

Bonding to Zirconia (A Systematic Review)

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Abstract

The aim was set for this systematic review: to analyze the recent techniques of bonding to zirconia. Current literature of in vitro studies regarding bond strength achieved using different surface treatments on zirconia in combination with adhesive cement systems was selected from PubMed and systematically analyzed and completed with reference tracking. The total number of publications included was 557 studies. The surface treatments are divided into two main groups: Surface micro roughness techniques and chemical surface treatment. Conclusion, Fusion sputtering, Selective infiltration etching, Plasma oxyfluoride, Nano-alumina coating, Application of a tribochemical silica coating, Zirconia & metal primers or Zirconia particle deposition can result in sufficient bond strength for the bonding of oxide ceramics. Resin cements containing esteric organophosphate monomer (MDP) have shown in different studies a higher capacity of adhesion and stability after aging process; this is attributed to the capacity to bind metal oxides such as zirconium oxide (ZrO₂).

Keywords: Zirconia; Adhesive Cements; Bond Strength; Surface Treatment

Introduction

Zirconium oxide (ZrO₂), or zirconia, is a metal oxide that was identified as a reaction product of heating the gem, zircon, by the German chemist Martin Heinrich Klaproth in 1789 [1]. Developments over the last 10–15 years in ceramic materials science for dental applications have led to a class of high strength materials (i.e. zirconia-based ceramics) which potentially provide better fracture resistance and long-term viability when compared to porcelain and other inorganic, non-metallic alternatives. There is a wealth of information in the scientific literature regarding the use of zirconia (ZrO₂) in dental applications [2-4].

Zirconia is a polycrystalline material, which can exhibit structural polymorphism (monoclinic, tetragonal and cubic form) depending on pressure and temperature conditions [5]. The most useful mechanical properties can

be obtained when zirconia is in a multiphase form, known as Partially Stabilized Zirconia (PSZ) In the presence of a small amount of stabilizing oxides. The finely dispersed tetragonal ZrO₂ grains within the cubic matrix, provided that they are small enough, can be maintained in a meta stable state that is able to transform into the monoclinic phase [6].

Although superior in terms of mechanical performance (strength, toughness, fatigue resistance), there are some inherent problems associated with ZrO₂. One problem is with adhesion to the variety of substrates (synthetics or tissues) that can be encountered in dental or other biomedical applications. Conventional cementation/attachment techniques used with ZrO₂ components do not provide sufficient bond strength for many of these applications [7-9]. In recent years, ZrO₂ has gained attention as a biomaterial due to superior mechanical properties, compared to alumina (Al₂O₃), and

chemical and biological inertness that makes it very biocompatible [10]. Bonding to ZrO₂ has become a topic of great interest in recent years. [7,8,11-13].

Traditional adhesive chemistry is ineffective on ZrO₂ surfaces, since they are essentially non-polar and inert. Additionally, acid etchants like hydrofluoric (HF) do not sufficiently roughen the surface for simple micromechanical attachment. Surface abrasion with Al₂O₃ particles and application of a tribochemical silica coating allows for chemical bonds to a silane coupling agent and to resin cement. This is a somewhat complicated procedure that does not produce bond strengths as high as those reported for silane bonded porcelain [7,12,14].

The bonding of zirconia substructures should be based on both micromechanical and chemical bonding since the micromechanical retention supports chemical bonding and if bonding is based only on chemical compounds some debonding might happen in moist environments such as in the mouth [15].

Micromechanical retention is determined by the structure of the restoration's cementation surface. Depending on the manufacturing technique used, the cementation surface will differ in surface roughness, thus influencing the bond strength [16]. With a rougher surface, the size of the surface area and the surface energy will increase and in turn affect the wet ability, allowing the cement to flow into the micro retentions, thus creating a stronger micromechanical interlock [16, 17]. Machining of the surface or airborne particle abrasion can be used to increase surface roughness and consequently the bond strength of the oxide ceramic's cementation surface. Using these methods will increase the bond strength to oxide ceramics, but the roughening process applied might introduce micro-cracks into the ceramic and result in unintentional process-related flaws, cracks or grain pull-outs and subsequently phase transformation which will affect and reduce the strength of the ceramic [18-22].

The monomer 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) was originally designed to bond to metal oxides and its use has been extended to oxide ceramics [23]. MDP-containing resin cements seem to be the most appropriate due to the chemical interaction between the hydroxyl groups of the passive zirconia surface and the phosphate ester group of the MDP. Suggestions have been made that a chemical bond might be established between MDP and alumina or zirconia [17, 23-26]. Regular bisphenol-A-diglycidyl-methacrylate (bis-GMA) resin cements which do not contain the MDP itself might improve the bond strength to alumina and zirconia if they are combined with an

MDP-containing primer [27, 28]. Aim of this systematic review was to analyze the recent techniques of bonding to zirconia.

Methods

The research question was as follows: how to obtain strong and durable bond to zirconia?

Systematic literature search

Four databases were screened: MEDLINE (PubMed), science direct, springer and wiley online library from 2000 to 2016. The keyword related to the search strategy was (zirconia and bond).

Inclusion and exclusion criteria

- The material reviewed consisted of mainly laboratory studies and a small number of systematic reviews.
- Publications containing characterizations of zirconia materials after surface treatments were also included.
- All test methods for bond strength where the bond is tested (micro tensile, macro tensile, micros hear or macros hear).
- Studies in English language.
- Exclusion criteria were: descriptive studies, case reports, studies based on glass-infiltrated ceramic or endodontic posts or orthodontic brackets made of oxide ceramics, veneering porcelain and repair, and data were not presented in MPa, sample size of subgroup less than 5 and studies performed with Pull out test (Figure 1).

Study selection

Only papers that fulfilled all of the eligibility criteria were included, 557 titles retrieved from the electronic searched articles. After initial elimination, based on the titles and the abstracts, 80 papers were accepted for inclusion.

Data Extraction

Surface micro roughness techniques

Airborne-particle abrasion: Airborne-particle abrasion (APA) can be applied to metals and ceramics, [29,30] and hard dental tissues (enamel, dentin), [31] and has also been proposed for roughening the surface of zirconia as a way of increasing mechanical interlock and total contact area [32-36] (Table 1).

