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THE EFFECT OF DIFFERENT SURFACE TREATMENT AND Ceramic PRIMERS ON SHEAR BOND STRENGTH OF SELF ADHESIVE RESIN CEMENT TO ZIRCONIA CERAMIC AFTER THERMOCYCLING

MASTER THESIS

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ABBREVIATION AND SYMBOLS

FPD: Fixed Partial Denture

ZR: Zirconia Ceramic

YTZ: Yttrium Stabilized Zirconia

CCP: Clearfil Ceramic Primer

ZPP: Z-Prime Plus Primer

HF: Hydrofluoric Acid

APA=SB: Airborne Particles Abrasion= Sandblasting

MDP: 10-Methacryloxydecyl Dihydrogen Phosphate

CAD-CAM: Computer-Aided Design and Computer-Aided Manufacturing

µm: Micrometer Unite

SBS: Shear Bond Strength

N: Newton Unite of Force

ANOVA: Analysis of Variance

Tukey HSD: Tukey Method, Honest Significant Difference Test

P: P Value (Calculated Probability)

etal: And Others

ABSTRACT

Purpose: The purpose of our in vitro study was to evaluate the effect of different surface treatment and ceramic primers on the shear bond strength (SBS) of self-adhesive resin cement to Zirconia ceramic after thermal cycling.

Materials and methods: Forty discs shaped Zirconia specimens (n=40) were milled from pre-sintered Zirconia blocks. The final diameter of the Zirconia discs was 10mm and the thickness was 3mm. The surface of Zirconia specimens were polished with silicon carbide paper number 1200, 800, 600grit respectively under cooling water then cleaned by distilled water in ultrasonic machine. All specimens divided according to the type of surface treatment used into two main groups. Group1: twenty (n=20) Zirconia specimens conditioning with airborne particles abrasion (sandblasting) with Alumna particles size 70$\mathcal{M}$m for 20 seconds. Group2: twenty (n=20) Zirconia specimens conditioning with Hydrofluoric acid etchant (9.5%) for 90 seconds. The each main group subdivided according to the type of primer used into two subgroups. Group1:Clearfil ceramic primers (Kuraray) were applied to 10 sandblasted Zirconia specimens. Group2: Z-PRIME PLUS primer (Bisco) were applied to 10 sandblasted Zirconia specimens. Group3: Clearfil Ceramic primers were applied to Hydrofluoric acid etched Zirconia specimens. Group4: Z-PRIME PLUS primer were applied to Hydrofluoric acid etched Zirconia specimens. After that, the Plexiglas mold 3mm in height and 4mm in diameter was used for placement of self-adhesive resin cement (Multilink Ivoclar Viva Dent) on the center of Zirconia surface. The resin cement light cured for 20 seconds then removed the Plexiglas mold and all Zirconia specimens thermocycled (5000 cycles at 5^0c-55^0c with a 30 seconds dwell time for each temperature), after that the specimens were tested in a Universal testing machine at the crosshead speed of 0.5mm/min in shear mode. Data (Mpa) were analyzing using one way ANOVA and Post Hoc Tukey ($p<0.05\%$)

Results: The one-way ANOVA test was used in the statistical analysis. The SBS values vary according to type of surface treatment and primer used ($p< 0.05$). The highest SBS was recorded for Group 4: Sandblasting / Z-Prime Plus. The lowest SBS was recorded for Group 5: Hydrofluoric acid / Clearfil Ceramic Primer.

Conclusions: From the present study we concluded that the airborne particles abrasion is the surface treatment choice of Zirconia ceramic. The Z-PRIME PLUS primer after sandblasting significantly improve the shear bond strength of self-adhesive resin cement to Zirconia.

Keywords: Zirconia, sandblasting, Shear bond strength, Hydrofluoric acid, ceramic primer, thermal cycling.

1. INTRODUCTION AND AIM

In the past the fixed dental prosthesis has been made from metal or metal covered by ceramic [93]. This prosthesis adhered to prepared tooth structure by traditional water based cements such as Zinc phosphate, glass ionomer cement or by oil based cements [93]. After that, the all ceramic restorations have been invented [93]. The all ceramic restoration has many good features such as wear resistance, perfect esthetic, biocompatible and adequate strength [95]. In last years the computer aided design/ computer aided manufacturing technique(CAD/CAM) becomes more popular because it need short time for fabrication of restoration with high esthetic characteristic and more accuracy than conventional restoration[94]. The ceramic used in dentistry may be classified into silica based ceramic such as Feldspathic, Lithium Dissilicate and Leucite reinforced dental cement, or high strength ceramic such as Zirconia and Alumina [93]. The Zirconia ceramic is polycrystalline ceramic, it has three phases which is monoclinic, tetragonal and cubic [95]. The pure Zirconia is monoclinic phase, when add some oxide to pure Zirconia it changed to Stabilized Zirconia [95]. The Zirconia ceramic used in dentistry is yttrium-tetragonal Zirconia polycrystals [95]. The Y- TZP produce high fracture toughness (5-9 MPa-m$^{1/2}$) and high flexural strength (900-1200MPa) three times more than lithium disilicate ceramic [95]. The all ceramic restoration adhered to prepared tooth by adhesive cements, these cement contain light activator or chemical initiator or both [93].The mechanism of attachment between restoration and the prepared tooth maybe mechanical attachment or micro mechanical attachment or chemical attachment or combination of them [94].

The aim of our study was to evaluate the effect of different types of ceramic surface treatment such as airborne particles abrasion and Hydrofluoric acid etching and the effect of ceramic primers that contain different functional groups such as Clearfil ceramic primer and Z-PRIME plus primer on the shear bond strength of self-adhesive resin cement to the Zirconia surface.

2. REVIEW OF LITERATURES

The loss of human teeth not only related to age, but also related to many other factors such as periodontal disease which mainly caused by insufficient care of oral cavity(poor oral hygiene), dental caries or due to systemic disease such as diabetes [24, 27].

The prosthodontics have been considered as one of the most important branch in dentistry [1], that always developed to find suitable treatment that used to restore the decayed teeth and compensate the missing teeth that which will maintain the oral function (mastication and speech), esthetic appearance of the patient [1, 25].

Prosthodontics Classified Into Four Major Categories:

1) Removable prosthodontics.
2) Fixed prosthodontics.
3) Maxillofacial prosthodontics.
4) Implant dentistry.

2.1. Fixed Prosthodontics

The fixed restoration is a prosthesis that cannot be removed by the patient himself. Because it adhered to natural abutment teeth or implant by permanent luting cements [1, 25]. The fixed dental prosthesis may be one unit (crown) to restore badly decayed tooth or endodontic treated tooth and may be constructed from many units (bridge) to replace the missing teeth to improve the function of mastication [25].

2.1.1. Classification of Fixed Partial Dentures (FPD)

Fixed partial dentures classified into three classes, these classes give information about the place of edentulous area [1].

i. **Class I:**

Posterior edentulous area occurred as a result of loss of one or some of the posterior teeth.

ii. **Class II:**

Anterior edentulous area occurred as a result of loss of one or some anterior teeth.

iii. **Class III:**

Antero posterior area occurred as a result of loss of one or some anterior and posterior teeth.

These classes are divided into three divisions, these divisions give information about which teeth have given support to fix partial denture [1].

a) Division 1: Cantilever FPDs abutment teeth found on one side of edentulous area**.**

b) Division 2: Conventional FPDs. Abutment teeth found on both side of edentulous area.

c) Division 3: Abutment teeth found among edentulous area which called pier abutment.

2.1.2 Types of Fixed Partial Denture

Table 1: Types of Fixed Prosthesis

1. Classification according to the type of connector	• Fixed fixed partial denture • Fixed movable partial denture • Removable fixed partial denture
2. According to type material	• All metal crowns • Metal ceramic crowns • All ceramic crowns • All acrylic crowns • Ceramic veneer • Acrylic veneer
3. According to the length of the span	• Short span bridges • Long span bridges
4. According to duration of use.	• Permanent fixed partial dentures. • Long span bridges(interim prosthesis, Maryland bridge and splint)
5. Type of abutment	• Ideal or normal abutment • Cantilever abutment • Pier abutment • Endodontically treated abutment • Mesialy tilted abutment
6. Bridge which require minimal preparation.	• Micro retention • Macro retention

2.1.3 Component of Fixed Partial Denture

The fixed partial dentures consist of retainers that have been used to obtain support and stability from abutment teeth [1]. According to the tooth coverage the retainer can be classified into full coverage retainer, partial coverage retainer and conservative retainer, and according to the material used it has four types (all metal retainers, metal ceramic retainers, all ceramic retainers and all acrylic retainers) [1].The amount of tooth preparation for placement the retainers different according to the type of retainers used. The second component of FPD is pontic that used to replace the missing teeth and restore function [1], it should be biocompatible to adjacent soft tissues, comfortable to the patient and provide good esthetic appearance, it can be made from porcelain or acrylic[1]. The last component is connectors that is used to connect the parts of FPD (retainers-pontic)[1]. The connector may be rigid which used in fixed-fixed partial dentures to transfer the load on the pontic directly to the abutment, or non-rigid connectors that used in non-parallel abutment which cannot produce single path of insertion [1, 28]. Therefore, the non-rigid connectors will provide some movements between retainers and pontic [1, 28].

2.2. Removable Prosthodontic

The removable prosthesis have been fabricated to compensate the missing teeth and surrounding tooth structures [1]. It less effective prosthodontics treatment to compensate the missing teeth, but easy in construct with low cost when compared to fixed prosthetic restoration [1,26].It take the support and stability from abutment teeth and residual alveolar ridge in the removable partial dentures or only from residual ridge and covering soft tissues in complete removable dentures [26].

There are four types of removable dentures

- Complete removable dentures
- Partial removable dentures
- Immediate dentures
- Overdentures **[1]**

2.2.1. Complete Removable Denture

Complete removable dentures divided into two main types **[1].**

2.2.1.1 Conventional Complete Removable Denture

Dental prosthesis that replace all missing teeth and surrounding tissues to restore function of mastication, speech and compensate vertical dimensions and provide good esthetic appearance of the patient [1,26].

2.2.1.2. Special Complete Removable Denture

Some clinical cases need conventional complete denture with some modification. These modifications can be done to improve the comfortable, retention and suitability to the patient. The special complete denture classified into four types [1].

2.2.1.2.1Conventional Complete Denture with Mechanical Retentive Component

In this type of special denture of mechanical components such as springs, intra-mucosal magnets and suction discs are used to increase stability and retention of denture [1]

2.2.1.2.2. Single Complete Denture

Single complete denture which divided into [1];

- ✓ Maxillary complete denture facing mandibular natural teeth
- ✓ Maxillary complete denture facing mandibular partial denture
- ✓ Mandibular complete denture facing maxillary natural teeth
- ✓ Mandibular complete denture facing maxillary partial denture

2.2.1.2.3. Immediate Denture

Immediate dentures can be partial or complete denture fabricated before extraction of teeth and placed in patient mouth directly after extraction of natural teeth [1, 29, and 2]. It used for maintain the esthetic appearance and mastication of the patient, and used as splint to inhibit bleeding and protects the injury [2, 29]. The most important disadvantage of this type was decreased retention of denture in order to make rapid changes in alveolar bone and tissues [2, 29].

2.2.1.2.4 Overdenture

Overdenture prosthesis is more comfortable for the patient when compared to complete denture [1, 2]. It has perfect retention and stability and enhances maintenance of alveolar bone by tensile motivation of periodontal ligament [2]. Overdenture has two types [1, 29].

- Tooth supported over denture.
- Implant supported over denture.

2.3. Removable Partial Dentures (RPD)

The RPD fabricated to replace some missing teeth [1, 26, and 29]. It can be extra coronal partial dentures that take the retention and stability from external attachments that surrounding the abutment teeth [1], or may be intra coronal partial dentures that used internal retentive parts to increase retention and stability [1]. The RPD contraindicated in patient with large tongue because it will effect on the stability of denture [23]. The main components of removable partial dentures are major and minor connectors, direct and indirect retainers, rest and artificial teeth [1, 26].

2.4. Maxillofacial Prosthodontics (MFP)

The maxillofacial prosthetics have been developed to fabrication of functional and cosmetic restorations for oral and facial defects [1]. These defects may be due to trauma, congenital defect or developmental defect. It can be extra oral or intra oral according to the site of defect [1].

2.5. Implant Dentistry (ID)

Implants are prosthesis that insert deeply into alveolar bone to support a crown, bridges or removable prosthesis [1]. The implants can prevent the residual alveolar bone resorption, restore oral function and provide excellent esthetic like natural teeth [1]. They are more comfortable and stable more than other prosthetic restorations [1].

2.6. Dental Cements

Dental cements are substrate that formed due to the reaction between acidic liquid and basic powder (5, 3, and 4). The result of this reaction is a past with different viscosity according to powder liquid ratio (5, 4). The appropriate selections of dental cements are considered as one of the most important steps related to long term stand of fixed prosthesis (3, 4). Therefore the choice of appropriate cement is not only depend on the knowledge of the physical and biological properties but also depend on a dentist experience and preferences (7, 5, 3, and 4).

