بِسْلَامِ الرَّحْمَنِ الرَّحِيمِ

قل هل يعتدؤ الذين يعاقبون الذين لا يعاقبون إنما يذكرون أول من أتبت

صلاة الله عليه وسلم

ش鸥 الغانم
EFFECT OF SURFACE MODIFICATION OF YITTRIUM PARTIALLY STABILIZED ZIRCONIA ON SHEAR BOND STRENGTH WITH RESIN CEMENT

Thesis
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Dedication

To the soul of my mother, may she be resting in heaven.
To my beloved father for everything he offered me,
without him i would not be here today
To my precious sisters and brother who are the joy of my life
To every person I meet during this work and gave me the hope and courage to continue all the way long.
The search for esthetics dental restorations is increasingly frequent in daily practice. Due to their high esthetic profile, mechanical properties, chemical stability and biocompatibility; all ceramic restorations have become the focus of dental practitioners, researches and manufacturers.

One of the most commonly used all-ceramic core materials for conventional and resin-bonded fixed partial dentures and complete coverage crowns is yittrium tetragonal zirconia polycrystal (Y-TZP; zirconia). These ceramics with high crystalline content (aluminum and/or zirconium oxides) have been shown to demonstrate clinical success rates higher than or comparable to those of feldspar, leucite and lithium disilicate-based ceramics.

With the development of computer-aided design-computer-aided manufacturing (CAD/CAM) technology, the design of zirconia frameworks could be achieved using a digital process. Therefore, restorations using a zirconia framework become more practical.

However, an important requirement for successful function of these ceramic restorations is adequate adhesion between ceramic and tooth substance. Different cements types (conventional cements, glass ionomer cements, and self-adhesive cements) have been proposed for luting zirconia; however, to ensure retentive and sealed restorations, resin cements are recommended.

Ceramic-resin cement bond may be more effective and durable if associated with micromechanical retentions. The
achievement of roughened ceramic surface may allow resin cements to flow into these micro retentions and create a stronger micromechanical interlock.

Since the development of ruby laser by Maiman in 1960, laser has become widely used in medicine and dentistry. CO$_2$ and Nd:YAG lasers are the most generally used instruments for both intraoral soft tissue surgery and hard tissue applications. Only a few studies have been performed on the laser treatment of zirconium oxide ceramics.

The CO$_2$ laser is well suited for the treatment of ceramic materials because its emission wavelength is almost totally absorbed by the ceramic. Its effect on zirconia-based ceramics has not been well established.

Few studies have evaluated the effect of different surface modifications on the shear bond strength of YZ ceramics to resin luting agent. Against this background, this study was set to evaluate the effect of surface modification on shear bond strength with resin cement.
The introduction of zirconium dioxide or zirconia opened the door for designing fixed all ceramic partial dentures without any limitation regarding the size of the fixed partial denture (1). Its unique qualities, strength, transformation toughening, white color, chemical and structural stability made zirconia the core material of choice (2).

Although many types of zirconia-containing ceramic systems are currently available, only three are used to date in dentistry. These are yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP), magnesium cation-doped partially stabilized zirconia (Mg-PSZ) and zirconia-toughened alumina (ZTA) (3, 4).

Chemical stability and superior physical and mechanical properties have made zirconia-based materials the framework material of choice for fixed dental prostheses. To achieve better esthetics, the zirconia framework is veneered with a ceramic material, which is built in successive layers giving the final restoration individual optical characteristics that can barely be distinguished from the surrounding natural dentition (2-7).

