Current scenario on adhesion to zirconia; surface pretreatments and resin cements: A systematic review

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Abstract

Several methods have been proposed to increase bonding of zirconia with resin. However, we are still to find the Holy Grail. A systematic literature review was performed through PubMed on international literature from January 2000 to May 2021 with relevant Medical Subject Headings terms. 56 articles were found to be relevant. Of all the different methods proposed, mechanochemical pretreatment of zirconia surface with alumina oxide and use of 10-methacryloyloxydecyl dihydrogen phosphate were found to be most effective as per majority of studies. New methods that require further research also surfaced.

Keywords: Resin cement, surface pretreatment, zirconia

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INTRODUCTION

In the second half of the 20th century, dentistry faced challenges as to meet the escalated esthetic needs. With the advent of glass ionomer cement and composite resins, esthetic dentistry reached a new height. Similarly, metal-ceramic restoration slowly lost its popularity and the time had come for a metal-free era. With the introduction of zirconia in dentistry, bigger possibilities emerged in the field of indirect restorations. However, from the start of the new millennium, a new question had arrived – how to bond the zirconia restorations to the tooth.

In the past 20 years, innumerable researches have been conducted to establish a possible solution for achieving a predictable bonding between tooth and zirconia. However, a single method is yet to be declared the "gold standard." The aim of this article is to systematically review the various studies dealing with zirconia bonding and to draw a conclusion as to which method is the best to date.

MATERIALS AND METHODS

This study was performed through the search engine PubMed on international literature. Studies published from January 2000 to May 2021 were searched. Keywords were zirconia, ceramic surface treatments, zirconia adhesion, MDP, bond strength test, resin bonding. These Medical Subject Headings (MeSH) were used individually or in combination. The literature search was performed by two independent reviewers.

The inclusion criteria were English language publication, *in vitro* studies, reviews, studies performing micro/macro, and shear/tensile bond strength tests. The exclusion criteria were case reports, clinical trials, studies with less than five

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samples, and studies without thermocycling and moisture storage. Any disagreement regarding the eligibility of the studies was resolved through discussion. Only articles pertaining to dentistry were considered.

RESULTS

The search carried out in PubMed identified 63 articles primarily. After screening the titles and abstracts, 45 articles were selected as relevant.

Then, with other MeSH or keywords, following results were obtained [Table 1].

Of the total 90 articles, 22 were repetition. Sixty-eight articles were finally selected and read, along with their relevant references. Twelve articles were further excluded and 56 articles remained.

DISCUSSION

Bonding to traditional silica-based ceramics, employing mechanical and adhesive retention, is well researched and bond strengths are predictable. While hydrofluoric acid (HF) etching along with methacryloxypropyl trimethoxysilane (MPS) application is a commonly recommended method for roughening the surface of silica-based ceramics and increasing their wettability,[1] zirconia is a polycrystalline nonetchable material.[2-6] Owing to its chemical inertness, cementation of zirconia indirect restorations has been problematic over the years. Thus, researchers have attempted to come up with various methods to overcome this handicap. This article aims to review all such employed techniques.

Factors that are assessed when considering adhesion of zirconia to any substrate are zirconia surface pretreatment, resin cement used, artificial aging, and the bond strength test performed and are discussed accordingly.

Zirconia surface pretreatment

Majority of the studies agree that zirconia surface needs to be modified before applying the luting cements since all the pretreatments increased bond strength. In this review, pretreatment techniques are classified into three groups:

- Mechanical
- Chemical
- Mechanochemical.

All studies are equivocal on the need of a contaminant-free surface before any treatment. Most studies started the surface conditioning by polishing zirconia with paper sprays or milling cutters of silicon carbide. Ultrasonic cleaning before conditioning is also considered a beneficial method.[7-21] Several solutions were used that include distilled water, alcohol, acetone, and ethanol.

Mechanical

These methods aimed to modify the zirconia surface so as to either roughen it to enhance micromechanical retention or deposit various compounds (mainly silica) on its surface so as to make it suitable for bonding. They are discussed subsequently.