2.6.1. Classification of Dental Cement

Dental cement classified into tree main categories (5, 90):

2.6.1.1. Liner and Bases Dental Cements

The cement bases are materials placed under permanent restoration to protect pulp from thermal and chemical irritation (3, 4). It thicker than liner (1-2mm)(3). The most common cement base used is resin modified glass ionomer cement (5, 3). Because the RMGI (Resin Modified Glass Ionomer) easy to use, setting fast when exposed to light cure and can etch it to increase adhesive bond with dentin bonding agent (5). But cement liner is material used under cement bases to protect pulp from chemical irritation for example use calcium hydroxide as liner under glass ionomer base to protect the pulp (3,4). The most common liner cement is calcium hydroxide. Because it has antimicrobial action and has possibility to form secondary dentin when used as direct and indirect pulp cupping (3, 4). There are another type of cavity liner such as ZOE (zinc oxide eugenol), Glass ionomer, Resin cement and MTA (mineral trioxide aggregate) (4)

2.6.1.2. Temporary (Provisional) Cements

Temporary cements used to cavity for short period before placement of permanent or final restoration [3,4]. There are many type of temporary cement such as eugenol, non-eugenol, resin or polycarboxylate based [5]. The eugenol containing cement contraindicated to use before permanent resin composite because remnant eugenol may inhibit the polymerization of resin composite and decrease bond strength of adhesive systems to dentin [3, 5, 8, 9, 10, and 11].

2.6.1.3. Luting (Permanent) Cements

Luting cement is substrate that used to attach dental prosthesis to prepared teeth or dental implant and to occupy marginal voids.

2.6.1.3.1. Classification of luting cements

Table 2: Classification of Luting Cements

1. Classification according to chief ingredients(6,12)	▪ Zinc phosphate ▪ Zinc silicophosphate ▪ Zinc oxide_eugenol ▪ Zinc polyacrylate ▪ Glass ionomer ▪ Resin
2. According to matrix bond type(6,13)	▪ Phosphate ▪ Phenolate ▪ Polycarboxylate ▪ Resin ▪ Resin modified glass ionomer
3. According on knowledge and experience of use(6,14)	▪ Conventional(zincphosphate,polycarboxylate,glass ionomers) ▪ Contemporary (resin modified glass ionomers, resin)
4. According to the principle setting reaction(6,15)	▪ Acid base cement. ▪ Polymerization cements.

2.6.1.3.2. Requirements of Luting Cements

Luting cements must be having strong adhesion to prepared tooth and restoration (4, 6, 16) biological compatible with dental pulp (4, 5, 6, and 16). It must be is insoluble or low soluble in oral fluid (6, 16).It should has sufficient working time especially when used to cement long span bridge (6,16).Luting cements must have low film thickness to allow crown to seat adequately (13, 16, 6).Must be translucent and not opaque[6,16].Luting cements should have a proper viscosity which allows it to flow easily along the interfaces between tooth and fixed prosthesis [6, 13, 16].The excess cement must be easy removal to prevent tissues irritation [6, 16]. It should has high compressive, tensile strength and high modulus of elasticity to resist the force of mastication [6, 16].It must be contain fluoride ions which taken up by the underlying tooth to decrease susceptibility of caries [6, 16].

2.6.2.Resin Cements

Resin cement was first introduced in the 1980[86, 87, and 90]. It contains polymer and filler, the filler added to decrease the coefficient thermal expansion and water sorption that lead to increase the strength of polymer [87, 90, 85, and 91]. The resin cement indicated for most prosthetic prosthesis such as Ceramic crown, porcelain veneers, resin and ceramic onlay and inlay [85, 86, 87, 88, 89, and 90]. The resin cements may classified according to the polymerization into light cure, auto or dual cure and chemical cure, and may be classified according to bonding of mechanism into total etch, self-etch and self-adhesive [91, 85, 86, 87, 88, and 89]. The resin cement forms chemical bond to etched and silane conditioned porcelain, therefore, it considered as the best cement used for cementation of all ceramic prosthesis [85, 88]. The resin cements have many advantages such as low solubility in water and oral fluid, good esthetic, adequate compressive and tensile strength, fluoride release and pulpal compatibility[85, 86, 87, 88, 89, 90, and 91].

2.6.3. Self-Adhesive Resin Cements

Self-adhesive resin cement is the latest types of resin cements [92, 87]. It takes less time to use, because the application of self-adhesive resin cements have been done in one step [92, 87]. The main advantages of self-adhesive resin cements are can provide chemical bond to tooth and restoration that will increase the retention and durability of the restorations; decrease postoperative sensitivity and contamination when compared to total etch resin cements [92]. The bond strength of self-adhesive resin cement to Zirconia surface is similar to the bond strength of self-adhesive resin cement to tooth surface [91].

2.6.3.1. Mechanism of Action of Self-Adhesive Resin Cements

The self-adhesive resin cement contains organic matrix and filler with acidic monomers [86, 85, 92]. The low PH of acidic monomers can etch the tooth surface[92], but it etch the dentine more easily than enamel, therefore, the bond strength of self-adhesive resin to dentine is more than to enamel[92, 87]. The acidic monomers continue to etch the dentine for long times which lead to penetration of cement to etched tooth surface [92]. The cement after polymerization provides micromechanical bond to enamel and dentine [92].

2.7. Classification of Dental Ceramics

2.7.1. Feldspathic Porcelain

This type of ceramic can be used for metal or metal free ceramic system [33, 30, 31]. The Feldspathic porcelain consist of Potassium feldspar which form the leucite crystals (crystalline phase) that increase the intrinsic strength of feldspathic porcelain [33, 34, 30]. And consist of Quartz, Kaolin mixed with oxides (Sodium, Potassium, Calcium, Aluminum and Magnesium)[33, 30, 37]. These oxides effect on coefficient of ceramic expansion. In addition to some pigments such as zinc, iron, copper, titanium, nickel, manganese and cobalt then add of opacifiers as tin, zirconium and titanium [33, 30].The feldspathic porcelain has been considered as highly brittle material [38].

2.7.2. Synthetic Glass Matrix Ceramics

2.7.2.1. Leucite Reinforced Glass Ceramics

The Leucite particles are added to ceramic to make some modification on the coefficient of thermal expansion [30, 31, 32]. The added leucite mainly indicated for ceramic fused to metal [31, 32, 30]. The flexural strength of leucite reinforced ceramic (RLC) is depend on the volume (vol) of leucite particle [30]. Therefore the recent type of RLC use more fine leucite particles (10Mmto 20Mm) which will made the materials have high flexural strength, less abrasive and decreased crack propagation [30, 31, 32]. But the LRC has inadequate strength for all ceramic restoration [30]. Pinto et al. evaluated the effect of pH on LRC. He found the acidic pH cause reduction of all strength of Leucite Reinforced Glass Ceramic [33].

2.7.2.2. Lithium Disilicate Glass Ceramic

The lithium Disilicate is heat pressed ceramic [38, 32]. Crystalline phase of this type consist of lithium Disilicate ($Li_2Si_2O_5$) approximately %70 and lithium orthophosphate (Li_3Po_4) [30, 31]. The $Li_2Si_2O_5$ consist of small plate like crystals, these crystals improve the strength of ceramic [30, 31, 32]. Because the plate shape of the particles will deflect crack and stopped the propagation of it [30, 31, 32]. The Lithium Disilicate Glass Ceramics have high fracture toughness, high flexural strength 350-450Mpa more than Leucite Reinforced Glass Ceramic and high esthetic properties and adequate translucency [30, 31, 32]. Therefore it can be used in all ceramic restorations [30].

2.7.2.3. Fluoroapatite Synthetic Glass Matrix Ceramic

This type is mainly contains potassium, sodium and aluminum oxides with silicon dioxide combined with apatite and Leucite to improve thermal expansion and strength when used with metal [36, 30]. The Fluoroapatite crystals improve the optical properties of porcelain veneers and remain the porcelain translucent even when have high crystalline content [30]. So can be used when needed high esthetic ceramic [30, 31, 32]. The amount and shape of crystals effect on flexural strength. So it has high flexural strength approximately 360Mpa [30].

2.7.3. Glass Infiltrated Alumina

Aluminous porcelain contains %35 vol of Alumina (Al_3O_3)[30]. When it used for fabrication of all ceramic restorations mainly contain core ceramic with a thin Platinum foil [30]. Al_3O_3increased used in last year because it considered as hardest and strongest oxides [30, 33, 34]. The flexural strength of Al_3O_3 is 700Mpa [30] and it have high fracture toughness 3.5 to 4 MPa [34].

2.7.3.1.Glass Infiltrated Alumina and Magnesium

This type indicated in anterior teeth [34, 30]. Because it considered as the most translucent ceramic material and it has very high strength [30].

2.7.3.2. Glass Infiltrated Alumina and Zirconia

It available as block form need different machine to produce milled restorations. Alumina and Zirconia matrix has very high strength and has low translucent properties [36, 30]. Therefore it mainly used for three unit posterior bridges [36].

2.7.4. Resin Matrix Ceramic

Resin matrix ceramic defined as polymer matrices which mainly contain inorganic compounds (>%50 by weight)[36]. That includes glasses, ceramic, glass ceramic and porcelain [37]. There are three types of resin matrix ceramic(1) Resin nano ceramic, (2) Glass ceramic in a resin interpenetrating matrix, the second type it considered as hybrid ceramic and It consists of a Feldspathic Ceramic network and polymer network (Triethylene Glycol Dimethacrlate) and (3) Zirconia silica ceramic in resin interpenetrated matrix. The inorganic compound of last type comprises %60, It consists from %85 ultra-zirconia silica ceramic particles mixed with polymer matrix of bisphenol A Glycidyl Methacrylate [36]. After that, ceramic materials are developed to produce material with modulus of elasticity very similar to modulus of elasticity of dentin. And to obtain material easy to mill and adjust and can be repaired by composite resin [36].

2.7.5 Polycrystalline Ceramic

Polycrystalline ceramic is nonmetallic inorganic ceramic materials that do not contain any glass phase [37, 36, 30, 31, 32].The best advantage of polycrystalline ceramics is have a fine grain crystalline structure [36]. These crystalline providing strong fracture toughness and high strength [36, 32], but become less translucent [36]. The polycrystalline ceramics are more resistance to acid due to absence of glass phases [36]. Therefore, when etched with hydrofluoric acid it needs more time or high temperature [36]. The first polycrystalline ceramic introduced in dentistry was Procera All Ceram with strength 600 MPa [30, 31].

2.7.5.1. Alumina

The pure crystalline ceramic used until now in dentistry is alumina oxide (Al_2o_3) [30]. This is first introduced in 1990s by Nobel Biocere [36]. The Alumina polycrystalline ceramics have very high hardness17 to 20 Gpa and high strength and highest modulus of elasticity when compared with other types of ceramic (E=300-350Gpa) [36, 34, 33, 37].

2.7.5.2. Stabilized Zirconia

The monoclinic zirconia is stable up to 1.117 C^0but when the temperature increase to 2.370 C^0 the monoclinic zirconia transform to tetragonal and then cubic form [36, 82, 84]. The tetragonal or cubic phases must be stabilized at room temperature by add some oxides to pure zirconia such as Yttrium, Magnesium, Calcium and cerium this procedure will lead to the maintaining of cubic phases which is needed to increase fracture toughness of the material [36, 30]. The zirconia can be classified according to microstructure in to fully stabilized zirconia (FSZ), which contain %8 mol Yttrium oxide, partially stabilized zirconia (PSZ)that is mostly contain yttria or ceria, and tetragonal zirconia polycrystals (TZP). The dental zirconia is TZP, and the most common Yittra tetragonal zirconia polycrystals (Y-TZP) it has the highest strength and fracture toughness after maching and sintering [36].

2.7.5.3 Zirconia-Toughened Alumina and Alumina-Toughened Zirconia (ZTA / ATZ)

In 1976, Claussen reported that when the unstabilized zirconia (Zr) added to Alumina (AL) will increased the fracture toughness of alumina [36]. The ZTA should have %50by weight AL and ATZ should have %50by weight Zr[36]. The composite consist of different percentage of (AL&Zr) that percentage of alumina and zirconia can be changed according to demand or manufacturer[s] manipulation [36]. The advantage of this composite when compared to Y-TZP are resistance to low temperature degradation, fracture toughness, and higher strength, and more twice Y-TZP cyclic fatigue strength [36]

2.8. Phases of Zirconia

The Zirconia have three shape; the first shape is monoclinic structure that represent the pure Zirconia which stable and not change until 1170 C^0, the second phase is Tetragonal Zirconia that produced from 1170 C^0 to 2370 C^0 and at temperature over 2370C^0 until the melting point 2680C^0 Cubic Zirconia is formed. The transformation from Tetragonal phase to monoclinic phase is combined by %4-5 volume expansion that leads to increase the compressive stresses in the material [(82, 84)].

2.9. Mechanical Properties of Zirconia

The Zirconia has mechanical properties higher than all ceramics used in dentistry. It has excellent fracture toughness (6-10 Mpa), good esthetic, biocompatibility and cause wear of opposite natural teeth less than porcelain, it has highest flexural strength more than all types of ceramic (900Mpa)[(83,82, 30)]. The Zirconia restorations have high marginal fit and low chipping fracture failure especially when it constructed in CAD/CAM machine [(83, 81)].

2.10. Computer-Aided Design/Computer-Aided Manufacturing (Cad-Cam)

CAD/CAM has been first introduced in 1971 by Dr. Duret [(39)]. The CAD/CAM system is digital system allows the dentist to collect all date about the prepared abutment teeth from digital impression taken by intra oral scanner or 3D printer [(39, 40)]. After that send the digital impression in to laboratory (lab) to heat the ceramic in milling machine for fabrication of the dental restoration with more accuracy and more marginal adaptation with save time better than conventional restoration [(39)]. The CEREC is the first commercially CAD/CAM system which was invented in 1985 by Mormann. After that Dr.Andersson developed Procera system which worked on titanium, he used titanium instead of nickel chromium because increase in the number of allergic cases of this substance [(39)].