Due to their chemical inertness, zirconia frameworks are resistant to aggressive chemical agents such strong acids as hydrofluoric acid (Derand and Derand, 2000) (8), alkalis, and organic and inorganic dissolving agents. Other techniques which are based on increasing their surface roughness failed to establish a strong and durable bond with adhesive resin cements. Different investigations have examined and measured the shear bond strength of different cements on zirconium. Oxide ceramic surface
after different pre-treatments. These studies provide varying and controversial results \(\text{Derand and Derand, 2000; Wegner and Kern, 2000; Piwowarczyk et al., 2005}^\text{(8, 9, 10)}\)

**The CAD/CAM system**

A major determinant of the quality of fixed prosthodontics is close internal and marginal fit of the crowns. To accomplish that, accurate and precise replicas of the teeth are essential. The manual handling in the fabrication of a crown makes it difficult to identify critical factors and achieve accurate, precise quality control. Because the measurements are taken at a limited number of sites, the object is only partly analyzed for geometrical changes. Uniform irregularities are hard to detect because the measurements are compared without relation to an absolute reference: thus a discrepancy may be detected, but not necessarily correctly located. Surface digitization devices continue to be improved\(^{(11,12)}\). The possibility of digitizing free form objects, such as teeth, allows the accurate measurement of small changes.

Computer aided device (CAD) technology can be used as an evaluation method to analyze geometrical changes. In dentistry, this opens up new opportunities for three-dimensional evaluation of the entire surface, \(\text{e.g. to detect irregularities between the original preparation and the stone replica}^\text{(13, 14)}\). A dental surface digitization device can be based on contact or non-contact methods where three-dimensional images are captured\(^{(15-18)}\).
However, it is important to establish the accuracy and precision of the digitization device \(^{(19-22)}\).

The major developments of dental CAD/CAM systems occurred in the 1980s. **Dr. Duret** was the first to develop dental CAD/CAM1. From 1971, he began to fabricate crowns with an optical impression of abutment followed by designing and milling. Later he developed Sopha system. Dr. Mormann developed CEREC System, an innovative approach to fabricate same day restorations at the chair side in the dental office. \(^{(23)}\) Dr. Anderson developed Procera System\(^{(24)}\). He attempted to fabricate titanium copings by spark erosion and introduced CAD/CAM technology into the process of composite veneered restorations. \(^{(25)}\) This system later developed as a processing center networked with satellite digitizers around the world for the fabrication of all ceramic frameworks \(^{(26,27)}\).

**Surface treatments of ceramics for improving bond strength to resin cement:**

**Sandblasting**

Sandblasting with aluminum oxide particles is a surface treatment option that produces irregularities in acid-resistant ceramics. It relies on blasting the ceramic surface with different particle sizes ranging from 30 to 250 micron. The abrasive process removes loose contaminated layers and the roughened surface provides some degree of mechanical interlocking with the adhesive. It can be argued that the increased roughness also forms a larger surface area for the bond. It may also introduce
some physio-chemical changes that affect surface energy and wettability.\(^{(28)}\)

In 2002 Ozcan & Vallittu\(^{(29)}\), evaluated the effect of three different surface conditioning methods on the bond strength of a Bis-GMA based luting cement to six commercial dental ceramics (leucite reinforced, glass infiltrated alumina, glass infiltrated zirconia), lithium disilicate, high alumina, experimental alumina. The specimens in each group were randomly assigned to one of the following surface treatments: hydrofluoric acid etching, airborne abrasion and silica coating. The shear bond strength of luting cement to ceramics was measured. Hydrofluoric acid gel was effective mostly on ceramics having glassy matrix in their structures. Roughening the ceramic surfaces with airborne particle abrasion provided higher bond strengths for high alumina ceramics.

**Laser surface treatment**

The term **laser** is acronym for **light Amplification by the** Stimulated Emission of **Radiation**. Laser energy is produced when suitable medium (solid, Gas or Liquid) is subjected to certain physical constraints at high energy. The laser radiation has unique properties of being coherent, monochromatic and collimated.

The collimating property results from the parallelism, narrow width, and high intensity of the beam that makes it easy to be delivered via thin flexible fibers and focused to a sharp spot without
a fuzzy peripheral area. This feature makes it ideal for cutting, drilling and welding of different tissues and materials. \(^{(33, 34)}\)

*Andrews in 1986* \(^{(35)}\) added that laser is attributed by its coherence property as all the photons have the same frequency and wave length. While being monochromatic means that all the produced photons have the same color.