Sandblasting

Sandblasting with alumina particles increased bond strength by increasing surface energy, wettability, roughness, and the appearance of hydroxyl groups, which facilitate bonding with the primer/universal adhesive/cement.[10,13,15,20,22-24] Particles with size ranging from 25 to 110 μm at 0.5–4 bar for 10–20 s were used.[25-27] Bond strength was not affected by varying particle size despite the difference in surface roughness created.[28-31] However, an increase in particle size and pressure has long been associated with the formation of microcracks and weakening the mechanical properties of zirconia.[11,13,21,32-39] It has also been reported that sandblasting before sintering caused fewer phase transformations than after sintering. However, sandblasting before or after sintering had no influence on adhesion.[18,19]

Recent *in vitro* studies report that airborne particle abrasion (APA) may have a deleterious effect on the zirconia surface due to the creation of microcracks which might reduce the flexural strength.[40] Moreover, the tetragonal phase of Y-TZP is converted to the monoclinic phase with volume expansion (4%–5%) under the high stresses caused by this abrasion, and this unique transformation can produce different types of damage that affect the structural integrity and material reliability.[41,42] While this process may result in an increase in the crack propagation resistance of Y-TZP for a certain period, functioning as a toughening mechanism,[43] the presence of the unstable and stressful monoclinic structure makes the zirconia in this phase fragile, thus increasing the fracture tendency over longer term. The tetragonal (t)-monoclinic (m) phase

Table 1: Results obtained with other Medical Subject Headings/keywords

Keywords	Total received paper	Total selected paper
Zirconia surface treatment effect on bond strength	14	8
Zirconia-resin cement bond strength with thermocycling (*in vitro*)	31	20
Zirconia adhesion review	54	17
Total	99	45

transformation is directly related to abrasive particles' size.[44]

Silica coating

Zirconia has silica-free surface and possesses relatively nonpolar surface. They are more chemically stable than silica-based ceramics, so traditional silane treatment is not usually effective on zirconia.[45] Silica coating techniques have been explored to convert silica-free into silica-rich zirconia surface for utilizing the chemical bonding provided by silanization.

Silicoater[45] technology is a method to impregnate silica pyrolytically on a substrate surface, followed by application of silane, before bonding using resin cement. However, it proved to be too expensive and complex and thus commercially nonviable.

Tribochemical silica coating (i.e., Rocatec or CoJet systems) (TSC) is a commonly used commercial technique in which zirconia surface is air abraded with alumina particles that have been coated with nano-silica, resulting in the impregnation of nano-silica into the zirconia surface. Studies have shown that tribochemical silica coating followed by silanization has resulted in enhanced initial bond strengths between zirconia and resin materials.[46-50] However, it is not clear whether it was caused by silica coating or the surface roughening effect of air abrasion.

Some studies have shown that similar effects were obtained with tribochemical silica coating/silanization and regular air abrasion with alumina particles on improving zirconia–resin bond strengths, thus indicating tribochemical silica coating only provided air–abrasion effect for creating surface roughness.[48,51] It has also been reported that tribochemical silica coating does not provide stable resin–zirconia bond strength,[52] probably because silica was not strongly attached to zirconia surfaces. EDXS analysis and SEM studies showed that the silica coated on zirconia surface could be cleaned away by ultrasonication in water or pressurized water spray,[53] indicating that no stable chemical bond was formed between silica and zirconia. The silica was probably deposited on the zirconia surface via weak physical force, such as Vander–Waals forces, which might not be strong and stable enough in a clinical situation.