2.10.1. Types of CAD/CAM Fabrication

2.10.1.1. Chair Side Fabrication

This is the fastest system to save time. Because the impression of prepared tooth/teeth and the restoration are done in same appointment in dental clinic without the need the help of laboratory technician [39, 40].

2.10.12. Laboratory Fabrication

In lab fabrication the impression taken by the dentist, then send it to the lab to complete the steps of fabrication of different dental restorations [39].

2.10.1.3. Centralized Fabrication

In this type after took the impression, the master cast is digitized in lab [39]. Then send it to outsource laboratory through internet for fabrication of high quality final restoration [39].

2.10.2. Comparison between Conventional Restoration and CAD/CAM Restoration

The CAD/CAM system does not require temporization because the final restoration is permanent cemented to prepared tooth on the same day of the preparation. Therefore, no temporization will decrease post-operative sensitivity because temporization in the conventional restoration undergo to the digital impression is more comfortable for patient and dentist more than the conventional impression [39, 40]. Therefore the digital impression need less time than conventional impression [39]. The digital impression not require model poring, but in conventional impression the model poring is needed and shelves are required to keep the model [39].

2.10.3. The Types of Methods Used to Obtain Data for CAD/CAM Systems

2.10.3.1. Intra Oral Scanning

Intra oral scanners are available in dental market in many types such as CEREC, Lava C.O.S [39, 40]. These scanners take 3D picture of the prepared teeth and adjacent structure [39]. When taking 3D picture must use tissue retraction, hemostasis and the moisture must be controlled [39].

2.10.3.2. Contact and Non-Contact Digitization

The conventional impression is taken by dentist [39], and then he made model from it. When the data transferring from model to CAD through probe digitization the process called (contact) digitization [39]. But when the data transferred through laser light the processes are called (non-contact) digitization [39].

2.10.3.3. Computer Tomography / Magnetic Resonance Image (CT/ MRI)

It is a newer method used for transferring of data to CAD/CAM [39]. The CT scans radiation for its data, but MRI does not[39]. MRI data is indicated for soft tissue modeling, but CT scan data is used for hard tissue modeling [39].

2.10.4. Indication of CAD/CAM Restoration in Dentistry

In 1994 Maede et al. proposed that the removable complete dentures can be fabricated by CAD/CAM system, but there are not many studies supporting this proposal [39]. The framework of removable partial dentures can be fabricated through CAD/CAM [39]. The most successful material used with CAD/CAM for fabrication of crowns and bridges is zirconia [39]. Also onlay, inlay, veneers, metal and porcelain crowns and bridges also can be produced through CAD/CAM [39, 40].

2.11. Methods of Surface Treatment of Ceramic Restoration

Surface treatments have been used to improve adhesion and bonding between luting cement and ceramic restoration [43], but the composition of ceramic determine which treatment is suitable for it [41,43].

2.11.1. Mechanical Surface Treatment

Mechanical surface treatment used to produce roughness on the ceramic surface [43]. This roughness will increase surface wettability, surface energy and increase surface area of ceramic exposed to resin cement which lead to increase mechanical interlocking and bond strength [41, 43, 44, 45, 46, 48, 50].

2.11.1.1. Airborne Particles Abrasion (Sandblasting)

The sandblasting method used air with specific pressure containing aluminum oxide (Al_2O_3) particles with different size [41, 42, 44, 46, 48]. In spite of sandblasting easy to apply and need short time but it can improve bond strength to zirconia [43, 42, 48, 50]. Air abrasion is more effective in alumina more than zirconia, because the alumina is less ductile with high surface hardness and larger grain [42]. The small size of alumina oxide particles less than 50Mm and use excessive pressure of air with short distance between nozzle and ceramic surface can cause micro crack which lead to weaken of the restoration and decrease fracture toughness [43, 44, 41, 46, 50, 51]. So the success of the indirect restoration closely related to the retention of the restoration. Because good retention prevent micro leakage and increase fracture resistance.

2.11.1.2. Silica Deposition by Air Abrasion

This method has been first introduced in 1984s [42]. In this type of air abrasion use Silica-coated Alumina particles (110Mm or 30Mm) [42, 48, 43]. These particles produce ceramic surface more react to silan which lead to improve adhesion and increase micromechanical bond [42, 43, 45, 48, 50, 51,]. Some studies reported that, there is very little difference between shear bond strength produced from conventional air abrasion and Si-coated air abrasion [42, 50]. The first commercially laboratory device (Rocatec system) was introduced in 1989s and the Cojet is considered as first chairside device [42].

2.11.2. Chemical Surface Treatment

2.11.2.1.Hydrofluoric Acid Etching:

The hydrofluoricacid (HF) etching is used to dissolve silica content and glassy matrix of glass ceramic to produce surface porosity [43 , and 41, 42, 44, 46, 47, 49]. This porosity will increase surface area for penetration and polymerization of resin cement [41, 42, 44.43, 46, 47, 48, and 49]. Therefore care must be taken to avoid over etching, which will decrease bond strength of ceramic[44].HF is more effective on silica based ceramic and cannot used effectively on non-silica based ceramic[43, 46, 49]. So many studies reported the HF acid cannot etch Y-TZP because it does not have glassy matrix [43, 48, and 49].

2.11.2.2. Functional Monomers (primer)

The ceramic primers are different from silane coupling agents, because the ceramic primer has acidic adhesive monomers that used with high strength ceramic such as Alumina and Zirconia but the silane coupling agent used with silica based ceramic such as lithium disilicate [100].

The ceramic primer classified into [101]:

1. Unhydrolyzed single liquid silane primer.
2. Prehydrolyzed single liquid silane primer.
3. Two or three liquid primer with separated silane coupling and acid activator.

The prehydrolyzed single liquid silane primer exhibit better bonding than unhydrolyzed, but the self-life and stability of prehydrolyzedsilane primer is less than yhe multi component liquid primer [101].

The 10-methacryloyloxyidecyl-dihyidrogenphosphat (MDP) is phosphate monomer that contain hydrophilic phosphate terminal end that form chemical bond with Zirconia and has polymerizable methacrylate terminal end that adhere with resin [102].

2.11.2.3.SilaneCoupling Agent

Silane coupling agents are hybrid inorganic-organic bifunctional molecules that usually contain silane coupler and a weak acid [41, 42, 44, 46, 48]. The silane coupling agent improves the wettability, produce chemical covalent and hydrogen bond of ceramic surface [41, 43, 44, 45, 46, and 50]. Siloxan bond is sensitive to hydrolytic degradation which affects the stability of adhesive interfaces [42, 46]. Silane coupling agent cannot produce good effect on ceramic surfaces does not have silica content such as zirconia [42, 43].

Lace et al., the silane coupling agents improve the wettability of the silica based ceramic surface when placed after air particles abrasion [41]. Sorensen et al., the use of silane coupling agent after acid etching that will decrease the micro leakage between adhesive interfaces [41, 44].

2.12. Measurement of Adhesion Bond Strength for Dental Materials

The selection of dental material depends on their bond strength [17]. The bond strength test classified into qualitative test that measure bond failure and quantitative test that measure the load capacity and life time of the bond[17].The laboratory dental bond strength may be static or dynamic [18,17]. The static test divided into macro test when the bonded area more than 3mm^2. It measures the shear and tensile bond strength. And micro test here bonded area less than 3mm^2 [17, 19, 20].

2.12.1 Shear Bond Strength Test (SBS)

The shear bond strength used to measure the bond strength of two materials connected to each other by adhesive agent. After that loaded until fracture occur [17]. The force applied down on the specimens produced by many types such as wire loops, points, knife edges [17, 21].

2.12.2. Tensile Bond Strength (TBS)

The tensile bond strength applied force on both side of the specimen [17]. The specimens fixed in place on TBS device by active gripping methods such as glue or clamps, or fixed by passive gripping methods in this type does not contain glue or other mechanical gripping. The TBS used to measure the bond strength of cement which adhered to metal or ceramic [17, 22].

2.12.3. Flexural Bond Strength

The flexural bond strength widely used to measure bond strength of ceramic which is loaded under direct tension (96). The FBS have two types. The first type is three point flexural tests this type is sensitive and simple method used to measure bond strength of ceramic veneer adhered to zirconia core (98, 97). The applying force increasing until the separation occurs. The second type is four point bending test. It has been first introduced by Charalambides et al and it used to measure the interfacial fracture toughness of some types metal-ceramic restoration (99)

2.13. Thermocycling Procedure

The temperature in oral cavity changed constantly due to eating, drinking and breathing (60). These changes in the temperature cause mechanical stresses which lead to crack propagation through bonded interfaces and also made cause dimensional changes on it, which lead to gap formation that site for fluid and bacteria accumulation (60, 63). Therefore thermocycling machine used to stimulate most effective factors in oral environment such as change in temperature between $4C^0$ and $60C^0$ with rapid cycles (54). The thermocycling regimens are stimulated more than clinical situation. It has effects on most bonded dental material even the newest materials such as self-adhesive self-etch resin by sudden changes in temperature that lead to decrease its bond strength (60).

3. MATERIAL AND METHODS

3.1. Detection of Experimental Groups

In our study we used 40 disc shaped specimens produced from Zirconia (Zr) (n=40). The specimens had the following dimensions 10 mm in diameter and 3 mm thickness.

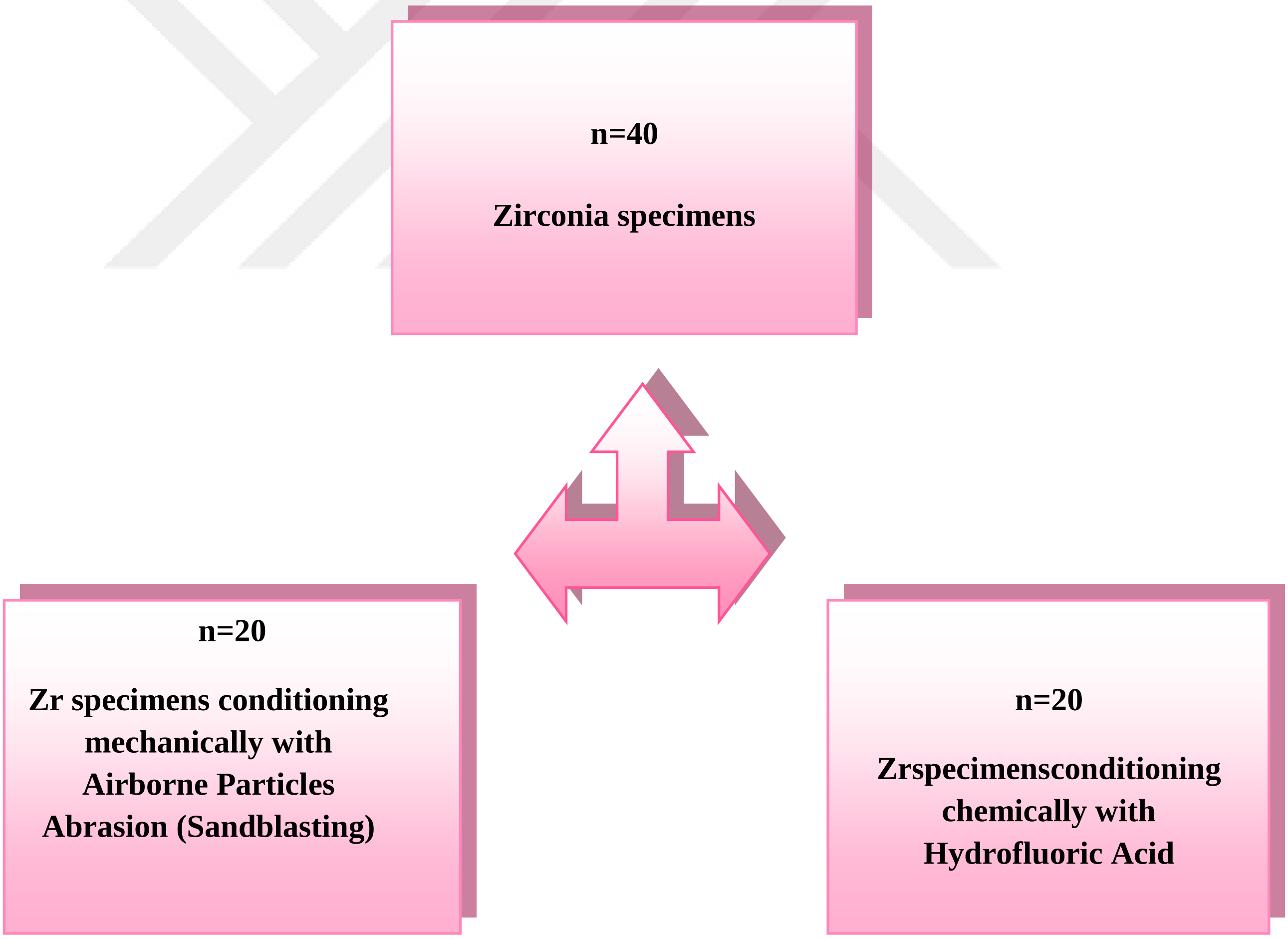

Figure-1:Flow-Chart Illustrate Experimental Group

3.2. Fabrication of Zirconium Discs

These discs were prepared from the pre-sintered Zirconia blocks (StarCeram Z-Med Germany) which fabricated to the final required dimension (10mm diameter and 3 mm thickness) by using CAD/CAM system (Cortitec T 350I loader imes-icore, Germany). After that the Zirconia specimens were sintered in special high temperature furnace. The dimension of the specimen has been produced according to ISO standards for dental ceramics (ISO 6872, 2008).