**Carbon dioxide laser**

Carbon dioxide laser was first developed by *Putel* and others in 1964. It has a wavelength of 10.6 nanometers deep in the infrared range in the electromagnetic spectrum. It has an affinity and highly absorbed in wet tissue regardless tissue color with a penetration depth of only 0.2 to 0.3 cm. The active medium in CO\(_2\) laser is a mixture of carbon dioxide, nitrogen and helium. \(^{(36)}\)

**Carbon dioxide laser delivery system**

According to *Walsh 1994* \(^{(37)}\), CO\(_2\) laser cannot be transmitted through fiber optics, because of its long wavelength. The only way for the delivery is through articulated arms or hollow wave guides. This lead to a major difficulty in manipulation of the articulated arms. Recently, with the development in optic science, hand pieces are provided with different tips that give more accessibility. Hollow wave guide also provides a good degree of flexibility that allows better access. Because CO\(_2\) laser beam is invisible, a second coincident visible aiming beam (red indicating
marker) usually a He-Ne laser is used so that the operator can determine where the beam will be applied.\(^{(36)}\)

_Stubinger et al. 2008\(^{(39)}\)_ investigated the influence of Er:YAG, CO\(_2\) and Diode laser irradiation on surface properties of zirconia ceramic. Yttria-stabilized tetragonal zirconia polycrystal ceramic discs were irradiated at different power settings with either an Er:YAG, CO\(_2\) or diode laser. The surfaces of the discs were analyzed by scanning electron microscopy (SEM). The SEM analysis demonstrated that, regardless of the power settings, neither the diode laser nor the Er:YAG laser caused any visible surface alterations to zirconia. At various power settings, the CO\(_2\) laser treatment was characterized by material cracking and melting. They concluded that in contrast to diode and Er:YAG laser irradiation, the CO\(_2\) laser revealed distinct surface alterations to the surfaces of the zirconia discs at various laser parameters.

_Ersu et al. 2009\(^{(40)}\)_ compared the effects of CO\(_2\) laser and conventional surface treatments on surface roughness and shear bond strengths of glass-infiltrated alumina-ceramics to dentin. The ceramic discs were randomly assigned to 5 groups, according to the surface treatments applied. Group A (control) received no treatment; Group B: were sandblasted with 50 microns alumina particles; Group C: were airborne particle abraded with 27 microns alumina particles; Group D: ceramic discs were etched with 9.5% hydrofluoric acid; Group L: ceramic discs were irradiated by CO\(_2\) laser. The results showed that all surface treatments tested
produced rougher surfaces in comparison to the untreated groups of all ceramic groups. The shear bond strength was then tested.

Group L had significantly higher bond strengths compared with other surface treatments. They concluded that there is no significant relationship between surface roughness (Ra) and shear bond strength values (MPa) among ceramic groups. The surfaces irradiated with CO$_2$ laser showed crater-like irregularities in and around glazed patches, which may provide mechanical retention.

