20% shrinkage). (Figure 3).



**Figure 3: Zirconia substructures samples.**

### **Sandblasting Procedure**

The prepared surface for bonding of all samples was sandblasted with 50 $\mu$ m aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles (Korox, Bego, Germany) at air pressure 0.2 MPa for 10 second at a distance about 10mm, after that, all zirconia samples were cleaned in ultra- sonic device (Delta D68H, Taiwan) that contained distilled water for 5 min., then dried.

### **Application the zirliner on zirconia core**

A brush was used for application of Zirliner on the prepared surface for bonding of each sample to form an even layer. Then fired in ceramic furnace (P310, Ivoclar Vivadent, Schaan, Liechtenstein) according to manufacturing instruction.

### Application of veneering ceramic on zirconia samples

A custom- made mold was fabricated from clear acrylic material. This mold consists of two parts connected by four screws and has three holes with larger dimensions (14mm length, 5mm width, and 5mm thickness) to compensate for the shrinkage that would occur during sintering process. Figure 4.



**Figure 4: Build up of ceramic.**

After complete firing, the dimensions of veneering ceramic were checked by digital vernia, and then it was adjusted by using straight hand piece (Marathon, Taegu, Korea) with diamond bur. And finally, the samples were glazed by mixing the glaze powder with corresponding liquid, then the samples were sintered in the oven according to the manufacturing instructions.

### Preparation the artificial saliva with different pH values

Artificial saliva solutions with PH values of 3.5, 7.0 and 10.0 were prepared.<sup>[9]</sup> For the neutral solution (PH 7.0) artificial saliva of the following composition was prepared: 100mL Na<sub>2</sub>HPO<sub>4</sub> (2.4 mM), 100mL of KH<sub>2</sub>PO<sub>4</sub> (2.5mM), 100mL of NaCl (1.0mM), 100mL of KHCO<sub>3</sub> (1.50mM), 100mL of CaCl<sub>2</sub> (1.5mM), 100mL of MgCl<sub>2</sub> (0.15mM), and 6mL of citric acid (0.002mM). The pH of neutral artificial solution was raised to 10.0 by adding NaOH, and lowered to 3.5 by adding HCl. H of the solution was measured by (Etekcity PH-009 digital meter, China).

### Sample grouping

Group M (Metal core): The 30 metal core samples (M) were randomly divided into (3) subgroups, each subgroup had (10) samples divided according to pH used.

- 1- Group M1: samples in neutral solution of pH (7.0) (control group).
- 2- Group M2: samples in acidic solution of pH (3.5).
- 3- Group M3: samples in alkaline solution of pH (10.0).

**Group Zr (Zirconia core):** The (30) zirconia core samples (Zr) were randomly divided into (3) subgroups, each subgroup had (10) samples divided according to pH used.

- 1- Group Zr1: samples in neutral solution of pH (7.0) (control group).
- 2- Group Zr2: samples in acidic solution of pH (3.5).
- 3- Group Zr3: samples in alkaline solution of pH (10.0).

Storage period of samples in solutions.

All Samples of were immersed in different pH solutions separately.

Then, all samples were kept in an incubator (Memmert, 854 Schwabach, Germany) at 37°C for 21days. At the end of period, the samples were washed under tap water to remove any effect of solutions. Then, they were tested with Profilometer device and measurements were recorded in Ra before and after storage.

## RESULTS

**Table 1: Mean, Standard Deviation SD, Minimum, and Maximum of Surface roughness of M1, M2, and M3 before and after storage in Ra. (A after storage, B before storage).**

Group	Mean± SD	Minimum	Maximum
M1B	1.615±0.049	1.54	1.68
M1A	2.645±0.051	2.58	2.72
M2B	1.617±0.067	1.53	1.71
M2A	3.217±0.049	3.14	3.28
M3B	1.607±0.066	1.51	1.69
M3A	2.809±.037	2.75	2.86

**Table 2: Mean, Standard Deviation SD, Minimum, and Maximum of Surface roughness of Zr1, Zr2, and Zr3 before and after storage in Ra. (A after storage, B before storage).**

Group	Mean± SD	Minimum	Maximum
Zr1B	1.637±0.010	1.59	1.67
Zr 1A	2.061±0.041	2.00	2.12
Zr 2B	1.620±0.020	1.59	1.65
Zr 2A	3.343±0.029	3.30	3.39
Zr 3B	1.620±0.020	1.59	1.65
Zr 3A	2.653±0.026	2.61	2.69