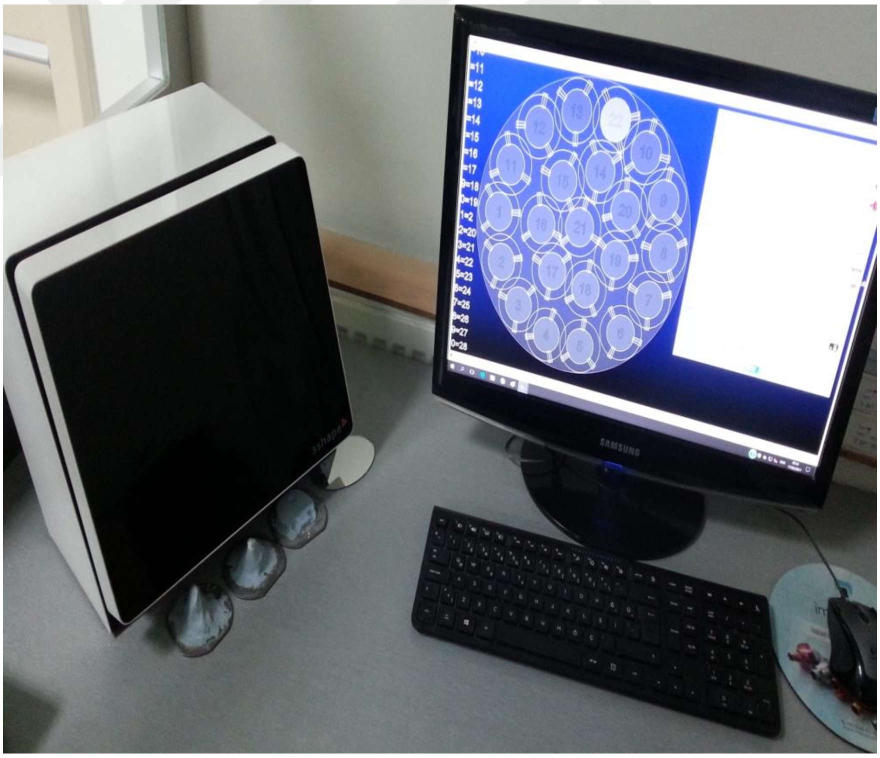

Figure-2: Design of the Zirconia Discs on Computer

Figure-3: CAD/CAM Machine

Figure-4: Place the Zirconia Block in the CAD/CAM Machine

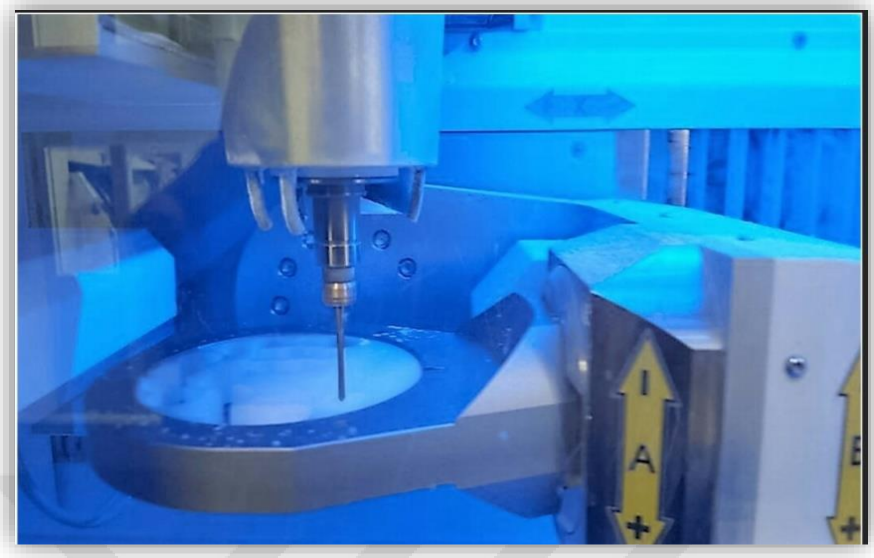

Figure-5: Milling of Zirconia Discs from Pre-Sintered Zirconium Blocks

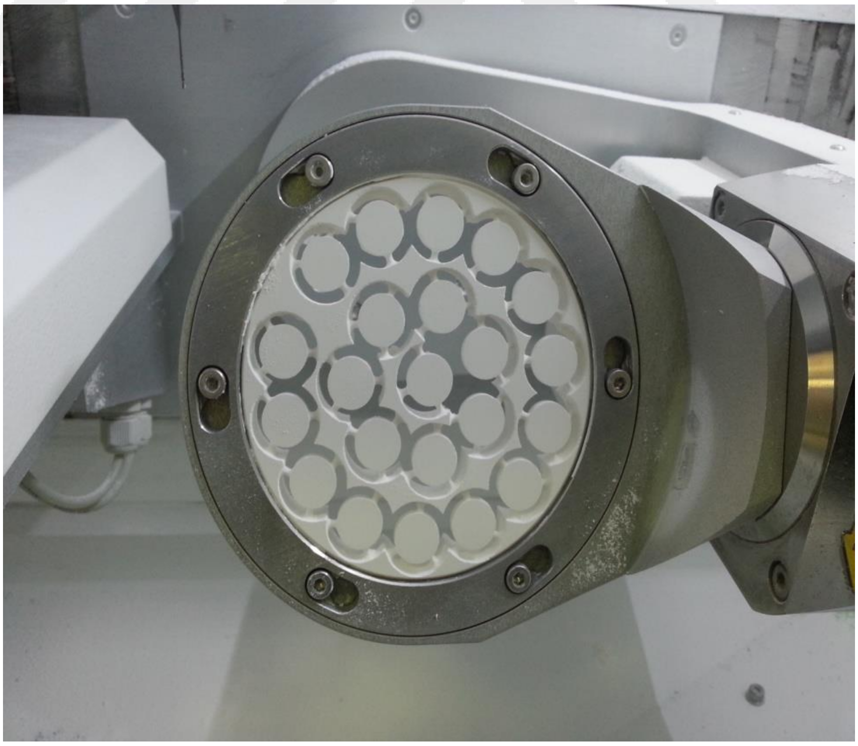

Figure-6: Zirconia Block after Milling

Figure-7: The Furnace Used for Sintering Zirconia Discs

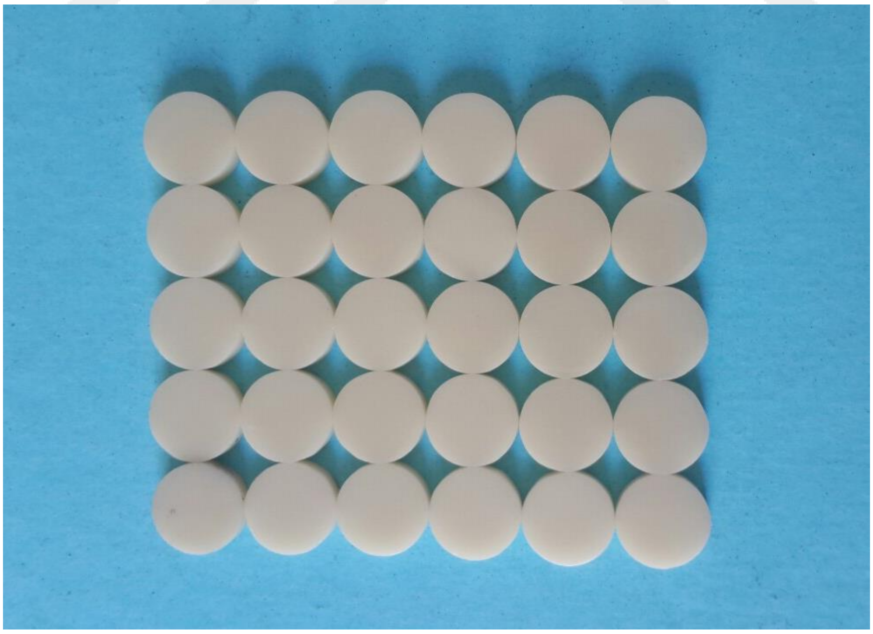

Figure-8: Zirconia Discs after Sintering

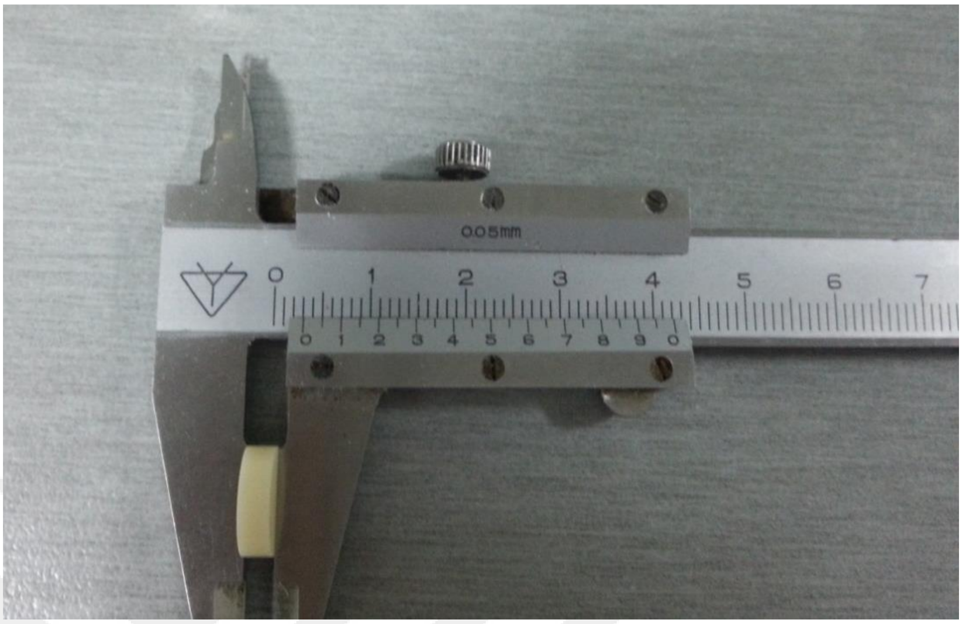

Figure-9: Show Thickness Measurement of Zirconia Specimens (3mm)

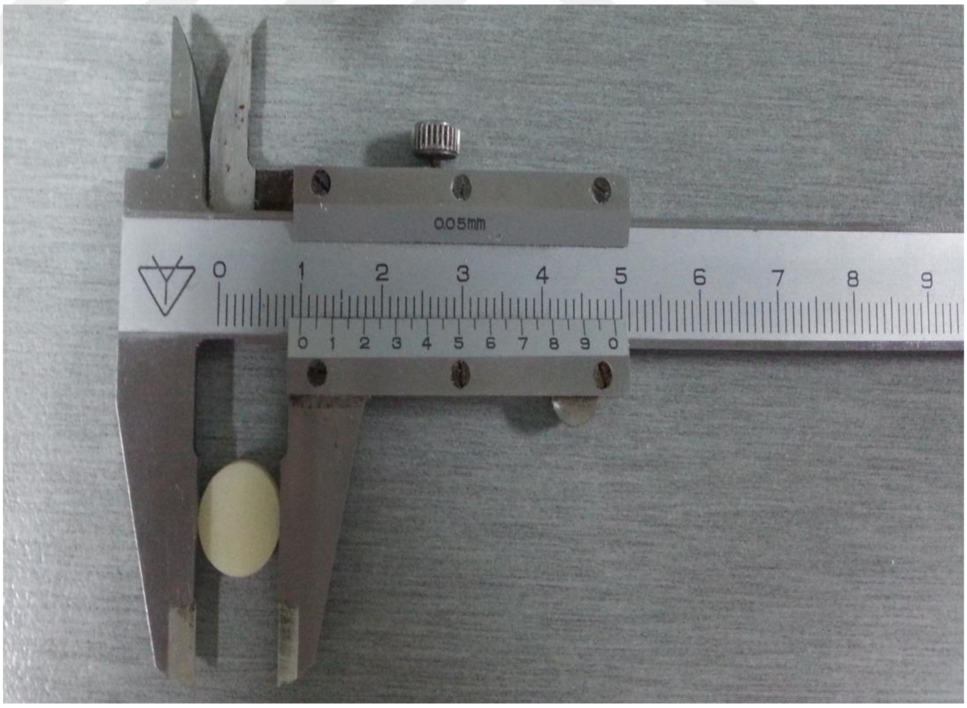

Figure-10: Diameter of Zirconia Specimens (10mm)

Table 3: The Material Used in Our Study

Brand	Manufacturer
Zirconia Ceramic	StarCeram Z-Med Germany
Porcelain etchant BISCO, INC 9.5% HF)1100W.Irving Park Rd Schaumburg, IL 60193 U.S.A 847-534-6000	
Z- prime plus 11OOW.Irving Park Rd Schaumburg, IL 60193 U.S.A 847-534-6000	BISCO, INC
Clearfil ceramic Primer	Kuraray Noritake Dental INC Japan

Figure-11: Material Used in the Present Study

3.2. Surface Treatment

After sintering the Zirconium discs in special oven, all the Zirconium specimens were polished manually by silicon carbide paper with number 600, 800 and 1200 grit respectively under cooling water for a minute for each Zr specimen. After that the specimens were cleaned in ultrasonic cleaning machine with distilled water.