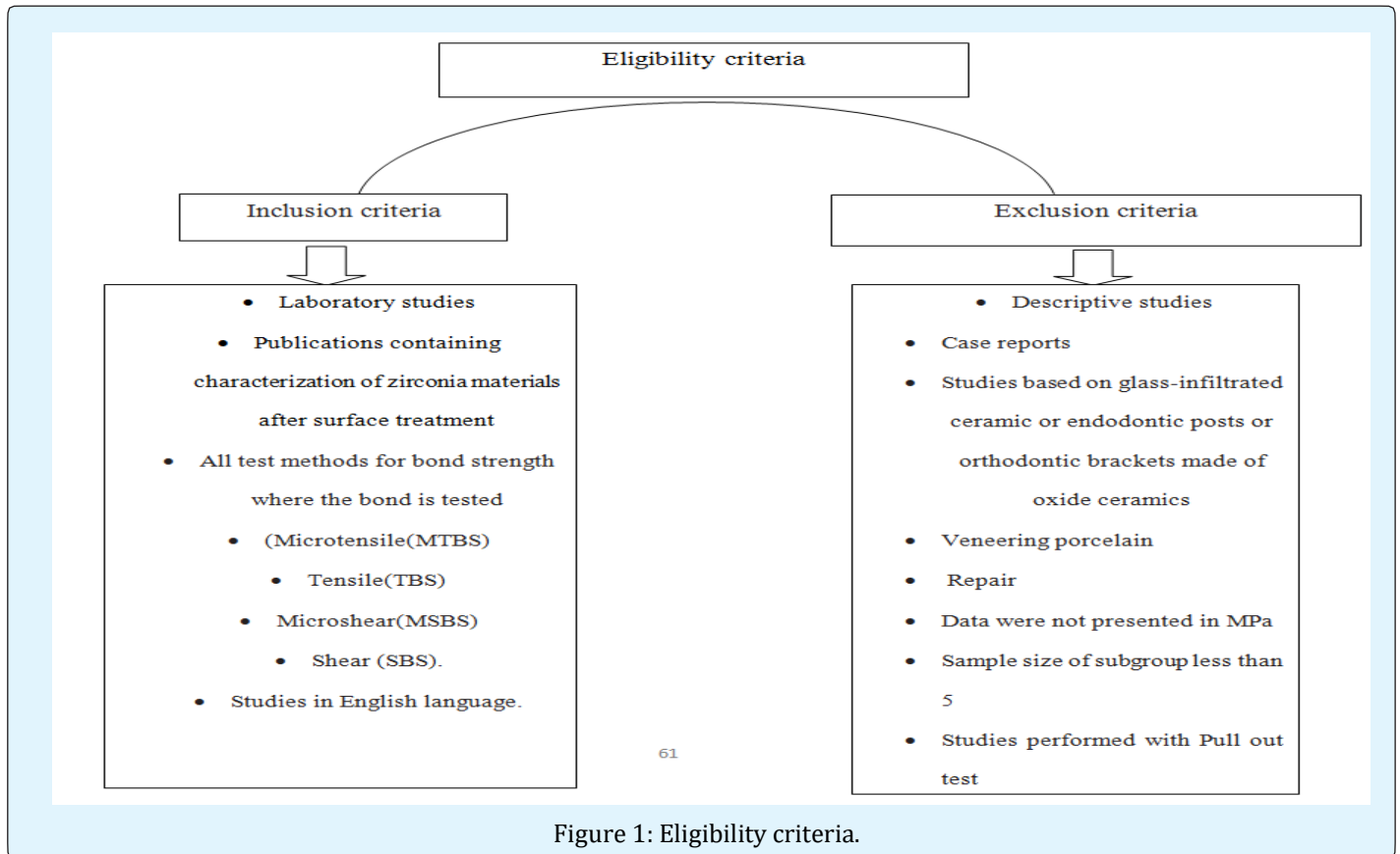


Figure 1: Eligibility criteria.

Grinding with disks and diamond rotary instruments:

The main disadvantage of grinding methods is the possible creation of micro cracks in the surface [37]. The high hardness of zirconia necessitates grinding with coarse diamond rotary instruments (120 to 200 μm grain size) [38].

Grinding conditions also seem important in that wet grinding with a 91- μm diamond wheel did not dramatically diminish flexural strength [39]. Coarse-grained diamond grinding produced a rougher surface than other techniques and improved bond strength but was not acceptable because it induces micro cracks and cause damage to zirconia surfaces [40, 41] (Table 2).

Selective infiltration etching (SIE)

SIE is based on inter-grain Meta stable tetragonal grains, created during thermal pre-stressing of the surface grains using a specific thermal regime. Due to this procedure, the bonding zirconia surface is ready to accept the adhesive resin which infiltrates and interlocks the bond [42]. SIE transforms dense, nono-retentive,

relatively smooth and low energy surface of zirconia to highly active and well bonding surface [43,44]. In SIE method the surface of zirconia is coated with a glass-containing conditioning agent silica (65% wt), alumina (15% wt), sodium oxide (10% wt), potassium oxide (5% wt) and titanium oxide (5% wt) with closely-matched thermal expansion coefficient to zirconia. Later on, the material is heated above glass transition temperature, until the optimal grain boundary diffusion is achieved. After cooling to room temperature, the glass is dissolved in an acidic bath to eliminate all traces of conditioning agent. The cooling and heating rates are controlled by a computer-calibrated induction furnace [45] (Table 3).

Coatings of low melting temperature porcelain micro pearls.

Another way to establish a firm bond between the zirconia surface and the resin luting material could be by fusing glass (porcelain) pearls to the zirconia surface. This treatment should, however, be done with caution in order not to jeopardize either the crown build up or the fit of the reconstruction to the tooth [46] (Table 4).

Fusion sputtering

Fusion sputtering is a new technique used to create rough zirconia surface by spraying an air-water jet carrying microscopic zirconia particles onto unsintered zirconia frameworks. Upon impact with the surface, the particles achieve good contact and adherence. After sintering, these particles become structurally fused with the underlying framework and create undercuts suitable for establishing mechanical retention with resin adhesives [47] (Table 5).