On the other hand, for some researchers, TSC showed better bond strength than conventional sandblasting, favoring long-term stable adhesion.[11,22,38,39,54]

It has also been reported that when zirconia was air abraded with aluminum oxide (Al_2O_3) (110 lm), it resulted in higher roughness values, but air abrasion protocols with silicon dioxide (SiO_2) (110 lm; Rocatec) promoted better adhesion to 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based resin cement.[55]

Air abrasion with alumina is essential to obtain durable bonding of resin cement to highly translucent partially stabilized zirconia and yttria-stabilized tetragonal zirconia polycrystals (Y-TZP). Different air abrasion conditions affected the bond strength of resin cement, in the case of Y-TZP air abraded with 50-μm alumina at 0.2 MPa and 30-μm alumina at 0.12 MPa. When alumina air abrasion was used to treat the inner surface of zirconia crowns, even with larger particles, the system behaved as a bonded crown, promoting a higher fatigue resistance for the cemented crowns.[56]

There are other methods for silica coating such as modification of zirconia surface by utilizing flame treatment with tetraethoxy silane containing butane as fuel gas,[57] gas-phase chloro-silane pretreatment,[58] and sol–gel process silica coating.[59] However, further investigations into these techniques are required before clinical recommendation.

Laser

The application of lasers to the surface of zirconia is based on the same principle as sandblasting, i.e., obtaining a rough surface and increasing its wettability that allows micromechanical retention with the resin.[16] Different types of lasers have been described (Er:YAG, Nd:YAG, Yb:YAG, CO_2), with different parameters of power, energy intensity, distance, and duration. Most of the studies concluded that the application of laser did not increase the bond strength compared to sandblasting and did not obtain acceptable adhesion values,[8,12,15,60] due to the appearance of microcracks on the surface of the zirconia, leading to a phase transformation and weakening of mechanical properties.[60] Therefore, laser is not currently considered a valid mechanical pretreatment tool.[8,15] However, there have been reports where application of Er, Cr:YSGG laser with adjusted parameters on zirconia appeared to be useful as a nondestructive surface treatment method.[44]

Acid etching

It is well-established fact that unlike glass ceramics, acid etching is not effective for polycrystalline ceramics such as zirconia and alumina as they did not undergo significant structural change after HF acid etching.[61]

Hence, silanization and acid etching are not effective on zirconia because it is inert and without glassy matrix on which these agents act.[62]

Plasma spraying

Plasma has been used to increase the surface energy and alter the surfaces of the substrates without affecting their structural properties. However, the application of oxygen or argon plasma did not obtain good adhesion values after artificial aging, which added to the appearance of impurities on the surface of zirconia and indicated its susceptibility to hydrolytic degradation.[21,63,64]

Selective infiltration etching

It is based on the principle of heat-induced maturation and grain boundary diffusion and transforms the relatively smooth nonretentive surface of Y-TZP into a highly retentive surface. It also creates a three-dimensional retentive feature where the adhesive resin can infiltrate.[65]

Studies reported that selective infiltration etching (SIE), based on ceramic infiltration by molten silica and other oxides, and subsequent removal with HF acid create micromechanical irregularities that enhance the zirconia to resin bonds.[65-67] However, as creator of the SIE method stated "... SIE requires an investment of time and effort in order to achieve the required surface properties, and remains sensitive to the handling procedure during every step of the technique."[67]

Other methods such as ceramic coating,[26] fusion sputtering,[68] nanostructured alumina coating,[39] and titanium dioxide tube incorporation[69] were used, but more research is needed for them to be of any practical use.

Chemical

At present, following compounds are known to chemically bond to zirconia:

a. MDP containing zirconia primer
b. Primers composed of other monomers
c. A universal adhesive.

MDP monomer can make a chemical bond with metal oxides, such as zirconium oxide.[70,71] Researchers have found that adhesion between 10-MDP and zirconia was not only ionic bonding but also hydrogen bonding.[72]

The adhesives that contain chemical promoters are known as "Universal adhesives." Most of these universal adhesives contain 10-MDP at different concentrations and on application to zirconia after sandblasting increased adhesion and have even been proposed to replace mechanical conditioning and the need for primer application.[10,73] However, hydrolytic degradation of 10-MDP causes a decrease in adhesion over time in all its application forms, compromising the adhesive protocol.[36,74-76]