Figure-12: Ultrasonic Cleaning Machine Used with Distilled Water

The all Zirconia discs (n=40) were divided into two groups according to the type of surface conditioning. n=20 Zr discs were conditioning mechanically with airborne-particle abrasion with Alumina (sandblasting) and other n=20 Zr discs were conditioning with Hydrofluoric acid. After that each group were subdivided into two subgroups according to the type of primer used.

Group-1: 10 Zr specimens conditioning with Sandblasting after that Clearfil Ceramic primer were applied.

Group-2: 10 Zr specimens conditioning with Sandblasting and after that Z-Prime Plus primer were applied.

Group-3: 10 Zr specimens conditioning with Hydrofluoric acid after that Clearfil Ceramic primer were applied.

Group-4: 10 Zr specimens conditioning with Hydrofluoric acid after that Z-Prime Plus were applied.

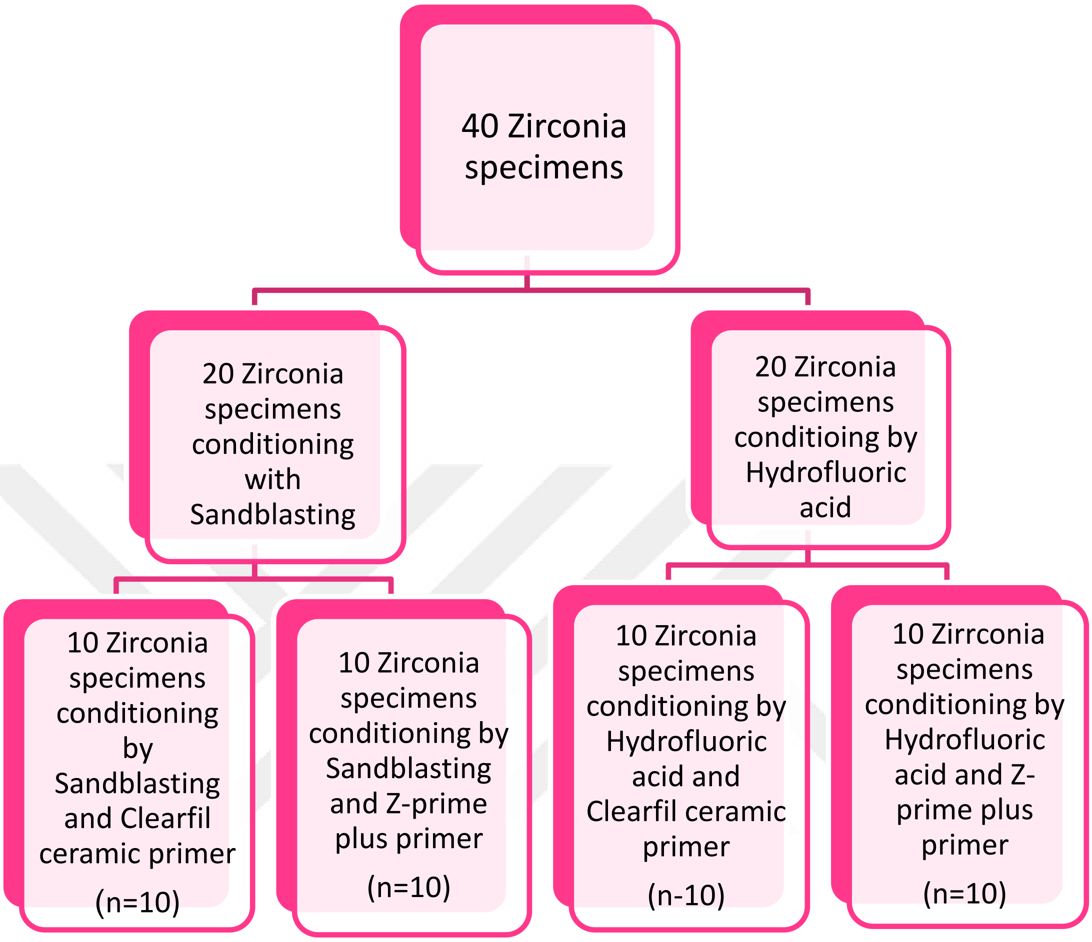

Figure-13: Low-Chart Illustrate All the Groups in the Test

The n=20 Zr specimen surfaces were sandblasted with 50Mm Aluminum oxide particles (Al_2O_3)with air pressure 0.2 MPa for 20s.The nozzle of sandblasting machine placed vertically to the Zr specimen surface at distance 20 mm. After that the specimens were cleaned ultrasonically with distilled water and air-drying.

Figure-14: Sandblasting Machine with Pressure 0.2 Mpa

The other n=20 Zr specimens were conditioning with 9.5% Hydrofluoric acid gel (Bisco) for 90s. After that, the etching gel was washed and rinsed with a copious amount of water and then dried by air.

Figure-15: Etching of Zirconia Specimens by Hydrofluoric Acid

3.3. Primer Coating

After we finished the sandblasting and Hydrofluoric acid etchıng, the main group divided into two subgroup according to the type of primer used. In the group-1 and group-3 we use Clearfil Ceramic primer, and in the group-2 and group-4 we use Z-prime plus primer. The primer was placed in one layer on dry zirconium surface and then left for 1 min to react. Then dried with gentle air for 10s.

Figure-16: Clearfil Ceramic Primer

Figure-17: Z-Prime Plus Prime

Table 4: The composition, indication and operating procedure of Clearfil Ceramic primer

Material	Composition	Indication	Operating procedure
Clearfil ceramic primer (CFC) (Kuraray)	- 10methacryloyloxydecyl dihydrogen phosphate (MDP) - 3-methacryloxyoropyl trimethoxysilane and ethanol	- Surface treatment of prosthetic restoration made of hybrid ceramics, composite resin or metal. - Intraoral repairs of fractured restoration made of hybrid ceramic, composite resin or metal.	- Treat the adhered surface of the restoration either by Hydrofluoric acid then clean it by water and dry. Or roughen the adhered surface by sandblasting with Alumina powder(30-50Mm) after that clean it by water in the ultrasonic machine for 2 minute then dried with air. Apply the CFC primer to the adhered surface by brush then dry it by oil free air. Place resin cement.

Table 5: The composition, indication and operating procedure of Z-PRIME Plus primer

Material	Composition	Indication	Operating procedure
Z-prime plus primer (Bisco)	MDP(phosphate monomer) PPDM(carboxylate monomer)	- Zirconia - Alumina - Metal/alloy - Composite - Endodontic post - Intraoral repairs	- Clean the adhered surface of the restoration by using one or more of the options: pumice scrub, ultrasonic cleaning, steam cleaning and/or sandblasting using - Aluminum oxide (30-100 micron grit at 30-45 psi) then rinse and dry. - Apply 1-2 coats of Z-prime plus primer, uniformly wetting the bondable surface after that, dry with air syringe for 3-5 seconds. - Place resin luting cement.

3.4. Cementation Procedure

After the surface treatment (sandblasting and Hydrofluoric acid) and apply the ceramic primer, the self-adhesive resin cement (Multilink speed Ivoclar Vivadent) was applied on the surface of zirconium specimens. The Plexiglas mold 4mm in diameter and 3mm height was placed in the center of the specimen. Then put the self-adhesive resin cement by mixing tip in the mold. After that the cement was light cured for 20 seconds.

Figure-18: Multilink Self-Adhesive Resin Cement

Table-6: The composition, indications and operating procedure of Multilink self-adhesive resin cement

Materials	Composition	Indication	Operating procedure
Multilink self-adhesive resin cement	- The multilink self-adhesive resin cement made from monomer matrix that contain dimethacrylates and acidic monomers and contain inorganic fillers are barium glass, ytterbium tri fluoride, copolymer and highly dispersed silicon dioxide. - Additionally it contains initiators, stabilizers and colour pigments. The size of inorganic fillers are between 0.1Mm to 7Mm and total content of fillers is approximately %40vol.	-Permanent cementation on natural teeth in conjunction with restorations made of metal and metal ceramic restoration such as inlays, onlays, crown, bridges, endodontic posts and fiber reinforced composite and cementation of All ceramic restoration made from zirconium oxide, Lithium Disilicate and aluminum oxide ceramic. -Permanent cementation of crowns and bridges made of metal, metal ceramic and high strength all ceramic such as zirconium oxide, lithium disilicate and aluminumoxides ceramics. -On implant abutment made of oxide ceramic such as zirconium oxide and metal as titanium.	Seat the restoration and retain it in place under uniform pressure then the excess cement is light cured for 1s per quarter and removed it by scaler. After that, complete light cure for 20s.