Chemical treatment (Hot acid)

This procedure based on corrosion-controlled process and metallic nature of pure zirconium. It selectively etches the zirconia and creates micro-retentions on the surface by modifying the grain boundaries through removal of the less arranged atoms [48]. Hot acid etching with combinations of highly corrosive acids (HNO_3 , H_2SO_4 , and HF) improved both initial bond strengths and durability [49]. An experimental hot hydrochloric acid (HCl) solution significantly increased roughness, basically a controlled corrosion process. The application of an HCl and Fe_2Cl_3 solution for 30 minutes enhanced the bond better than APA [50]. Sulfuric acid in solution with hydrogen peroxide (H_2O_2) (Piranha solution) appeared to have a positive effect on the bonding of zirconia with resin cements [51] (Table 6).

Effect of lasers

A neodymium-doped yttrium aluminum garnet (Nd:YAG) laser improved roughness and bond strength,[52-56] but the point of application left a silver spot or greatly increased the monoclinic phase at the surface (26.5% and 30.5%) [57].

A carbon dioxide (CO_2) laser

Its emission wavelength (2.3 to 10.6 μm) is absorbed by ceramics. Improved bonding was found after this laser application at a setting of 3 W and 4 W with various settings [58,59]. In a recent study, a CO_2 laser improved both roughness and the zirconia-porcelain bond [60].

An erbium-doped yttrium aluminum garnet laser(Er:YAG) laser

Er: YAG laser had been used for various clinical uses in operative dentistry, [61] and its action on high-strength ceramics had been studied extensively [19]. Also it has the ability to remove particles through ablation process by micro-explosions and vaporization. Laser effects obtained due to temperature changes (heating and cooling), induce phase transformation in the material leading to its damage. It has been suggested to lower the laser power

and to enable surface irradiation with constant water cooling. But, the results are still questionable regarding the fact that laser irradiation is not as effective in increasing bond strength as air-particle abrasion under an in vitro experiment [62,63] (Table 7).

Influencing chemical surface treatment

Silane coupling agents: Silanes increase the wetting capacity of an inorganic surface, allowing a better flow of resin cement across the surface and appear to enhance the micromechanical retention with low-viscosity resin cements [37]. One end of a silane molecule is organically functional (e.g., vinyl $-\text{CH}=\text{CH}_2$, amino $-\text{NH}_2$), and can polymerize with an organic matrix (e.g., a methacrylate). The other end is generally comprised of alkoxy groups (e.g., methoxy $-\text{OCH}_3$, ethoxy $-\text{OCH}_2\text{CH}_3$), which can react with a hydroxylated, silica containing surface, like porcelain. Silanes are commonly used in dentistry to coat glass filler particles in polymer matrix composites, to achieve adhesive bonding of porcelain (or other silica-containing ceramics) to resin luting cements for restorative applications. Silanes are also believed to promote surface wetting, which enhances potential micromechanical retention with low viscosity resin cements [13,20]. Silanes react with the zirconia powder in humid air or water to form Si-O-Zr linkages and stabilize t-phase [64,65]. They can be used alone or in combination with other surface treatments to increase bonding ability with resin cements [44,46] (Table 8).

Plasma oxyfluoride

This process presents a method to chemically modify zirconia by creating thin oxyfluoride conversion layer on its surface that is receptive to organosilane attachment. The recommended procedure is complex and involves fluorination of zirconia in a planar, inductively coupled plasma reactor, vacuum (≈ 35 mT) and handling of fluorine containing precursor gas. The goal of the process is to apply the fluorinated plasma to the zirconia surface, to convert the top 1-3 nm into a ZrO_3F_4 , [14,66] and significantly increased bond strength when combined with silane and resin cements containing MDP (Table 9).

Other surface coatings

Application of a tribochemical silica coating: Due to the lack of silica in ZrO_2 , silica-coating techniques have been explored to utilize the chemical bonding provided by silanization. The use of a tribochemical silica coating is a common practice for coating metal alloys and alumina- and zirconia-based dental ceramics with silica [67]. TBC is similar to airborne-particle abrasion, except that the aluminum oxide is coated with silica [67,68]. Silicon concentration at the surface increases significantly,

[11,68] but surface cleaning in an ultrasonic bath destroys this effect [69,70].

Increasing the pressure increased the roughness, the number of particles in contact with the surface, and the amount of silicon and eventually improved the bond strength [70] (Table 10).

Silicoating

The pyrolytic deposition of silicon to form a SiO_x-C coating with a thickness of 0.1 μm, [71] this surface can then be silanated to provide stronger bonds with metal and resin cements [72,73]. In this technique, with the use of special laboratory equipment, butane gas is burned with atmospheric oxygen and guided to a container filled with tetraethoxy silane. Via a thermal reaction, a layer of SiO_x-C is coated on the surface of the substrate [71].

The Silicoater system (Kulzer) commercially available in the market has yielded successful results for metals in several studies [74-75]. However, this technique is expensive and complex and is not suitable for standard application in dental clinical setting [37] (Table 11).

PyrosilPen Technology

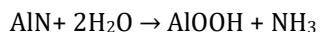
(SURA Instruments) is another silicoater designed for chair side use. Application of this device for zirconia ceramics has been evaluated as well. However, in order to be selected as a standard technique, it requires further investigations [37,71] (Table 12).

Plasma spray

Plasma spray is another type of silicoating to create a siloxane coat on the surface. Plasma is an ionized gas that contains ions, electrons, atoms and neutral spaces. Surface treatment by plasma spraying hexamethyl disiloxane produced a thin (<1μm) siloxane coating [46]. In molecular vapor deposition, zirconia specimens are exposed to 1-chloro silicide gas (SiCl₄) in the presence of water vapor for 15 minutes and produce an activated siliconized surface [14,76] (Table 13).

Nano-alumina coating

Nano-alumina coating is a simple and nondestructive method to improve the bond to zirconia [42]. It is based on the idea of a rapid precipitation of aluminium hydroxides that originate from the hydrolysis of AlN powder in a diluted aqueous suspension.



The result is heterogeneous nucleation of lamellar boehmite (γAlOOH) onto the surface of the immersed Y-

TZP substrate. The nano structured coatings consist of 6 nm thick and 240 nm long interconnected polycrystalline γAlOOH lamellas that grow perpendicular to the zirconia surface. During a heat treatment up to 900°C, these coatings are transformed into transient alumina, but without any change in the morphology [77] (Table 14).