Application of luting and priming agents containing the adhesive monomer MDP provides better bond strength to zirconia than do other systems. However, some studies concluded that MDP in ceramic primer is effective for bonding zirconia and a luting agent does not necessarily have to contain an adhesive functional monomer when appropriate priming agent that contain such monomer is used. However, the strength and durability were not sufficient to satisfy the clinical requirements of retention, if the restorations were retained only by chemical bonding systems. Additional mechanical retention was still necessary.[77] Hence, combination of mechanical and chemical pretreatment appeared particularly crucial to obtain durable bonding to zirconia.[78] A recent study has also opined that combination of micromechanical and chemical surface treatment is a prerequisite for increasing adhesion to zirconia.[79]

Second to micromechanical roughness, adhesion strength was significantly increased by the adhesive system used. It has been proposed that the use of MDP-containing primers with resin composite cement containing the MDP monomer is required to enhance the bonding efficiency.[44] Hence, primers that contain MDP monomer should be used with resin cement even if it contains the same.[78,80,81]

Mechanochemical

Researchers have found that combined mechanical (TSC) and chemical (silane/MDP-containing ceramic primers) surface pretreatment of zirconia improved the bond durability of composite cement bonding to zirconia.[82] When zirconia was air abraded with Al_2O_3 (110 μm), it resulted in higher roughness values, but air abrasion protocols with SiO_2 (110 μm) promoted better adhesion to MDP-based resin cement.[55] Regarding the type of particle, studies found similar bond strength values between Y-TZP specimens subjected to airborne abrasion with conventional alumina particles and silica-coated alumina particles.[83,84] However, after 6 months of aging, silica-coated zirconia surfaces presented a higher bond strength,[84] which may be because conventional alumina particles are sharp and hard, whereas silica-coated alumina particles are softer and smoother, being less aggressive on the ceramic surface and facilitating the chemical bond. One study also reported that silica coating, irrespective of the use of primer or universal adhesive, provided significantly higher microshear bond strength values than other methods (sandblasting, laser).[85]

Although sandblasting can modify the surface of the zirconia, when used alone, it has been found to be ineffective in increasing adhesion to zirconia, and a chemical surface conditioner is required to make it stable in the long term.[22,26,39,44] These chemical conditioners contain various molecules found in primers, adhesives, or cement. Surface conditioning methods, particularly physicochemical conditioning methods, tend to increase the bond strength values for resin-based cements to zirconia.[86]

The use of APA with 50 μm Al_2O_3 before sintering and the application of primer-containing MDP seem to be valuable methods for durable bonding with zirconia. APA with 50 μm Al_2O_3 after sintering induced the highest (t-m) phase transformation.[79]

One recent study concluded that the best treatment to promote greater bond strength to zirconia is to associate tribochemical treatment with self-adhesive resin cement containing a functional phosphate monomer.[87]

Resin cement

Evaluation of shear bond strength of different cements used with zirconia indicated that zinc phosphate and conventional and modified glass ionomer cements are not able to form a lasting bond with zirconia; only resin cement and resin cement-containing MDP monomer show good results even after aging.[88] It was also seen that bond strength of glass ionomer cements and conventional Bis-GMA-based composites is significantly lower, especially after aging by thermocycling. Only resin cement and resin cement-containing MDP monomer withstand thermocycling, with the latter achieving a higher bond strength.[70] Similar results were also obtained on evaluating the shear bond strength of five cements, before and after long-term stocking (2 years) and thermocycling at 37500 cycles. The results revealed that Bis-GMA-based cements lack long-term stability. The efficiency of different surface treatment, i.e., sandblasting with aluminum oxide (Al_2O_3) at 50 μm and silanization was also studied and found that surface treatments improve the initial bond strength, but their effect decreases with time. Only resin cements with phosphatic monomer have shown high adhesion values and reliability after thermocycling in association with sandblasting.[89]