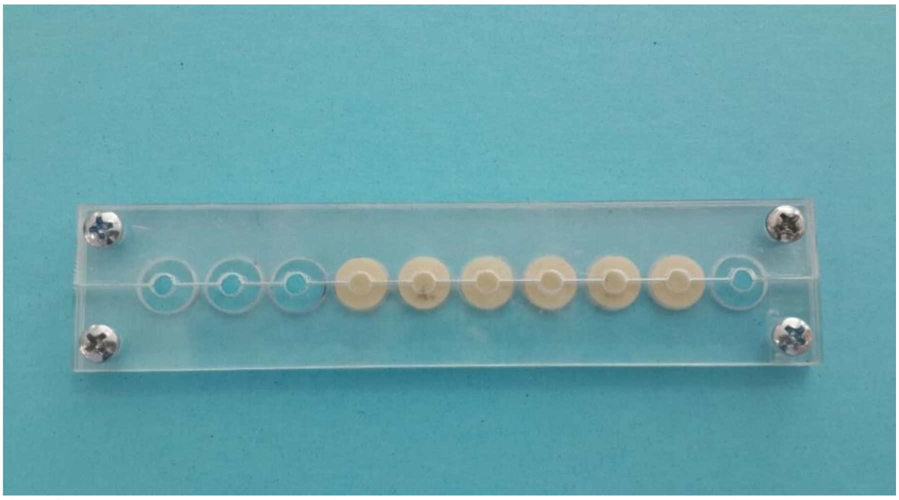

Figure-19: Translucent Plexiglas Mold

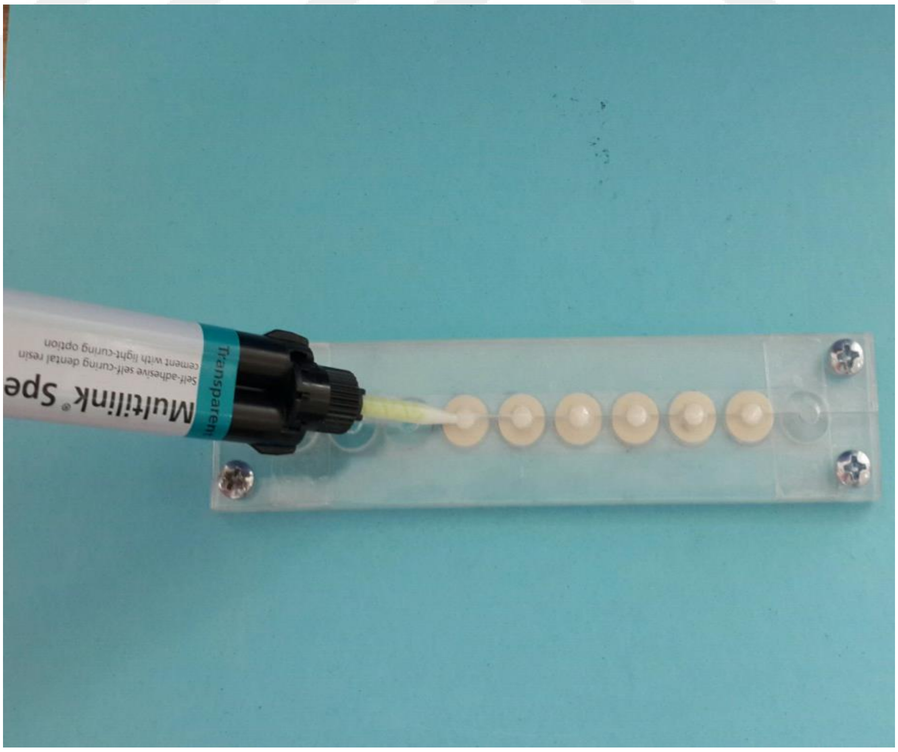

Figure-20: Put the Multilink Cement in the Mold

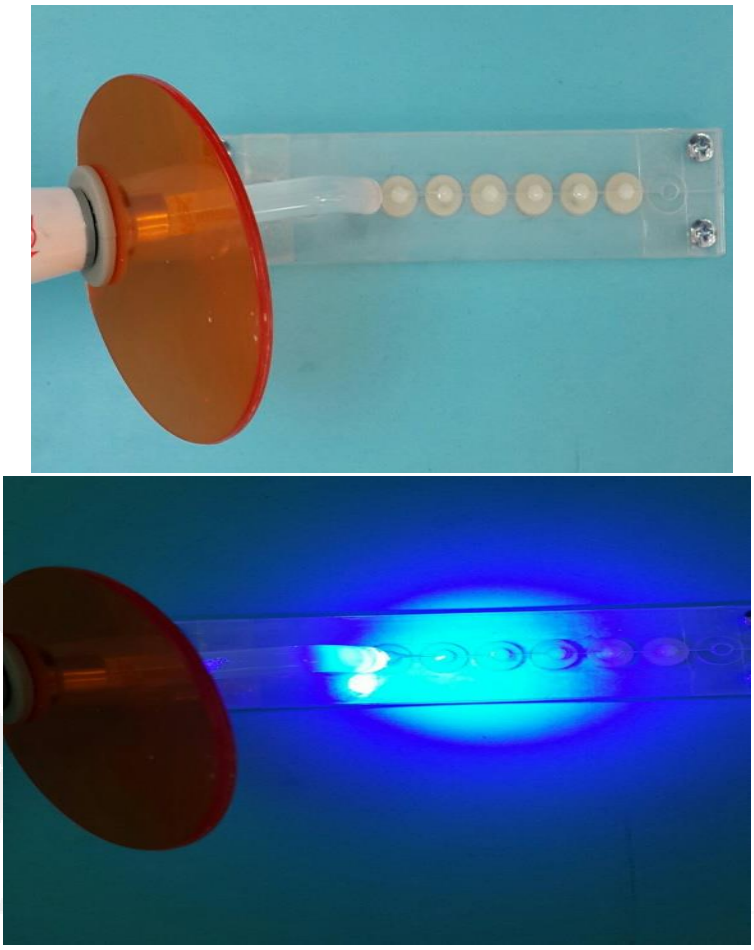

Figure-21: Light Cure the Self-Adhesive Resin Cement

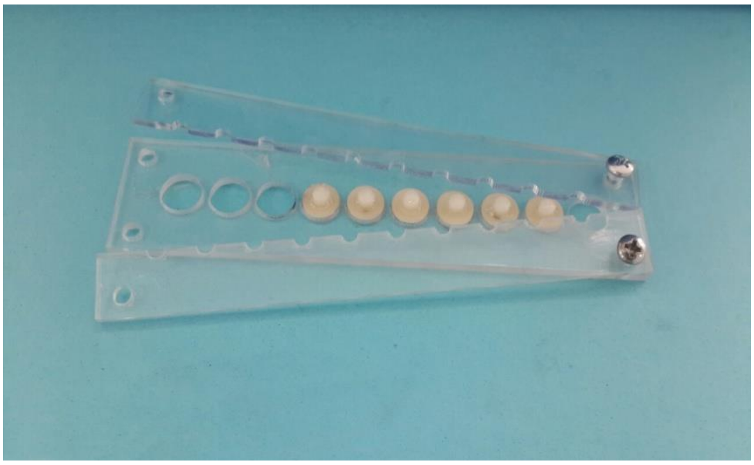

Figure-22: Remove the Plexiglas Mold from Zirconia Specimens

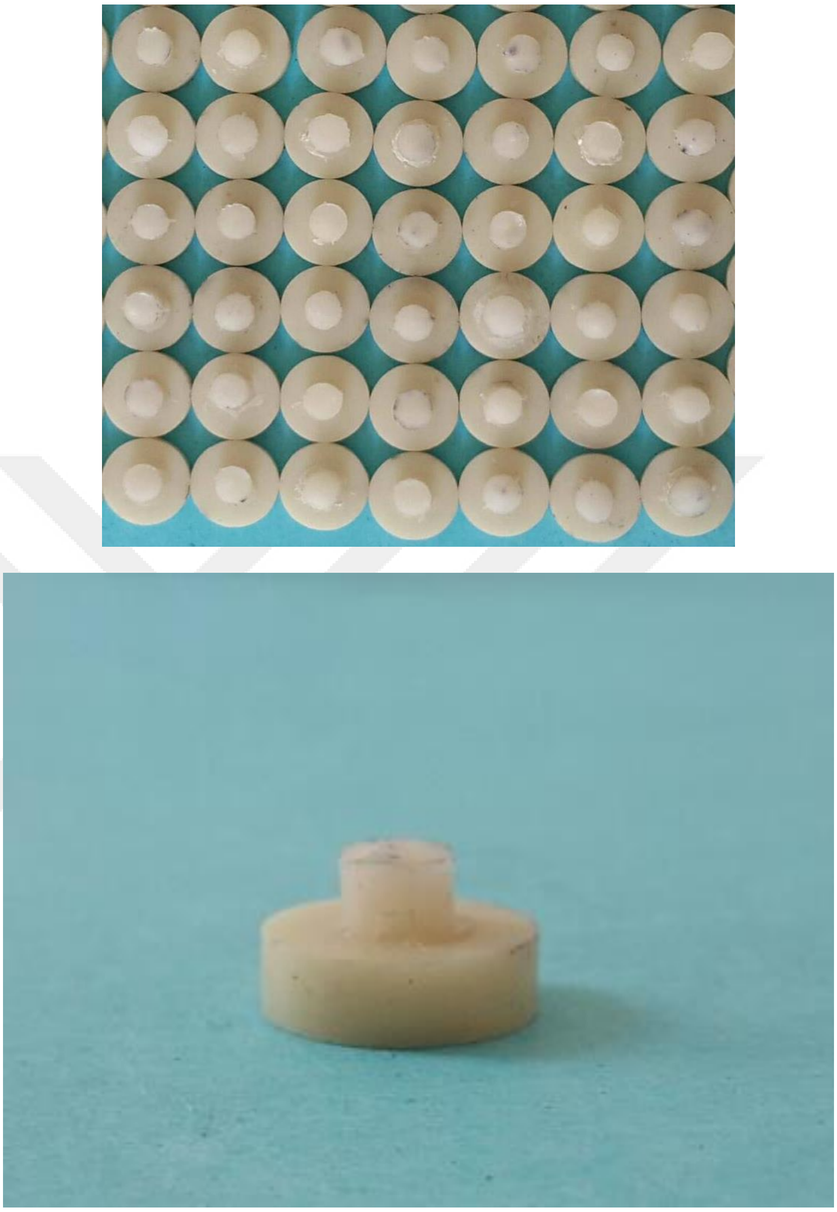

Figure-23: Self-Adhesive Resin Cement after Complete Curing

3.5. Thermocycling

The specimens were thermal cycled in Salibrus-Technica, Turkey for 5000 cycles and temperature changed between $5C^0$to$55C^0$after cementation procedure. The specimens transferred between two baths with different temperature and it remained in each bath about 30 seconds.

Figure- 25: Salibrus-Technica for Thermocycling

3.6. Shear Bond Strength Test

After finished the thermocycling, the all specimens were fixed on the stainless steel mold (12mm height and 20mm in diameter) by self-cure acrylic resin. After that we put the specimens in the universal testing machine to measure the shear bond strength between zirconium surface and self-adhesive resin cement.

Figure-25: Stainless Steel Mol

Figure-26: Self-cure Acrylic Resin

Figur-27: The Zirconia specimens after adhered it by self cure acrylic resin a stainless steel mold.

Table 7: The composition, indication and operating procedure of self-cure acrylic resin

Material	Composition	indication	Operating procedure
Self-cure acrylic resin IMICRYL	- Powder polymethylmethacrylate -Liquid methylmethacrylate	Using reformig and prosthesis repairs	- Use suitable container to prepared the acylic dough. -The polymer is poured over the monomer in the indicate ratio. -The mixture should be mixed for 30 seconds in crosswise motion to ensure the complete incorporation of monomer and polymer particles. -The container should be covered by lid to prevent the inclusion of air until mixture reaches a thickness in a fluid phase. -After that, put the mixture immediately to the corresponding area.

The universal testing machine was used to measure shear bond strength consist of knife edge blade was parallel to the interface of self-adhesive resin cement and zirconium specimen surface. The debonding of shear bond strength was registered in Newton (N) and the failure load (N) was divided by the bonding area (mm^2) then the debonding sear bond strength was converted into MPa.

$$\text{Stress (MPa)} = \text{failure load (N)} \backslash \text{surface area } (mm^2).$$

Figure-28: Universal Testing Machine

4. STATISTICAL ANALYSIS

Statistical calculations were performed with (Number Cruncher Statistical System) 2007 statistical software (Utah, USA) program for windows. Besides standard descriptive statistical calculation (Mean and standard deviation), one way ANOVA was used in the comparison of groups. Post Hoc Tukey multiple comparison tests were utilized in the comparisons of subgroups. Unpaired t test was used in the comparison of two groups.

Statistical significance level was established $P<0.05$.

5. RESULTS

In our study , a statistically significant difference was observed between the shear bond strength (Mpa) averages of Group 3 (Hydrofluoric Acid + Clearfil Ceramic Primer), group 4 (Hydrofluoric Acid + Z Prime Plus Primer), Group 1(Sandblasting + Clearfil Ceramic Primer) and group 2 (Sandblasting + Z Prime Plus Primer) groups (P 0.0001).

Table-8: Shear bond strength means for all Zirconia groups

One-Way ANOVA	**Number (n)**	**Shear Bond Strength (Mpa)**	**Shear Bond Strength (N)**
Hydrofluoric Acid + Clearfil Ceramic Primer	8	0,96±0,42	13,63±2,54
Hydrofluoric Acid +Z Prime Plus	10	1,78±0,26	22,38±3,23
Sandblasting + Clearfil Ceramic Primer	10	2,16±0,45	27,14±5,67
Sandblasting + Z Prime Plus	10	2,94±0,42	36,92±5,27
Shear Bond Strength (Mpa)		P o,ooo1	
Shear Bond Strength (N)			P o,ooo1

Table-9: Comparison between the subgroups

Tukey Multiple Comparison Test	**P**
Hydrofluoric Acid + Clearfil Ceramic Primer/ Hydroflouric Acid + Z Prime Plus Primer	0,001
Hydrofluoric Acid + Clearfil Ceramic Primer/ sandblasting + Clearfil Ceramic Primer.	0,0001
Hydrofluoric Acid + Clearfil Ceramic Primer/ Sandblasting + Z Prime Plus.	0,0001
Hydrofluoric Acid + Z Prime Plus Primer/ sandblasting + Clearfil Ceramic Primer.	0,157
Hydrofluoric Acid + Z Prime Plus Primer/ sandblasting + Z Prime Plus Primer	0,0001
Sandblasting + Clearfil Ceramic Primer/ sandblasting + Z prime plus primer.	0,001

The shear bond strength (Mpa) of group 3 (Hydrofluoric Acid + Clearfil Ceramic Primer) was found to be statistically significantly lower than that of group 4 (Hydrofluoric Acid + Z Prime Plus Primer), group1 (Sandblasting +Clearfil Ceramic Primer) and group2 (Sandblasting + Z Prime Plus Primer groups) (p=0.001, 0.0001).

The shear bond strength (Mpa) of group 4(Hydrofluoric Acid + Z Prime Plus Primer) was found to be statistically significantly lower than the shear bond strength (Mpa) of group 2 (Sandblasting Z Prime Plus Primer group) (p=0.001).

The shear bond strength (Mpa) of group 1 (Sandblasting + Clearfil Ceramic Primer) was found to be statistically lower than the shear bond strength (Mpa) of group 2 (Sandblasting Z Prime Plus Primer) (p=0,001).

There was no statistically significant difference between the shear bond strength (Mpa) averages of group 4 (Hydrofluoric Acid + Z Prime Plus Primer) and group 1 (Sandblasting + Clearfil Ceramic Primer) (p= 0.157).

Table (10) Shear Bond Strength (MPa) means for Ceramic Primers (Umpaired Test)

Shear bond strength (MPA)	**Hydrofluoric acid**	**Sandblasting**	**P**
Clearfil Ceramic Primer	0,96±0,42	2,16±0,45	0,0001
Z-Prime Plus primer	1,78±0,26	2,94±0,42	0,0001
P	0,0001	0,0001	

Table-11: Shear Bond Strength (N) means for Primers groups

Shear Bond Strength (N)	Hydroflouric Acid	Sandblasting	P
Clearfil Ceramic Primer	13,63±2,54	27,14±5,67	0,0001
Z Prime Plus Primer	22,38±3,23	36,92±5,27	0,0001
P	0,0001	0,0001	

The shear bond strength (Mpa) of the Hydrofluoric Acid + Clearfil Ceramic Primer group was found to be statistically significantly lower than the shear bond strength (Mpa) of the Sandblasting + Clearfil Ceramic Primer group (p=0,0001).

The shear bond strength (Mpa) of the Hydrofluoric Acid + Z Prime Plus Primer group was found to be statistically significantly lower than the shear bond strength (Mpa)of the Sandblasting Zprime Plus group (P 0.0001).

The Hydrofluoric Acid + Clearfil Ceramic Primer group had a significantly lower shear bond strength (Mpa) than the Hydrofluoric Acid + Z Prime Plus Primer group shear bond strength (Mpa) (P=0,0001).

The Shear bond strength (Mpa) of Sandblasting + Clearfil Ceramic Primer group was statistically significantly lower than Sandblasting + Z Prime Plus Primer group. Shear bond strength (Mpa) averages (P= 0,001).