Zirconia ceramic glazes (glaze-on technique)

The coating of surfaces with zirconia ceramic glazes (glaze-on technique) aiming at creating an intermediate etch able layer of the glaze material with low melting point for zirconia surface coating, and gave improved values in shear tests, with the main disadvantage being a large thickness (120μm) coating [78] (Table 15).

Zirconia particle deposition

Zirconia particle deposition using a milling residue suspension seems promising and effective as airborne particle abrasion [79,80] (Table 16).

A solid-gel process (sol-gel)

The sol-gel process has several advantages over the sandblasting method. Sandblasting potentially introduces surface defects or flaws. These surface irregularities generate compressive stresses on the surface layer, which adversely affect the long-term clinical performance of dental ceramics [81]. Another advantage is cost-related. Sol-gel processing requires less working space and less expensive equipment and chemical reagents as compared to the large and expensive sandblasting unit and silica-coated alumina sand particles used. But it is impractical because it takes many hours to create a silicate network in the surface (24 to 141 hours), [82] when compared with conventional TBC, this technique gives the same shear bond strength, higher silicon content, and better durability [83] (Table 17).

Fluorapatite/leucite glaze or salt glaze

Coatings of zirconia surfaces with fluorapatite/leucite glaze or salt glaze have proved unsatisfactory. Aggressive acid etching has produced similar bonds to conventional methods [84] (Table 18).

Molecular vapor deposition tool (MVD)

Piasick et al in 2009 used molecular vapor deposition tool (MVD) for deposition of vapor molecules in order to create a thin layer on zirconia surface chloro silane in combination with steam was used to create a reactive surface with Si-O_y groups and it was demonstrated that this technique could improve resin bond to zirconia surface [14] (Table 19).

Silica coating by silicon nitride hydrolysis

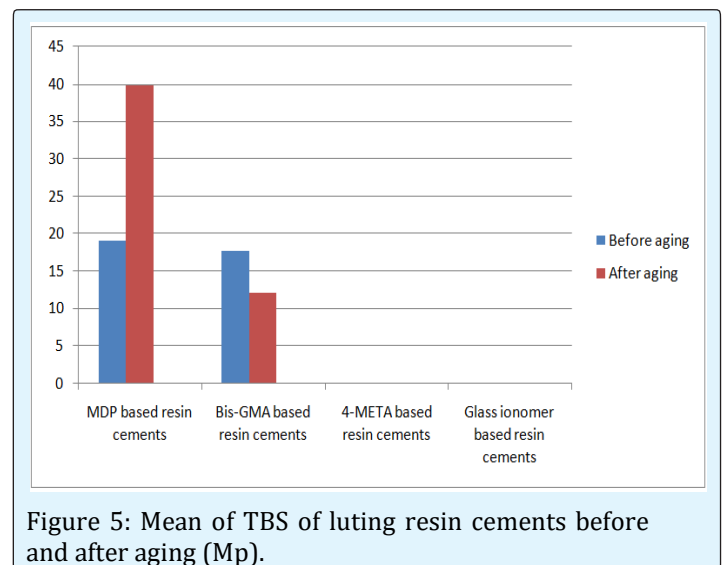
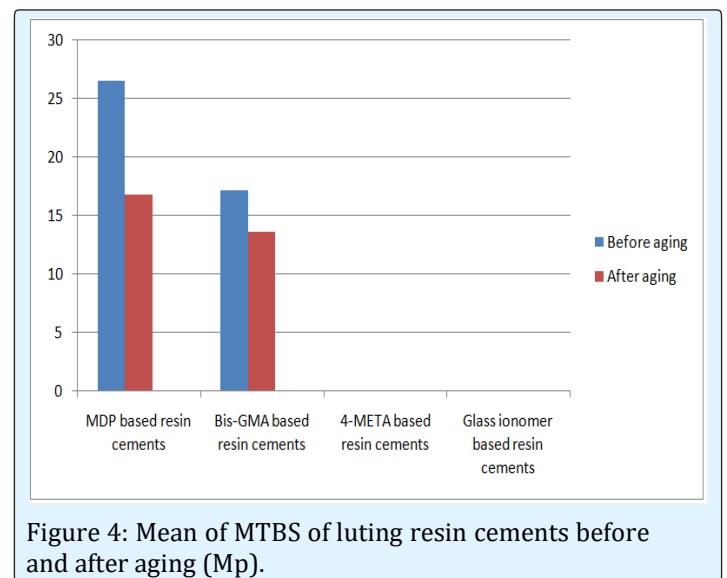
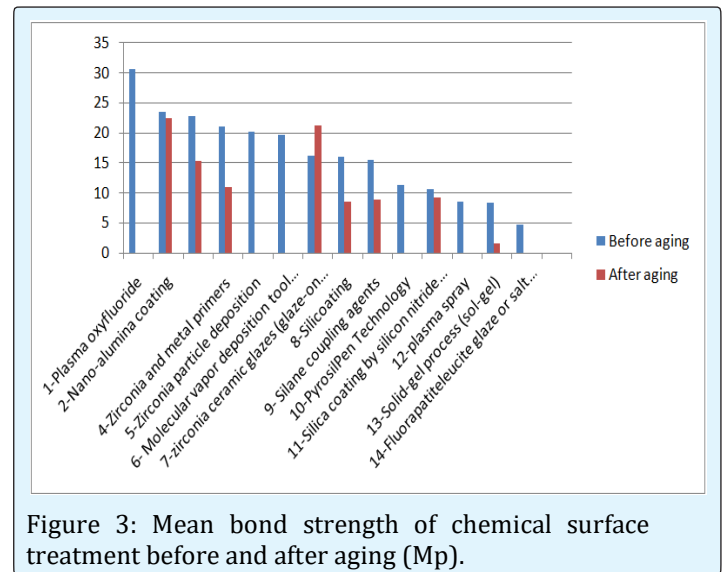
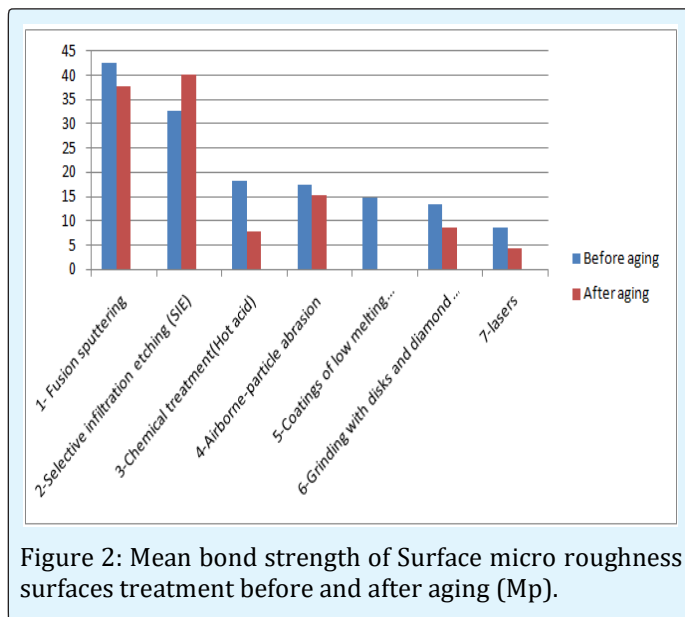
A silica coating is formed on zirconia surface by silicon nitride hydrolysis under alkaline condition. The hydrolysis of silicon nitride produces colloidal silica. The silica particles formed deposit on the zirconia surface. The coating is followed by a thermal treatment at 1400°C [85] (Table 20).