Ural et al. 2010\(^{41}\) evaluated and compare the effect of different surface treatments and laser irradiation on the shear bond strength of resin cement to zirconia based ceramics. Forty zirconia specimens were produced. Specimens were divided into four groups according to the surface treatment method. Group C; no treatment; Group SB, discs were airborne abraded with 110µm $\text{ALO}_2$; Group HF discs were etched with hydrofluoric acid; and Group L, were irradiated by CO$_2$ laser. A total of forty composite resin disks were fabricated and cemented with adhesive resin cement to the specimens. A universal machine was used for the shear bond strength test. The highest shear bond strength value were obtained with laser group (20.99 ± 3.77MPa) and the lowest values for the no treatment group (13.39 ±3.10MPa). It was concluded that CO$_2$ laser etching may represents an effective method for conditioning zirconia surfaces, enhancing micromechanical retention and improving the bond strength of resin cement to zirconia ceramic.
Ural et al. 2011 (42), evaluated the influence of different power outputs of a carbon dioxide (CO₂) laser on shear bond strength of resin cement to zirconium dioxide-based ceramic. Fifty zirconium dioxide core specimens were embedded in the centers of auto-polymerizing acrylic resin blocks. Ten specimens served as control and no surface treatment was applied and ten specimens for surface treatment with CO₂ laser with different output power; laser treatment with 2 W (Group 2 W), 3 W (Group 3 W), 4 W (Group 4 W) and finally 5 W (Group 5 W). Composite resin discs were fabricated and cemented with adhesive resin cement to the specimen surfaces. A universal test machine was used for shear bond strength test. Data were statistically analyzed by one-way analyses of variance (ANOVA) with Post-Hoc Tukey tests (α = 0.05). It was found that the shear bond strength values were affected by power outputs of laser (p < 0.05). Highest shear bond strength values were obtained with group 2 W and lowest values were obtained with group 5 W. The study revealed that there was a relationship between laser output power and shear bond strength for zirconium dioxide ceramics. However, output power of the laser and the energy level is a critical factor on micromechanical retention.

**Effect of adhesive Resin Cement on the shear bond strength**

A durable bond depends on the chemical composition of the adhesive agent and the surfaces that are connected. Dental luting cements form the link between a fixed restoration and the supporting tooth structure. (43)
The adhesive capacity and the cement stiffness are important material properties for the longevity of all-ceramic restorations. Regarding the bonding mechanism between composite resin and ceramics, hydrofluoric acid etching and silanization can enhance the mechanical bond strength of glass-ceramic materials to composite resin.\(^{(44, 45)}\)

Resin cements have been selected for their advantageous mechanical and adhesive properties compared with conventional luting cements. The clinical application of resin-bonded fixed restorations requires a strong and stable resin bond to the ceramic.\(^{(46, 47)}\)

Resin cements are increasingly used for luting all-ceramic, metal or composite indirect restorations due to their excellent mechanical properties, better bond strengths and improved esthetics when compared to conventional cements.\(^{(43, 49)}\)

_Ygili and Sahmali 2003\(^{(49)}\)_ evaluated the bond strengths of two resin luting cements (Panavia F and Clearfil Se Bond) to different all ceramic materials (In-Ceram and IPS-Empress). Four ceramic surface treatments were performed. (1) As received, (2) grinding with diamond bur, (3) sandblasting with 50 μm alumina grit and (4) HF acid treatment and sandblasting with 50 μm alumina grit. They concluded that the bond strengths for Panavia F was quite low but suitable for bonding In-Ceram and IPS-Empress material. Surface treatments such as acid etching or sandblasting had major influence on bond strengths.
The bond strength of five different commercial luting cements (Ketac Cem, Rely X, Fuji Plus, Panavia F and Xeno Cem) to the ceramic material were evaluated by Begazo et al. 2004, they concluded that the uses of resin composite based cements are preferred for cementation of an all-ceramic restoration with an aluminum oxide-reinforced glass ceramic base. The shear bond strength increased significantly for Ketac Cem, Rely X, Fuji Plus, Panavia F to Xeno Cem.

Wolfart et al. 2007 evaluated the bond strength and the durability of two composite resins to zirconia ceramic after using different surface conditioning methods. Groups of 20 specimens each were bonded either with a conventional composite resin (Variolink II) or with a phosphate monomer (MDP)-containing resin (Panavia F) to the ceramic discs. After 150 days storage, only the air abraded specimens bonded with Panavia F showed high bond strengths of 39.2MPa, whereas most other specimens debonded spontaneously or showed very low bond strengths. The use of the MDP-containing composite resin Panavia F on air abraded zirconia ceramic can be recommended as promising bonding method.