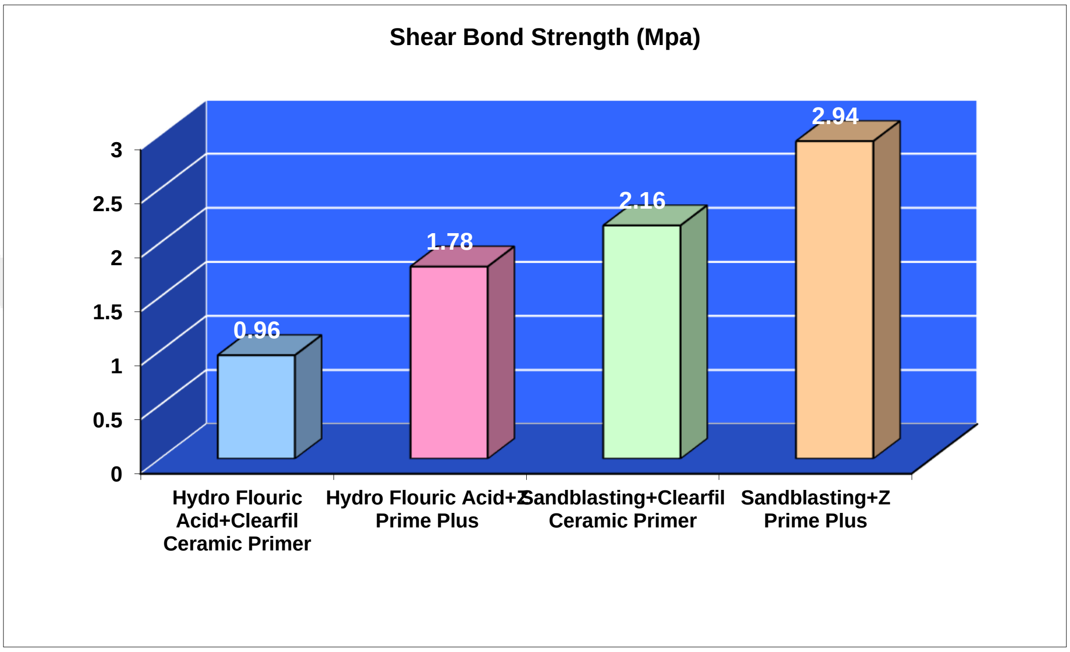

Figure -29: Bar-Chart shows the Shear Bond Strength (MPA) for ceramic Primer

The highest shear bond strength (MPA) was in the Sandblasting + Z Prime Plus Primer group (2,94±0,42).

In the groups which used sandblasting had significantly higher shear bond strength (MPA) than in the groups used Hydrofluoric Acid.

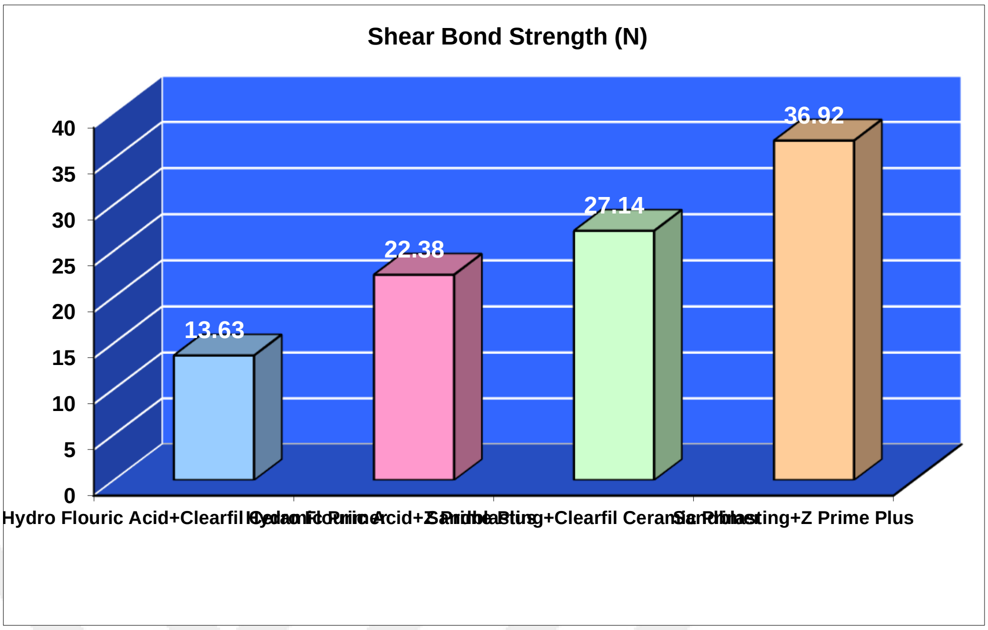

Figure-30: Bar-chart shows the Sear Bond Strength (N) for ceramic primer

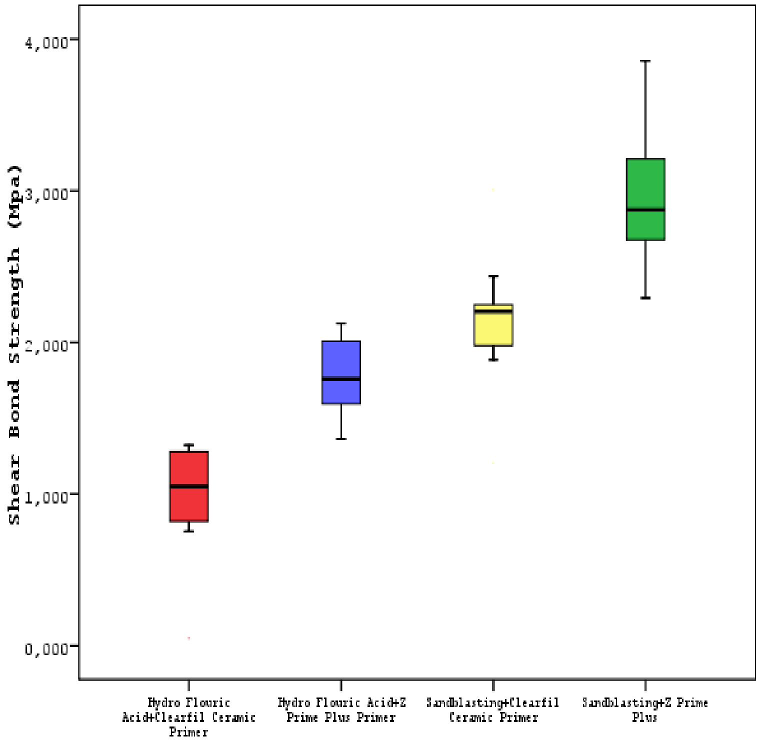

Figure-31: The shear bond strength of four groups

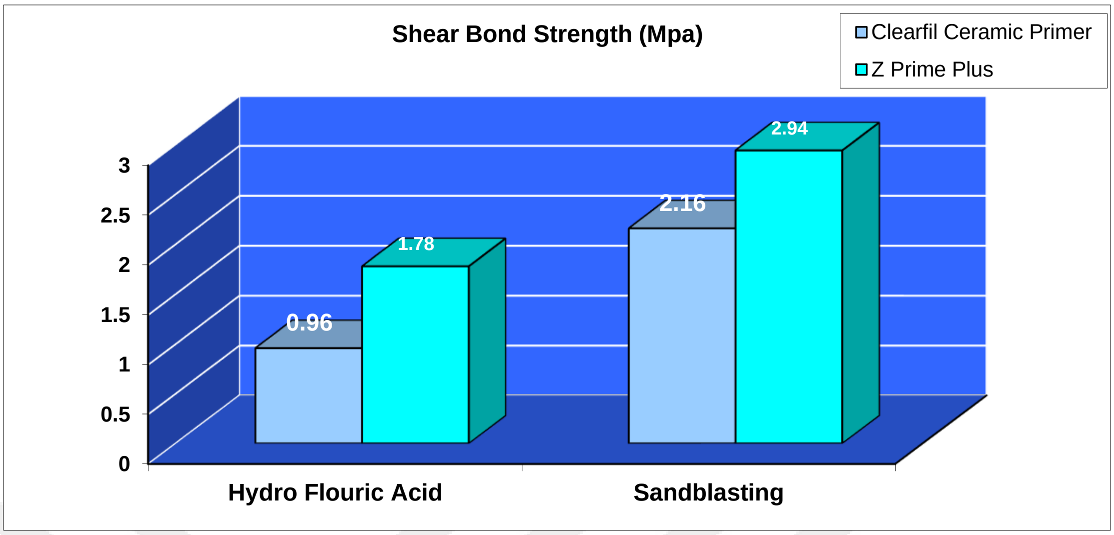

Figure-32: Bar-Chart shows the difference in the Shear Bond Strength (MPA) of sandblasted specimens and Hydrofluoric acid etched specimens

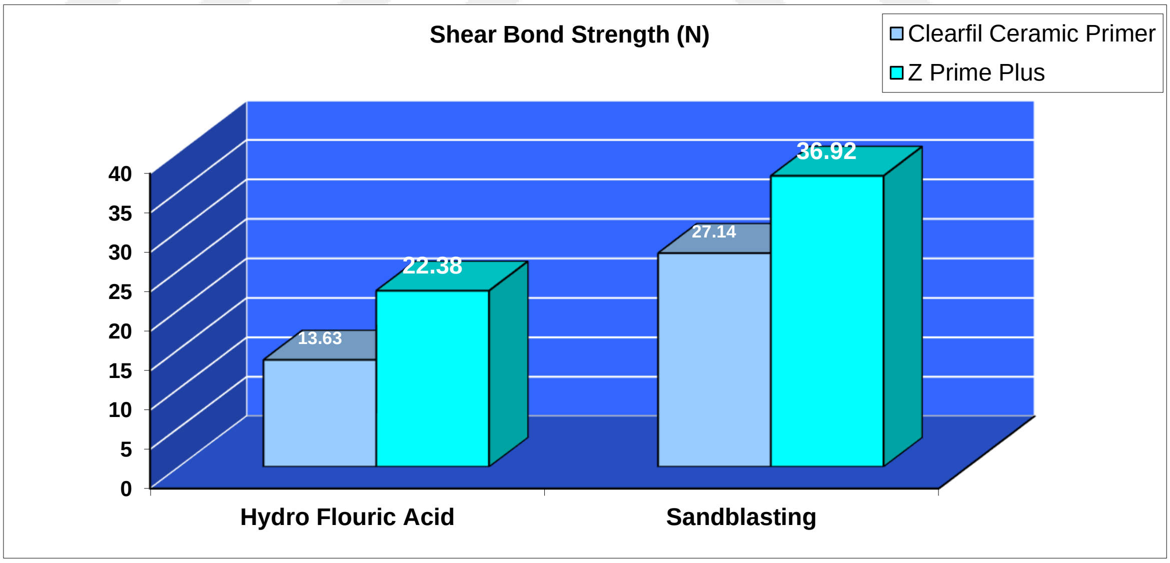

Figure-33: Bar-Chart shows the comparison between the Shear Bond Strength (N) of sandblasted specimens and Hydrofluoric acid etched specimens

6. DISCUSSION

All ceramic restorations are considered as the best option in a clinical situation that requires highly aesthetic properties. These aesthetic characteristics mean the ability of all ceramic restoration to give the color and translucency similar to natural teeth, the all ceramic restorations have another important properties such as high fracture toughness, chemical durability, wear resistance, excellent intraoral stability and biocompatibility(95, 30, 38, 33, 36).

6.1 Zirconia Ceramic

The name of Zirconium derived from Arabic word (Zargon) (84, 82, 83), which means golden in color and it first introduced in1789 by Martin Heinrich Klaproth(84, 83). There are three types of ZIrconia Ceramic used in dentistry which are Yttrium cation-doped tetragonal Zirconia(3Y-TZP), Partially Stabilized Zirconia(Mg-PSZ) and Zirconia-Toughened Alumina(ZTA)(82, 84). The Zirconia used in dental clinic is polycrystalline ceramic (83, 84). The Zirconia has highest flexural strength (900 MPa) and fracture toughness (9-10MPa.m$^{1/2}$) when compared it to another types of ceramic(81, 82, 83). The chipping of Zirconia veneering is considered as the most common failure occurred in dental clinic (81). There are many causes of chipping fracture such as the difference in the coefficient thermal expansion, toughness and flexural strength between Zirconia and veneering ceramics, amount and location of occlusal load, thickness of porcelain, improper framework design (81). The Computer-Aided Design (CAD) and Computer-Aided Manufacturing (CAM) techniques used to prevent this failure of Zirconia and provide good marginal fit(81, 83). Until now the best treatments of Zirconia surface still unknown (84). But the airborne particles abrasion, silica coating and hot chemical etching are the best treatment used to enhancing the micromechanical retention of Zirconia ceramic [84].

6.2. The Failures and Survival Rate of All Ceramic Restoration

The survival rate for all ceramic restoration depends on intrinsic and extrinsic factors. The intrinsic factors include high amount of crystalline phase that responsible for the improvement of mechanical and physical properties of ceramics [34, 38], in addition this crystalline phase increase the opacity of ceramic material [34, 38], other intrinsic factors such as crystal size, phase transformation, modulus of elasticity and thermal expansion. The common extrinsic factors include working condition, cyclic load and peak lo ads that increased during mastication of hard objects and oral environment such as acidic or basic PH and humidity. The humidity leads to stress corrosion, catastrophic failure in ceramic material and cause microstructure degradation in some high crystalline material such as 3Y-TZP [38].

Otto and Schnieder et al. reported that the survival rate of CAD/CAM inlay and onlay after 17 years is % 88.7and only % 11failed, and %76of these failed restoration due to ceramic fracture [65].The most common ceramic failure is bulk fracture. The fracture of ceramic material occurred due to micro crack [67, 68].

Thompson et al. reported that there are many factors associated with crack initiation and propagation in ceramic restoration including ceramic microstructure, shape of all ceramic restoration, stress induced by surface treatment such as polishing and thermal processing, ceramic/cement interfacial bond, modulus of elasticity of the component of restoration thickness of ceramic restoration, oral environment and the amount of applied load [64]

Beier et al. examined the results of patients who had used dental prosthesis constructed only from ceramic, they found that the main failure of all ceramic restoration is ceramic fracture and the rate of fracture failure increased in the presence of parafunction like bruxism (66).