D- Zirconia and metal primers

The most frequent phosphate monomer groups used in resin cement or metal primers are the following: 10-methacryloyl oxydecyl dihydrogen phosphate, MDP (the adhesive monomer in Panavia F 2.0, Alloy Primer, Clearfil SE Bond/Porcelain Bond activator, Clearfil Ceramic primer), methacrylated phosphoric ester (adhesive monomer in RelyX Unicem), and phosphoric acid acrylate (the adhesive monomer of multilink automix) [86]. The VBATDT (6-(N-(4-vinylbenzyl) propylamino)-1, 3, 5-triazine-2, 4-dithione) monomer (Alloy Primer, Total bond) [87] (Table 21).

Results

Table 22, 23 & (Figures 2-7) showing the mean of bond strength of different surfaces treatment and luting cements before and after aging.



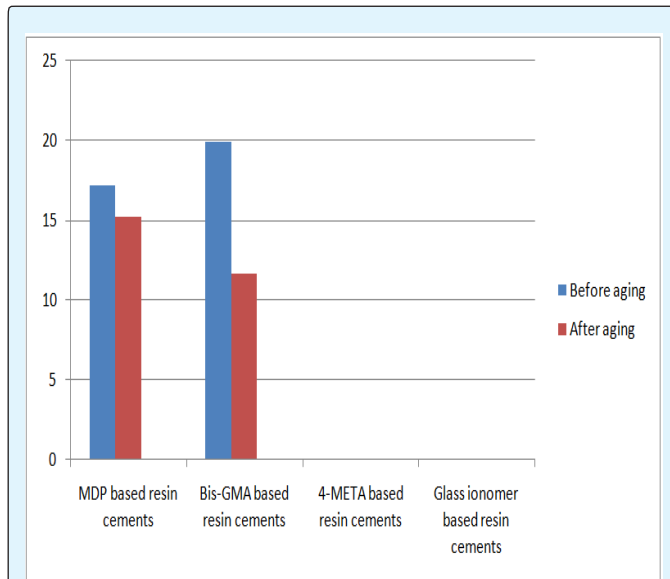


Figure 6: Mean of MSBS of luting resin cements before and after aging (Mp).

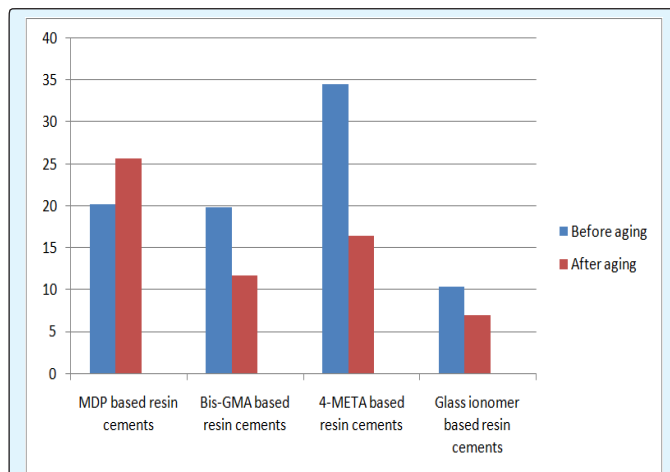


Figure 7: Mean of SBS of luting resin cements before and after aging (Mp).

Literature identification: The searches in the database identified a total of 557 publications of which abstracts were retrieved for evaluation. Applying the inclusion and exclusion criteria reduced the number of relevant publications to 80.

Interpretation of data on surface treatments of zirconia bond strength: The surface treatments are divided into two main groups: Surface micro roughness techniques and chemical surface treatment. Each group has its individual subdivision of surface treatments; the

cementation procedures and artificial aging were considered in the evaluation.

Sufficient bond strength is defined by the values achieved that are equal to or above 20 MPa regardless of the test methods that have been used [46].

Surface micro roughness techniques

Airborne particle abrasion: Airborne particle abrasion or sandblasting can be carried out using aluminum oxide particles with different particle sizes, air pressure during blasting and distance. It is one of the most common mechanical surface roughening treatments (n=36 of 80 included). Number of included studies: 36/80.

Mean values range: MTBS test (8.9-35.9 Mp before aging and 0 or debonding to 34.7 Mp after aging) [43,88,89] TBS test (42.5-44.1 Mp before aging and 27.5-40.1 Mp after aging) [90,91] MSBS test (10.12- 37.3 Mp before aging) [92, 93] SBS test (3.07-33.87 Mp before aging and 0-24.34 Mp after aging) [94-97].

Sufficient bond strength: Regardless of the test method 13/28 studies using airborne particle abrasion showed sufficient bond strength values before aging [11, 91, 93-103], and 5/23 after aging [43,90,91,95,103].

Grinding with disks and diamond rotary instruments: Number of included studies: 4/80.

Mean values range: SBS test (4.2-25.5Mp before aging and 4.4-24.38 Mp after aging) [57, 95, 104].