**Table 3: Student t-test between M and Zr groups after storage. (A after storage, B before storage).**

groups	t	P-Value	C.S
M1A & Zr1A	20.623	.000	P<0.01 (HS)
M2A & Zr2A	7.269	.000	P<0.01 (HS)
M3A & Zr3A	10.060	.000	P<0.01 (HS)

**Table 4: Student t-test for M groups before and after storage. (A after storage, B before storage).**

groups	t	P-Value	C.S
M1B & M1A	52.255	.000	P<0.01 (HS)
M2B & M2A	95.618	.000	P<0.01 (HS)
M3B & M3A	46.569	.000	P<0.01 (HS)

**Table 5: Student t- test for Zr groups before and after storage. (A after storage, B before storage).**

group	t	P-Value	C.S
Zr1B & Zr1A	29.899	.000	P<0.01 (HS)
Zr2B & Zr2 A	163.156	.000	P<0.01 (HS)
Zr3B & Zr3A	147.571	.000	P<0.01 (HS)

## DISCUSSION

The success of ceramic restorations and the advancements achieved in the development of improved dental ceramic systems have played a role in the increased popularity of the shift towards these systems.<sup>[5,8]</sup> dental ceramics are known for their excellent aesthetic and biocompatibility.<sup>[1]</sup> They have shown the most accurate reproduction of the appearance, color and texture of natural teeth,<sup>[2]</sup> so they are widely used for both anterior and posterior restoration such as inlays, onlays, veneers, metal- ceramic and all- ceramic restorations.<sup>[3]</sup>

However, porcelain fragile structure, the fracture of on the porcelain surface and the degradation of surface finish, as opposed to the attrition on the teeth as well as increased

accumulation of plaque causes.<sup>[16]</sup> Zirconia- based ceramics are among the most popular and the most widely used ceramics in the modern dental practice. This can be attributed to their superior mechanical properties<sup>[7]</sup> and excellent biocompatibility comparable to other dental ceramics.<sup>[8]</sup>

The stability of dental ceramics in the oral environment is directly related to the high surface polish, subcritical crack propagation and chemical inertness of these materials, which in turn enables them to resist degradation in the oral environment.<sup>[5]</sup>

The results of the present study showed that artificial saliva with PH values (3.5, 7.0, and 10.0) significantly effect on the ceramic and caused rough surface. This is clearly shown when comparing subgroups of metal ceramic and zirconia ceramic before and after storage in artificial saliva with different PH values. Regardless the veneering ceramic materials.

In this study two types of ceramic materials with different microstructure, chemical composition and properties. Feldspathic ceramics (VITA VMK Master®) are usually used as a veneering material for metal ceramic restoration and provide excellent esthetic and compressive strength. This type enclose 19 weight percentage (wt. %) of leucite crystals ( $K_2O Al_2O_3 4SiO_2$ ) after incongruent melting of a mixture of glass and potassium feldspar.<sup>[17]</sup> Veneering ceramic for zirconia (IPS e.maxceram Ivoclarvivadent) are composed of dispersed fluorapatite crystals [ $Ca_{10} (PO_4)_6F_2$ ] in a feldspathic glassy matrix without feldspar or leucite.<sup>[18]</sup> These crystals have needles like morphology and very small size in dental microstructures result in very special properties such as translucence and opalescence.<sup>[19]</sup>

Two types of veneering ceramic which is used in this study have a relatively similar behavior of roughness when immersed in neutral pH. Also they are undergoing similar behavior of changes in acid pH or alkaline pH. That is mean (Ra) values in this study not affected by the veneering ceramic brand. These results agree with Kukiattrakoon<sup>[17]</sup>, a study found leucite and flouraptite crystals appeared to have a comparable durability in acidic agent. Also, Milleding<sup>[20]</sup> found similar degradation processes will also occur for many dental ceramic because their microstructure is dominated by a glassy matrix. Another explanation may relate to the effect of surface glaze in both types of ceramic veneering.

When a material is exposed to a biologic environment, the first reactions that take place are confined to the extreme superficial layer of the material.<sup>[21]</sup> The degradation process is

principally controlled by reaction between the primary silica glass network and the surrounding aqueous environment.<sup>[3]</sup> Silicate as a major component of the vitreous phase present in some compositions of alumina, these materials are more susceptible to aqueous environments.