6.3. Surface Treatment

The adhered surface of all ceramic restoration must be treated before cementation to improve bond strength (42, 43, 46). There are two types of surface treatment of all ceramic restoration, mechanically surface treatment such as airborne particles abrasion(APA) or silica coating alumina particles and chemically surface treatment which can be done by acid etching or primer or both (42, 43, 46). The surface treatments were used to increase surface area of ceramic surface exposed to resin cement, which lead to increase mechanical interlocking, surface wettability and surface tension (42, 43, 44, 45, 46).

6.3.1 Effect of Airborne Particle Abrasion (APA)(Sandblasting) and Silica Deposition Air Abrasion

The airborne particles abrasion treatment use suitable air pressure with alumina particles, these particles used with different size (42, 43, 44, 45, 46). There are many factors that affecting the effectiveness of APA, these factors include size of alumina particles, distance between the ceramic surface and the nozzle and the air pressure strength used (43, 44, and 46).

The airborne particle abrasion surface treatment can be used to Zirconia surfaces with grains size approximately from 20Mm to 110 Mm. when use large grains this may be lead to microcrack initiation after finished air abrasion procedures, these microcrack lead to decrease the mechanical properties of the zirconium restoration [43, 44, 46, 50, 51]. In our study we used alumina particles size 70Mm with pressure 0.2MP for 20 seconds.

Özcan et al. found there is no significant differences in the shear bond strength between laboratory airborne particle abrasion with silica coated alumina particles(110Mm) and chairside airborne particle abrasion with alumina particles(50Mm), also they explain this result may be due to the airborne particles abrasion with large particles produce deep grooves on the zirconia surfaces, these deep grooves have less contact angle and wettability to silane coupling agent and also the use of large particles may cause ditching between zirconia surface and luting cement which lead to decrease shear bond strength[69].

Attiaetal.and Atsu et al. found the resin cement bond to zirconia ceramic surface increased by the use of silica coating alumina with silane coupling agent. This result may be due to the silica coating alumina increase roughness of ceramic surface which lead to increase surface area and improve mechanical and chemical bonding, also this procedures cause silica deposition on the ceramic surface that form chemical bond between ceramic surface and silane coupling agent[70,72].But in the same study they found there is no difference between bond strength of zirconia ceramic treated with silica coating alumina and zirconia ceramic treated with airborne particle abrasion[70].

Özcan M. and Vallittu P.Ket al, they found that the airborne particles abrasion is considered as the most important factor needed to produce adequate bond strength of resins to ceramics, and they found the bond strength for most types of ceramics significantly improved after airborne particle abrasion with the use of silane coupling agents[71].

6.3.2. Effect of Hydrofluoric Acid Etching

The dental ceramic consist of glassy matrix embedded in different amount of un dissolved feldspar and leucite crystals [79, 78, 80]. The Hydrofluoric acid dissolves the glassy matrix and the crystals remain, that lead to roughing of ceramic surface and increase surface energy which they needed to increase micromechanical retention [78, 79, 80]. The effectiveness of HF depends on its concentration and time of application [78].

Chaiybutr et al. reported that when they evaluate the effectiveness of HF etching in the occurrence of roughness on the surface of the different Zirconia ceramic. They found the effectiveness of HF on Zirconia different according to the composition of zirconia. In their study some type of zirconia produced lower bond strength and another types produced high bond strength when compared to conventional ceramic [78].

Qeblawi et al. reported that when they compared the effect of mechanical, chemical and combination of surface treatment on bond strength of Zirconia ceramic to resin cement. They found the combination of mechanical and chemical surface treatment on Zirconia ceramic that will produce strong bond to resin cement [79].

6.3. Effect of Primer

Ceramic primer is liquid substance that supplied as one bottle system, it's easy to apply with adequate shelf life and used to improve the bond strength between ceramic and resin cement. The ceramic primer consists of acidic adhesive monomer dissolved in a suitable and adequate amount solvent. The MDP (10- methacryloyloxydecyl dihydrogen phosphate) is considered as the most successful adhesive monomer used to improve the bond of ceramic to resin cement, because it contain phosphoric acid group that bond chemically to the metal oxide such as Zirconium dioxides and other end of double bond react to resin cement. Therefore, in our study we used ceramic primer containing MDP (Clearfil Ceramic primer and Z-prime plus primer) and our study found that the shear bond strength of ZPP to sandblasted Zirconia higher than the SBS of CCP to sandblasted Zirconia this result agree or similar to some studies such as:

Stefani et al, reported that the use of adhesive primer (Metal-Zirconia primer) increase bond strength of resin cement (MultilinkAutomix) to Zirconia(77).

Miragaya et al, found that the use of primer containing MDP (Alloy primer) is effective material used to increase the bond strength to Yttria-Stabillized Zirconia ceramic(Y-TZP)(75).

Keul C et al, reported that the use of ceramic primer had a good effect to improve the bond strength between Zirconia and resin cement (RelyXUnicemslf adhesive universal resin composite cement)(54).

TanişM.Ç. et al, reported that the use of primer containing adhesive phosphate monomer MDP (Z-PRIME plus primer) to sandblasted zirconia surface that will increase the bond strength between Zirconia ceramic and resin cement(conventional resin cement Variolink II)(76).

Al-Harbi et al, they found in their study the use of Clearfil ceramic primer before the place of Clearfil SA resin cement that increase shear bond strength of Y-TZP ceramic to core materials (103).

6.4. Bonding of resin cement to Zirconia

The resin cements are considered as the best choice and most effective cement that used for bonding of Zirconia restoration to Dentine and Enamel structures [109, 110]. The resin cements are low soluble in water that will increase the longevity of restoration and prevent recurrent caries, color alteration and decrease the sensitivity [110]. The success of Zirconia restoration and repairing fracture ceramic depends on the quality, strength and durability of the bond between Resin cement and remaining tooth structure and between the Resin cement and restoration [109, 110]. The bond of Zirconia restorations to resin cement depend on the surface treatment of Zirconia that produce micromechanical and chemical bond to resin cement [109, 110]. the micromechanical bond provided by roughing of Zirconia surface by Airborne particles abrasion using Alumina particle or Silica coated alumina particles, the roughness of Zirconia surface increase surface energy that will increase the wettability of adhesives to Zr, in addition to the bond increased when the cement place on clean and dry surface [109, 110]. The MDP based Resin cement make excellent adhesion to Zirconia even after thermocycling . Many in vivo studies reported the veneered Zirconia base restorations have survival rate (75%-100%) similar to other successful restorations [109].
The dual and chemical cure resin cement are best types of resin cement used with Zirconia because the opacity of Zr may cause incorrect polymerization of light cure resin cement [110].

De sa Barbosa et al, reported that the G-Cem self adhesive resin cement provided high bond strength to Zirconium oxide even after one year of water storage [52].

Da silva et al, compared between conventional resin cement and self adhesive resin cement that used to Yittria stabilized zirconia, they foud the micro SBS of self adhesive cement higher than conventional cement, but the micro SBS of self adhesive cement was decreased after 6 months in water storage aging [111].

6.5. Effect of Storage Medias

There are many storage media have been used before bond strength tests. These storage conditions include Distilled Water, %2 Glutaraldehyde, %0.5 Chloramines-T, %0.05 Saturated of Thymol and %10 Formalin solution(55,56). The bond strength decreased when use Sodium Hypochlorite (NACIO) as storage media (57). The long term water storage media with steady temperature is considered as a common technique used to evaluate the shear bond strength (54). In our study we used the distilled water as storage media for 20 days.

Keul et al, reported that the water storage media does not has significant deteriorating impact on the shear bond strength values (54).

De sâ Barbosa et al. found that the long term water storage (one year) decreased the bond strength of all resin cement used in their study. The reduction in the bond strength was at least %52 after long term water storage (52).

Silva DA et al. found that 6 months water storage decreased the bond strength of resin cement to Yttria-Stabilized Zirconia (53).

6.6. Effect of Thermocycling

The temperature of the mouth changes due to changes in the temperature of the food. Mair reported that when eating ice cream the temperature of the mouth changes from $4C^0$-$0C^0$ and when eating hot cheese the temperature of the mouth changes from $60C^0$-$65C^0$, these temperature changes affect the integrity of dental restoration (63). The thermal cycling used in vitro study to stimulate the temperature changes that occurring in the mouth [54]. In our study we used the thermal cycling as aging method for our specimens. The 6000 cycles equivalent to five years in the oral cavity [61, 62]. In the present study we used 5000 cycles that mean approximately equivalent 4 years in the mouth.the thermocycling has effects on dental material and it decrease the bond strength of most of them [60]

Gale and Darvell, summarized many studies used wide range of temperature changes of extracted teeth with restoration during thermal cycling. They found that the maximum comfortable tooth temperature was $55C^0$, but medium temperatures were $5C^0$ and $55C^0$, $0C^0$-$36C^0$ was low temperature and $40C^0$-$100C^0$ was high temperature. And they reported the dwell time(30 seconds) and wide range in thermal cycles(500 cycles)[54, 60].

Keul et al, compared the different aging procedure used in dental laboratory. They found that the thermal cycling has a noticeable influence than water storage when they used in the shear bond strength test that agree with our study because the SBS of our specimens decreased after thermocycling [54].

Buonocore et al and Keul et al, reported that the bond strength tests used in the in vitro study must be include thermal cycling of the specimens [59, 54, 58].

Ariciet.al in their study found that the thermal cycling has negative impact on the shear bond strength when they compared the effect of the thermal cycling on thebond strength of glass ionomer cement when used as orthodontic bonding agent [58].

6.7. Discussion of Groups

- Group 1 and group 2: 20 Zirconia specimens (Zr) treated with airborne particle (APA) abrasion and two different types of primer (Clearfil Ceramic primer (CCP) and Z-PRIME Plus primer (ZPP).

In our study the shear bond strength (SBS) result of group1 (Zr+APA+CCP) is lower than group2 (Zr+APA+ZPP). This result is similar to the result that has been done by Wang et al, when they compared the effect of air-drying pressure during ceramic primer coating (ZPP and CCP) on Zirconia. They found that the bond strength of Z-PRIME PLUS primer after sandblasting higher than SBS of Clearfil Ceramic primer after sandblasting at pressure 0.2 Mpa(74).

Taniş M.Çet al. found that Z-PRIME Plus primer significantly increase the shear bond strength of Zirconia with conventional resin cement (Variolink II). Our result agree with Taniş et al result but SBS values of Taniş M.Çet al study is higher than our study. This difference in result may be due toTanişM.Ç et al did not do long-term water storage and thermal cycling and they used different type of resin cement (76).

Magne et al, found that the shear bond strength of sandblasted Zirconia increased when use Z-PRIME Plus primer with different resin cement (104).

Zandsparsa et al. and Shin et al. reported that the use of Z-PRIME Plus primer with airborne particles abrasion increase bond strength of Zirconia compared to only airborne particle abrasion (105, 106).

- Group 3 and group 4: 20 Zirconia (Zr) specimens treated with Hydrofluoric acid(HF) and two different types of Ceramic primer Clearfil Ceramic primer(CCP) and Z-PRIME Plus primer(ZPP):

In this study the shear bond strength (SBS) result of group 3 (Zr+HF+CCP) is lower than group 4 (Zr+HF+ZPP). This result is agreed with the result of Sawad et al. compared the effect of different surface conditioning and use of universal primer (Monobond plus) on the SBS of Zirconia to self-adhesive resin cement. Sawad et al found theSBS of sandblasted Zr is more than the SBS of Zr etched with hydrofluoric acid. The lower SBS of zirconia specimens treated with HF in combination with primer may be due to the etched Zr was transformed from tetragonal phase to mono clinic phase which lead to decrease mechanical properties of Zr and cause crack propagation(108).

Sriamporn et al, reported that the nano-pores produced by HF cannot penetrated by high viscosity resin cement (107).

In our study we made group treated with Hydrofluoric acid only but this group completely failed after thermal cycling, this may indicate the hydrofluoric acid did not alter the Zr surface because HF act on glassy phase of ceramic and Zr does not contain glassy phase. This failed group happened also in Sawad et al study.

6.8. Comparison of Primer Types

In our study the shear bond strength of Z-PRIME Plus primer had higher shear bond strength than Clearfil Ceramic primer. This difference in the result may be due to differences in type and concentration of phosphate monomer used and time of application.

The CCP contain silane, but silane unstable in acidic environment nature of phosphate monomer that may lead to instability of silane component of this formula. The ZPP does not contain silane and it contains concentrated phosphate monomer and carboxylic monomer specially formulated for Zirconia and it does not contain silane (102).

7. STUDY LIMITATIONS

In our study we used shear bond strength test, this test has disadvantage of inhomogeneous stress distribution. Another limitation of this study is that only thermal cycling was used as artificial aging method to evaluate the durability, stability and hydrolytic degradation of the Resin-Zirconia bond.

In this study we used the distilled water as artificial media, but the distilled water does not have similar composition and PH of saliva.

Finally in present study we used one type of self-adhesive resin cement.

8. CONCLUSION

Within the limitation of this study, in our results suggest that;

1. The Z-prime plus (ZPP) improvethe shear bond strength (SBS) better than Clearfil ceramic primer(CCP).

2. The Z-prime plus (ZPP) improve the shear bond strength (SBS) of Zirconia (Zr) when used in combination with airborne particle abrasion (APA) better than when used with Hydrofluoric (HF) etching.

3. There for we can summarize from this study that use of airborne particle abrasion is better treatment for ceramic zirconia and the Z-prime plus (ZPP) is the best primer used with zirconia.

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10. DEDICATION

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