Sufficient bond strength: Regardless of the test method 1 /3 studies using grinding showed sufficient bond strength values before aging and none of studies 0/4 sufficient bond strength values after aging [95].

Selective infiltration etching: Number of included studies: 5/80.

Mean values range: MTBS test (28-51.9 Mp before aging and 15 to 55.9 Mp after aging) [43, 44, 98,119].

Sufficient bond strength: Regardless of the test method 5/5 studies using Selective infiltration etching showed sufficient bond strength values before aging [43,44,92,98,105], and 2/3 after aging [100,106].

Coatings of low melting temperature porcelain micro pearls: Number of included studies: 1/80

Mean values range: SBS test (11.3-18.4 Mp before aging) [46].

Sufficient bond strength: The study did not show sufficient bond strength values [46].

Fusion sputtering: Number of included studies: 1/80

Mean values range: MTBS test (42.5 Mp before aging and 37.9 Mp after aging) [47].

Sufficient bond strength: The study showed sufficient bond strength values before and after aging [47].

Chemical treatment (Hot acid): Number of included studies: 7/80

Mean values range: MSBS test (11.04 Mp before aging) (63) SBS test (0.1-46.71 Mp before aging and 0.1-13.48 Mp after aging)[49, 97, 104].

Sufficient bond strength: Only 1 /6 studies using Chemical treatment showed sufficient bond strength values before aging [97].

Lasers: Number of included studies: 13/80

Mean values range: MTBS test (3-15.8 Mp before aging and 0 Mp or debonding after aging) [44, 69] MSBS test (2-29 Mp before aging and 0-1.8 Mp after aging) [93,107] SBS test (1.76-18.12 Mp before aging and 3.7-9.2 Mp after aging) [96-110].

Sufficient bond strength: Regardless of the test method 1 /12 studies using lasers showed sufficient bond strength values before aging [93].

Chemical surface treatment

Silane coupling agents: Number of included studies: 6/80

Mean values range: MTBS test (13.7-31.5 Mp before aging)[100] SBS test (6.8-31.2 Mp before aging and 0-20.8 Mp after aging)[94,111,112].

Sufficient bond strength: Regardless of the test method 2 /6 studies using Silane coupling agents showed sufficient bond strength values before aging [94,100] and 1/4 after aging [111].

Plasma oxyfluoride: Number of included studies: 3/80

Mean values range: SBS test (22.9-37.5 Mp before aging) [66,113,114].

Sufficient bond strength: All SBS studies 3/3 studies using Plasma oxyfluoride showed sufficient bond strength values before aging. [66,113,114].

Tribochemical silica coating: Number of included studies: 23/80

Mean values range: MTBS test (6-27.2 Mp before aging and 3.4- 26.8 Mp after aging)[88, 99,115,116]TBS test (38.3 Mp before aging and 16.5 Mp after aging)[91] MSBS test (10- 37.4 Mp before aging and 1.3-20.5 Mp after aging)[107, 117] SBS test (2.5-63.4Mp before aging and 1.4-73.8 Mp after aging)[96,118].

Sufficient bond strength: Regardless of the test method 9/18 studies using a Tribochemical silica coating showed sufficient bond strength values before aging[11,91,95,99,116-119], and 4/16 after aging [115,117,118,120].

Silicoating: Number of included studies: 2/80

Mean values range: SBS test (4.1-30.9 Mp before aging and 0.01-25.2 Mp after aging)[37-42, 44, 46-75, 83, 85, 88, 89, 91-93, 95-97,103-114,116-119,121-135].

Sufficient bond strength: One study 1/2 studies using Silicoating showed sufficient bond strength values before and after aging [95].

PyrosilPen Technology: Number of included studies: 1/80

Mean values range: SBS test (9-16 Mp before aging) [71].

Sufficient bond strength: The only study using PyrosilPen Technology did not showed sufficient bond strength values before aging [71].

Plasma spray: Number of included studies: 2/80

Mean values range: SBS test (3.5-16.6 Mp before aging) [46,136].

Sufficient bond strength: The two studies using plasma spray did not showed sufficient bond strength values before aging [46,136].

Nano-alumina coating: Number of included studies: 2/80

Mean values range: SBS test (17.8-24.4 Mp before aging and 20.1-27.4 Mp after aging) [42,122].

Sufficient bond strength: The two studies using Nano-alumina coating showed sufficient bond strength values before and after aging. [42,122].

Zirconia ceramic glazes (glaze-on technique): Number of included studies: 4/80

Mean values range: MSBS test (17.4- 25.1 Mp before aging)[137] SBS test (4.9-27 Mp before aging and 3.7-34 Mp after aging)[78,138,139,140].

Sufficient bond strength: Regardless of the test method 2/4 studies using zirconia ceramic glazes (glaze-on technique) showed sufficient bond strength values before aging [78,137] and 1/2 showed sufficient bond strength values after aging [78].

Zirconia particle deposition: Number of included studies: 1/80

Mean values range: MTBS test (19-21.5 Mp before aging) [80].

Sufficient bond strength: The only study using Zirconia particle deposition showed sufficient bond strength values before aging [80].

A solid-gel process (sol-gel): Number of included studies: 3/80

Mean values range: SBS test (4.5-14 Mp before aging and 0.06-3.6 Mp after aging)[82, 83,141].

Sufficient bond strength: The three studies using a solid-gel process (sol-gel) did not showed sufficient bond strength values before and after aging [82, 83,141].

Fluorapatiteleucite glaze or salt glaze: Number of included studies: 1/80

Mean values range: SBS test (4.7Mp before aging) [84].

Sufficient bond strength: The only study using Fluorapatiteleucite glaze or salt glaze did not showed sufficient bond strength values before aging [84].

Molecular vapor deposition tool (MVD): Number of included studies: 1/80

Mean values range: MTBS test (16.2-23.2Mp before aging) [14].

Sufficient bond strength: The only study using Molecular vapor deposition tool (MVD) showed sufficient bond strength values before aging. [14].

Silica coating by silicon nitride hydrolysis: Number of included studies: 1/80

Mean values range: SBS test (9.3-11.4 Mp before aging and 5.9-12.2 Mp after aging) [85].

Sufficient bond strength: The only study using Silica coating by silicon nitride hydrolysis did not showed sufficient bond strength values before aging [85].