The effect of different surface treatments on bond strength between In-Ceram Zirconia ceramic and Panavia F Cement was evaluated by Spohr et al. in 2007. In-Ceram Zirconia received three different surface treatments, a group was treated with Sandblasting by alumina particles and silane; another group was treated with Silica coating by Rocatec System, In the last group the samples were treated by Sandblasting, Nd:YAG laser and silane. Microtensile bond strength was tested. The results showed that the
highest bond strength was recorded for the Nd:YAG laser surface treatment (18.7 MPa), followed by the Rocataec System (15.75 MPa), and alumina sandblasting (11.81 MPa). They concluded that Nd:YAG laser irradiation is an effective surface treatment for bonding between In-Ceram Zirconia and Panavia Fluoro Cement.

Re D et al. 2008(53), evaluated the shear bond strength of two resin cements to two types of zirconia ceramics after three surface treatment methods. (LAVA ceramic samples) and (Cercon ceramic samples) were divided according to surface treatments into: no treatment, sandblasting with 50 micron alumina particles, sandblasting with 110 micron alumina particles, silica coating with Rocatec System. Two resin cements were used RelyX Unicem and Panavia F2.0. Shear bond Strength was tested. In the lava specimens, the three surface treatment methods tested did not increase the bond strength of Panavia compared to control group. For Lava surfaces treated with Rocatec, there was a statistically significant difference between the two luting cements used (Rely X Unicem recorded 11.39 MPa while Panavia recorded 8.56 MPa). Regarding the Cercon specimens, there was a significant difference between the Unicem and the Panavia control groups (Unicem recorded 1.48 MPa and Panavia recorded 4.60 MPa). In conclusion, all surface treatments increased the bond strength of RelyX Unicem resin cement to both zirconia substrates. No statistically significant changes were found using Panavia on Lava. Sandblasting with 110 micron Al₂O₃ provided the highest bond strength for Panavia on Cercon.
De oyague et al. 2009 (54), evaluated the effect of surface conditioning on the microtensile bond strength of zirconium-oxide ceramic to dual-cured resin cements. Zirconium oxide ceramic blocks were treated with: sandblasting, silica coating a group with no treatment. The results showed that the bond strength were significantly influenced by the luting agent but not by the surface treatment, Bond strength of Clearfil cement to zirconia with the three surface treatments (sandblasting, silica coating, No treatment) respectively was higher than that of RelyX Unicem and Calibra regardless of the surface treatment performed on the ceramic surface. The phosphate monomer-containing luting system (Cleat’Fil Esthetic Cement) is recommended to bond zirconia ceramics and the surface treatments are not necessary. They concluded that Luting cement selection seems to be a more relevant factor when bonding to zirconia ceramics.

Phark et al. 2009 (55), evaluated the shear bond strength of composite resin cement to a zirconia surface using different luting techniques. Procera Zirconia with a modified surface and a machined surface were used. Three different adhesive luting cements (Panavia F2.0, Rely X ARC, Rely X Unicem) in combination with and without airborne particle abrasion. The highest value for the shear bond strength was recorded for the untreated modified zirconia cemented with Panavia F2.0 (20.01 MPa) and for the machined zirconia treated with 50 micron airborne-particle abrasion cemented with Panavia F2.0 (18.51 MPa). They concluded that Shear Bond Strength to the modified zirconia surface is higher than to airborne-particle abraded or
machined zirconia. Airborne particle abrasion of the modified zirconia surface is not recommended.

Akyil et al. 2010\textsuperscript{(56)}, evaluated the shear bond strength of a resin cement to yttrium-stabilized tetragonal zirconia (Y-TZP) surfaces treated with air abrasion, silica coating, or CO\textsubscript{2}, Er:YAG, or Nd:YAG laser irradiation, or irradiated by each laser after air abrasion. The shear bond strength test was performed. The highest bond strength was obtained in the air abraded group. Although air abrasion and silica coating were the most effective surface treatment methods, CO\textsubscript{2} and Er:YAG laser irradiation alone or Nd:YAG laser irradiation after air abrasion may be used as an alternative treatment method to increase the bond strength between resin cement and Y-TZP material.