According to freiman's model of the  $H_2O$ -  $Al_2O_3$  reaction, showing three following phases: A  $H_2O$  molecule orients itself in relation to a Al-O-Al bond of the crack tip in such a way that a pair of electrons isolated from the oxygen in the water lines up with the oxygen in the alumina and a hydrogen bond occurs, connecting the oxygen in the alumina with hydrogen in the water.

The transfer of an electron from the oxygen in the water to the alumina occurs simultaneously with the transfer of a proton to the oxygen in the alumina. Two new Al-O and H-O bonds are formed.

Rupture of the weak link of the hydrogen, giving rise to two Al-OH groups.

Another explanation, at neutral pH, a combination of two reaction occur (ion exchange and complete dissolution).<sup>[22]</sup>

### **Acidic PH solution**

The results of this study that showed statistically high significant difference in (Ra) values of metal groups before and after storage in acidic artificial saliva. Also there was found statistically high significant difference in (Ra) values of zirconia groups before and after storage in acidic artificial saliva. In addition the high significant difference was showed between metal group and zirconia group after storage in acidic PH. However, the samples stored in acidic PH (3.5) were representing. The highest (Ra) values compared to neutral and alkaline PH of artificial saliva. In particular, an acidic environment caused by the ingestion of beverages and food with low pH values is predominant.<sup>[23]</sup> In this environment, the mechanism has been explained for degradation process of dental ceramic is the selective leaching of alkaline metal ions.

This mechanism is controlled by the diffusion of hydrogen ions or hydronium ions ( $H_3O^+$ ) from an aqueous solution in to the glass coupled with the leaching of alkali ions from the glass surface to maintain electrical neutrality. Alkaline metal ions are for less stable in the glass than in the crystalline phase. Some alkaline ions are leached after exposure to acidic

solution<sup>[24]</sup> following the leaching out of the more easily released elements such as potassium and sodium; it was highly likely that pores or channels were created within the glass matrix. This then led to increased diffusion of water molecules and subsequent breaking of the Si- O-Si bonds.<sup>[25]</sup>

These acids may affect the elemental dissolution of ceramic due to their chelating effect. Although acetic acid has been found to be a weak organic acid, it will still fairly corrosive to ceramics due to its chelating.<sup>[20]</sup>

In a basic environment, the hydroxyl ions in solution attack the glass structure, breaking silicon- oxygen bonds, thereby resulting in generalized dissolution of glass network. Basic conditions (highly hydroxyl content) will present a more severe environment producing hydrolysis of the silica network and the resulting complete breakdown of the entire structure.<sup>[22]</sup>

Esquivel Upshaw<sup>[22]</sup> stated that the ceramic veneers were susceptible to degradation when in contact with low and high pH solutions, due to an ionic exchange mechanism and this degradation would be clinically significant only after long period use.

The (Ra) results of this study agree with Milleding<sup>[20]</sup>, Milleding<sup>[21]</sup>, Kukiattrakoon<sup>[26]</sup>, Kukiattrakoon<sup>[3]</sup>, and Kukiattrakoon<sup>[17]</sup>, their results showed roughness changes that occurred as a function of time, temperature and pH. Most of these studies were limited to acidic condition.

Also, this study agree with Oh<sup>[27]</sup>, Demirel<sup>[28]</sup>, Ccahuana<sup>[29]</sup>, Matsou<sup>[30]</sup>, Vechiato-Filho<sup>[31]</sup> since their results found that, the immersion of dental ceramics at water, acids and some fluoride solutions degrades their surfaces, sometimes increasing roughness by properties appearance.

There was disagreement with Duymus<sup>[16]</sup>, a study suggested that acidic agents were not statistically affect surface roughness of feldspathic porcelain, but the type of acidic agents were affected surface roughness.

## CONCLUSION

Within the limitation of this *in vitro* study, the following conclusions were drawn:

1. The artificial saliva with different pH values had an effect on surface roughness (Ra) of metal ceramic and zirconia ceramic groups.
2. Both veneering ceramic materials (Vita VMK Master) and (IPS e max ceram) which were used in this study had shown highly significant changes in surface roughness after storage period.
3. The highest surface roughness was found in acidic group followed by alkaline group then neutral group for both metal ceramic and zirconia ceramic group.

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