Zirconia and metal primers: Number of included studies: 14/80

Mean values range: MTBS test (6.1-17.4 Mp before aging and 11 Mp after aging) [134,142] TBS test (7.1-22.3 Mp before aging) [35,143] MSBS test (20.4- 34.5 Mp before aging) [101,102] SBS test (7.5-38.4Mp before aging and 0.1-31.8 Mp after aging)[66, 94,144].

Sufficient bond strength: Regardless of the test method 8/13 studies using Zirconia and metal primers showed sufficient bond strength values before aging [35,66,94,101,102,140,145-147], and 2/5 after aging [94,123].

Interpretation of data on resin cements and zirconia bond strength: Resin cements are divided into four main groups according to main chemical composition: MDP based, Bis-GMA based, 4-META based and Glass ionomer based.

MDP based resin cement:

Mean of MTBS (26.5 Mp before aging and 16.8 Mp after aging).

Mean of TBS (19.1 Mp before aging and 40 Mp after aging).

Mean of MSBS (17.2 Mp before aging and 15.3 Mp after aging).

Mean of SBS (20.3 Mp before aging and 25.75 Mp after aging).

Sufficient bond strength: MTBS showed sufficient bond strength before aging.

TBS showed sufficient bond strength after aging.
SBS showed sufficient bond strength before and after aging.

Bis-GMA based resin cement:

Mean of MTBS (17.2 Mp before aging and 13.6 Mp after aging).

Mean of TBS (17.66Mp before aging and 12.2 Mp after aging).

Mean of MSBS (20.9 Mp before aging and 11.6 Mp after aging).

Mean of SBS (19.9 Mp before aging and 11.7 Mp after aging).

Sufficient bond strength: MSBS showed sufficient bond strength before aging.

4-META based resin cements: Mean of SBS (34.5 Mp before aging and 16.5Mp after aging).

Sufficient bond strength: SBS showed sufficient bond strength before aging.

Glass ionomer based resin cements: Mean of SBS (10.4Mp before aging and 7 Mp after aging).

Sufficient bond strength: SBS did not show sufficient bond strength before or after aging.

Discussion

The results of this systematic review showed that there were large variations between surface treatments for zirconia and different test methods that evaluate the bond strength between zirconia and adhesive cement systems, making it difficult to compare studies. Two main groups were defined from the literature: Surface micro roughness techniques and chemical surface treatment. Each group has its individual subdivision of surface treatments. The bond strength was defined as clinically sufficient, regardless of the method used, when the values were equal to or above 20 Mp. It is always difficult to define what sufficient is from a clinical perspective but 20 MPa has proved clinically sufficient, for example, compared to the lower limit of bond strengths between resins and enamel [148].

The bond strength between adherent materials can be tested using various test methods. The most common test methods for evaluating the adhesive bond strength are the SBS or TBS test [7,149]. The advantage of the SBS test is that it is easy to use. One of the drawbacks, however, is

that the test's reliability is questioned [149]. The advantage of the TBS test is the small amount of material consumed when the test is performed and the fact that the stresses are less complex, that is, a more even stress distribution can be obtained. One of the drawbacks is production of the specimens, and attachment during testing is crucial since it may produce inhomogeneous stress distribution and affect the results [150]. Micro-specimens and MSBS or MTBS tests have been developed to overcome the drawbacks [151]. Despite the difficulties associated with cutting and testing micro-bars and the technical difficulty of the equipments employed MTBS remains a powerful tool for evaluating bond strength values [47].

Two of Surface micro roughness techniques (Fusion sputtering and Selective infiltration etching) show the highest values (42.5 and 32.8 Mp before aging, 37.9 and 40.3 Mp after aging) of all the surface treatments included. The values are well above the criteria set and can be defined as sufficient to achieve clinically relevant bond strength. The created surface beads become part of the framework and create 3-dimensional undercuts suitable for establishing micromechanical retention with the resin cement also the increased surface area of bonding, which explains the high MTBS values between the resin cement and fusion sputtered specimens [43]. The surface topography created by the SIE technique created a highly retentive surface where the adhesive resin was directly able to penetrate and interlock, created inter-grain porosities resulting in significantly higher bond strength [43].

Regarding chemical surface treatment (Plasma oxyfluoride, Nano-alumina coating, Application of a tribochemical silica coating, Zirconia & metal primers and Zirconia particle deposition) show sufficient bond strength values ≥ 20 Mp before aging (30.7, 23.5, 22.8, 21, 20.2 Mp) respectively. The plasma fluorinated surface demonstrated the lowest contact angle. Earlier work described the altering of zirconia's chemical bonding structure at the surface to a more ionic and reactive structure, a mixture of fluorides and oxyfluorides [70]. Pantano and Brow confirmed this model by showing the auto-hydroxylation of fluoride and oxyfluoride phases in the presence of water. When hydrolyzed surface reacts with the phosphate ester-containing monomer in the adhesive hydroxyl groups on the ester can form hydrogen bonds with surface hydroxyl groups. This increase in reactive sites on the surface was shown to translate into high bond shear strengths and is expected to translate into higher restoration lifetimes [152]. The application of the alumina coating (non-invasive surface

functionalization) is the main factor in the increase of the surface area [42,122].

Tribo chemical silica coating systems creating a fine rough surface that increases the surface area and, thus, enhances the mechanical and chemical bonding. These systems also produce a silica layer on the ceramic surface processed with high speed surface impaction of alumina particles modified by silica, forming a chemical bond between the ceramic surface and the silane agent. The silane agent used for silanization also contributes to the bond strength by promoting a chemical bond to resinous materials via cross-linkages with methacrylate groups, and also increases the substrate surface energy and improves the surface wettability to resin [68].

Mesoporous zirconia was produced by mixing $Zr(SO_4) \cdot 2.4H_2O$ with C16TMABr at a mole ratio of 0.32:1. During this procedure, the mesoporous zirconia was subjected to a thermal heat treatment at 500°C for 6 h. After sintering, the template of C16TMABr was removed and the mesoporous zirconia was characterized as a porous structure exhibiting favorable compatibility with the zirconia blocks. The modified surface possessed a rough mesoporous structure. This provided interlocking sites for the resin cement allowing it to penetrate and fill the mesoporous structure, generating a hybrid layer of resin cement and zirconia at the interface. Therefore, the superior characteristics of the material guarantee that a mesoporous zirconia coating is an effective mechanical method to improve the bond strength with resin cement [80].