The cements used in various studies for luting zirconia can mainly be divided into three types - Self-adhesive cements, cements containing 10-MDP, and Bis-GMA cements (which are neither self-adhesive nor contain 10-MDP). Bis-GMA cements showed lower adhesion values than the other two groups but better results in hydrolytic degradation.[22,90] A lot of studies have reported the synergistic effect on applying a 10-MDP primer, especially with self-adhesive resin cement.[13,91,92] Non-MDP-containing self-adhesive resin cements showed increased bond value with MDP-containing primer to zirconia ceramics. However, as per some studies, the bond strength of MDP-containing self-adhesive resin cements was not affected significantly by the use of zirconia primer due to the saturation of this molecule.[92] Hence, more studies are required to find the ideal resin cement although there is consensus on the need for prior mechanical surface conditioning to increase their adhesive values.[32,39,60,90] More studies regarding cement degradation following artificial aging are also required.[25,90]

Silanization and acid etching are not effective on zirconia because it is inert and without glassy matrix on which those substances act. For cementing zirconia restorations, the best procedure seems to be the combination of sandblasting with aluminum oxide (Al_2O_3) at 50 μm and resin cements-containing esteric organophosphate monomer (MDP).[62] However, during air abrasion with Al_2O_3 particles, large particles (>110 μm) and under high pressure (>3 bar) should be avoided, and an effective chemical component should be used[93] as air abrasion leads tetragonal to monoclinic (t-m) phase change on the surface of zirconia that in the long term can be detrimental to the restoration, not only because of the defects it creates[40] but also because of the low-temperature degradation suffered by zirconia.[94]

Artificial aging

This review is based on *in vitro* studies and so clinical guidelines cannot be established. Saliva contamination or parafunctional habits that negatively affect adhesion have not been accounted for.[95] Moreover due to variability in study designs contradictory results have been found. Hence, more dedicated studies are required to standardize specific techniques and to simulate clinical conditions for predictable results in zirconia bonding.

Majority of the articles selected for this review used liquid storage and thermocycling for artificial aging. These two methods in combination allow the evaluation of hydrolytic degradation and *in vitro* hydrothermal aging.[86,95]

Various liquids were used for storage from distilled water to artificial saliva. Storage in a liquid medium significantly reduced adhesion. However, among the studies with thermocycled groups, great variation was seen in the number of cycles, thus making it impossible to compare the results.

However, certain recommendations must be considered for any studies and reviews:[1] Studies should include a control group with no treatment, to more effectively assess the pretreatment tested.[2] It is necessary to standardize the artificial aging method used to compare the results in a more effective way.[27]

Tests

Due to the lack of an international standard, different types of tests have been used to assess the bond strength between zirconia and resin cement. Due to its ease of use, macroshear test was most commonly performed. Otani *et al*.[96] described the macro tests (macroshear and macrotensile) as those that presented more heterogeneity in the distribution of stress and loads due to the greater adhesion area tested. On the other hand, the micro tests (microshear and microtensile) showed less variation and higher adhesive values due to a smaller adhesion area and less possibility of finding defects in the cementing. Tensile bond strength was found to be more sensitive in detecting differences in bonding effectiveness of different surface treatments after aging.[52] Many proposed[97] that failure analysis based on fractographic principles should assist researchers to correctly interpret the fracture phenomena.

CONCLUSION

The clinical success of a zirconia restoration is strongly dependent on the quality and durability of the bond between restoration and resin cement. A durable and strong bond requires zirconia surface changes for mechanical retention and chemical adhesion. New methods to increase bond strength between resin cement and zirconia need further investigations. This paper reviews various methods which have been used to enhance zirconia–resin cement bond strength, published in last 21 years. After reviewing the literature, we found:

a. There has to be a standard protocol for aging and thermocycling to standardize the examination
b. In spite of some studies being contradictory, Al_2O_3 sandblasting remains the best surface treatment method to date
c. Mechanochemical surface pretreatment provides the best adhesion
d. The best procedure for zirconia cementing is combination of sandblasting with 50 μ Al_2O_3 particle and then applying self-adhesive resin cement containing 10-MDP
e. SIE and application of low fusing glassy porcelain methods are promising, but more studies and simplification are needed.

For bond strength evaluation and stability and to establish standardized clinical protocols, more studies are required.

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Conflicts of interest

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