The presence of phosphate monomers in primers allows the formation of covalent bonding to the zirconia surface and the copolymerization to methacrylate groups in resin cements by photo-activation [147]. Chemical modification with acidic adhesive monomers creates a "reactive" Y-TZP surface, which then facilitates chemical bonding between functional groups in acidic adhesive monomers and metal oxide groups on Y-TZP surface. Adhesive monomers usually contain a carbonyl group [$-C(=O)OH$] or phosphoric acid group [$-P(=O)(OH)_2$] [18-21], such as the 10-MDP monomer. A dehydration reaction occurs between phosphoric acid groups in adhesive monomers and oxygen atoms on zirconia surface, resulting in strong covalent bonding of 10-MDP to zirconia [143].

The use of new activators (zirconia primers) helps surface wetting by reducing the contact angle, but significantly less than fluorine plasma spraying [66]. A new formula of universal primer which contains a silane

and a phosphate monomer has been promoted (3-alcohol solution of methacryloxypropyl-trimethoxy silane, phosphoric acid methacrylate and sulphide methacrylate-Monobond Plus) In combination with silica coating gave high bond strength values. It creates promising bond strength to zirconia after artificial aging when used after air-borne particle abrasion with ultrasonic cleaning or after silica coating [91].

A new zirconia primer containing organophosphate monomers and carboxylic monomers (Z-Prime Plus; Bisco Inc) was compatible with many resin cements and had a positive effect on bonding with resin cements after APA with 50- mm alumina [144,153]. The AZ Primer (Shofu Dental Corp) containing phosphonic acid monomers (6-MHPA) gave a better bond than other silane primers [35]. Metalprimer II (GC Corp) containing adhesive monomer thiophosphoric methacrylate (MEPS), gave a better and more aging resistant bond than ceramic primer (GC Corp) [154].

Another new specialized primer is Signum zirconia bond (Heraeus Kulzer GmbH), which appears even more effective [155]. Even without previous APA treatment, a novel universal primer containing both MDP and methacrylates promotes high bond strength [143]. Regarding to resin cements used, the MDP based resin cements showed sufficient bond strength before and after aging also 4-META based resin cement showed sufficient shear bond strength before aging. There is evidence supporting the good bond strength of zirconia with the use of resin cements containing (MDP). The adhesive functional monomers are believed to have the ability to form chemical hydrogen bonds with metal oxides at the resin/zirconia interface, improving the wettability. On the other hand, the compositions of resin cement, for example, large filler size and high viscosity could affect the wettability significantly [25, 32,120]. The role of the inorganic compounds in MDP resin cements is important in creating resistance to hydrolysis [45]. Without surface treatment, an MDP-metal primer (Alloy primer; Kuraray Co Ltd) appears to improve the chemical bonding with the resin cement [87,156,157].

A primer with MDP and a coupling agent for zirconia were mixed in various proportions and found to improve the bond to resin cements not containing MDP [13]. Lorenzoni et al 2012 applied basic NaOH solution to zirconia surface and suggested that application of NaOH alone or prior to MDP based primers can increase bond strength by increasing surface reactivity/functionalization and availability of OH groups. However, this increase in bond strength was less than the

clinically acceptable threshold [158]. The anhydride group present in 4-methacryloxyethyl trimellitate anhydride (4-META) and phosphoric methacrylate ester monomers also bonds to zirconia. However, studies have reported that the bond strength of resin cements containing 4-META after aging was less than the required threshold [7,118].

Effect of aging on bond strength

The hydrolytic action of water on adhesive surfaces and the inhibition reaction phenomena due to the presence of moisture are the main reasons for up to (50%) reductions from the baseline bond strength [105]. Water thermo cycling causes repeated thermal expansion and contraction of the materials used, which causes fatigue at the inter phase and therefore a reduction in bond values [159]. De Castro et al (54) observed that the aging process did not significantly affect the bond strength in tensile testing, regardless of the type of luting agent. With MDP resin cements and surface pretreatment, no significant changes were found after hydrothermal recycling [39,113]. In contrast, in polished zirconia surfaces, many spontaneous detachments occurred after an aging process, despite the influence of activators [160].

Artificial aging gives a measure of bond durability and is done either by water storage or thermal cycling with thermo cycling having a greater impact than water storage at a constant temperature [161]. In the literature, there is no consensus on a regimen for artificial aging and the cycles are set arbitrarily, ranging from 100 to 50000, which make it difficult to compare results. Gale and Darvel concluded that 10,000 cycles correspond to approximately 1 year of clinical function [162].

Moreover, a meaningful test of ceramic bonding should involve cyclic loading, high numbers of low load chewing cycles and water storage for at least 6 months [163,164]. All these investigations were performed under controlled laboratory conditions and no clinical trials were done to validate the results. A decrease in the resin-ceramic bond strength value after artificial aging was observed for many commercial systems in several studies [9,27,32,68,70,98,116]. Several other studies, however, did not subject their specimens to artificial aging or the aging period was too short to simulate clinical conditions (11,14,35,37,44,46,65,66,68,106,145,146). An increase in the resin-ceramic bond strength value after artificial aging was observed for many commercial systems in several studies [43,49,98,104,112,121].

Conclusion

Based on the findings and within the limitations of this study, the following can be concluded:

1. Fusion sputtering, Selective infiltration etching, Plasma oxyfluoride, Nano-alumina coating, Application of a tribochemical silica coating, Zirconia & metal primers or Zirconia particle deposition can result in sufficient bond strength for the bonding of oxide ceramics. This conclusion, however, needs to be confirmed by clinical studies.
2. APA is a reference method included in most research protocols.
3. Adhesive monomers are necessary for chemical bonding.
4. Aging have negative effects on adhesion to zirconia.
5. Resin cements containing esteric organophosphate monomer (MDP) have shown in different studies a higher capacity of adhesion and stability after aging process; this is attributed to the capacity to bind metal oxides such as zirconium oxide (ZrO₂).
6. 4-META based resin cement showed sufficient shear bond strength before aging.
7. Glass-ionomer based resin cements and Bis-GMA based cements have shown a low adhesion.

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