

# Zirconia

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Zirconia is one of the indirect metal-free restorations used in dental medicine.

resin bonding

dental bonding

zirconia

3Y-TZP ceramic

cement

## 1. Introduction

In recent decades, the increasing aesthetic needs in dentistry have led to the progressive overcoming of metal-ceramic prostheses and led to a focus on indirect metal-free restorations. Yttrium-stabilized zirconia has occupied an increasingly important role and offers a wide variety of clinical applications, such as root posts, implant abutments or as a material of choice for indirect ceramic restorations. It has the most favorable mechanical properties compared to other high-strength ceramics with flexural strengths of 700–1200 MPa, fracture resistance of more than 2000 N and fracture toughness of 7–10 MPa <sup>[1][2][3][4]</sup>. However, not only strength is important but also cementation and the adhesion of cement both to the dental tissues and to the restorative material is critical for the long-term success of the restorations <sup>[5]</sup>.

Surface treatment with hydrofluoric acid (HF) and silane coupling agent application of the silica-based ceramics is a well-established method to achieve durable adhesion to resin-based materials <sup>[6]</sup>. However, this process has failed for adequate resin bond to zirconia ceramics because they do not contain a silica phase making adhesion impossible <sup>[7][8][9]</sup>. Therefore, in the last few years, several zirconia surface pretreatments have been suggested to enhance the bond strength of luting cement to zirconia ceramics. Some of these methods facilitate an increase of surface roughness, improving micro-mechanical retention of the resin cement employing airborne particle abrasion with alumina particles <sup>[10][11]</sup>, tribochemical silica coating (TSC) <sup>[1][12][13]</sup>, laser irradiation or chemical etching <sup>[14][15][16][17]</sup>. However, it has been reported that possible damage on the zirconia surface is created by air-abrasion methods <sup>[18][19][20][21]</sup>. To solve this problem, alternative methods have been introduced, such as chemical promoters and resin cement based on organophosphate/carboxylic acid monomers specific for zirconia <sup>[22]</sup> that have been considered as chemical surface treatments. Among them are functional monomers as 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP), phosphonic acid acrylate or anhydrides <sup>[23][24][25]</sup>. Furthermore, silane deposition <sup>[26]</sup>, selective infiltration etching (SIE) <sup>[27]</sup>, ceramic coating and the use of cement-containing MDP are proposed chemical methods <sup>[28]</sup>. However, hydrolytic degradation is still problematic <sup>[29]</sup>.

Several methods have been used to evaluate the bond strength of resin-based materials to dental ceramics, including macroshear, microshear, macrotensile and microtensile tests. Furthermore, methods to evaluate bond durability simulating the oral conditions include short- and long-term water storage and thermocycling at diverse

temperatures, dwell time, and number of cycles. Therefore, it is difficult to compare different studies on the same materials even when the same test method was employed [5][30].

Due to the great increase in in vitro studies in recent years and the lack of consensus on resin-bonding protocols for zirconia restorations, it is necessary to evaluate the current data to unify criteria and provide clinicians with relevant information for their daily activity.

## 2. Adhesion to Zirconia: Surface Pretreatments and Resin Cements

### 2.1. Zirconia Surface Pretreatments

Pretreatment techniques were classified into three groups: (1) mechanical: studies that used air-abrasion protocols, laser, ceramic coating, or chemical etching, (2) chemical: studies that employed coupling agents such as adhesive resins, silanes or primers, (3) mechanicochemical: when both mechanical and chemical conditioning methods were applied. Control groups were defined as zirconia substrates with no surface pretreatment.

The studies agree that the zirconia surface needs to be prepared before applying the resin cement since all the pretreatments increased the bond strength, improving the values of the control group [31][32][33]. The first requirement for adhesion is to achieve a surface free of contaminants. Most of the studies started the surface conditioning protocol by polishing with papers, sprays or milling cutters of silicon carbide ranging between 220 to 4000 grit. Although several studies did not mention this step, they may have done it too. Ultrasonic cleaning before surface conditioning or the resin cement is also widely used [34][33][35][36][37][38][39][40][41][42][43][44][45][46][47]. Likewise, several solutions were used, including distilled water, alcohol, acetone, ethanol, and isopropanol, with a usage time between 1 and 10 min. In almost no studies, the effect of cleaning methods on adhesion to zirconia has been considered, but all authors considered it as a beneficial element [35][40][42][46].

Several mechanical pretreatments have been investigated. Sandblasting with alumina particles improved the bond strength values due to the increase in surface energy, wettability, roughness, and the appearance of hydroxyl groups that will facilitate bonding with the primer/universal adhesive/cement [31][36][39][41][46]. The particle size used ranged from 30 to 110  $\mu\text{m}$ , at 0.5–4 bar for 10–20 mm [30][48][31][32][33][36][37][38][39][41][42][43][44][46][47][49][50][51][52][53][54][55][56][57][58][59][60][61][62][63][64][65][66][67][68][69][70][71]. An increase in particle size and pressure had long been associated with the formation of microcracks and weakening the mechanical properties of the material [32][37][39][47][57][60][61][64][66][67][69]. However, the bond strength was not affected by the variation in particle size and pressure [46]. 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 [46][66].

The application of lasers to the surface of zirconia is based on the same principle as sandblasting, obtaining a rough surface and an increase in wettability that allows micromechanical retention with the resin [42]. Different types of lasers have been described (Er: YAG, Nd: YAG, Yb: YAG,  $\text{CO}_2$ ), 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 [33][38][41][50] due to the appearance of microcracks on the surface of the zirconia, leading to a phase transformation and weakening the mechanical properties [50]. Therefore, the laser is not currently considered a valid mechanical pretreatment [33][41].

An electrical discharge machine (EDM) described by Rubeling et al. [72] was used in one study, obtaining better adhesion values than sandblasting and TSC, but the presence of microcracks was also seen on the surface of zirconia [37].

## 2.2. Resin Cements

The classification of resin cement was complicated because of the great variation in their chemical compositions: phosphoric acid esters, 10-MDP, HEMA, glycerolphosphate dimethacrylate (GPDM), 4-META, bis-GMA or triethylene glycol dimethacrylate (TEGDMA). In addition, the exact composition or percentage of each component is hardly shown due to the lack of information from manufacturers. Therefore, their classification was structured in self-adhesive, cement with 10-MDP, and Bis-GMA cement (without 10-MDP or were not self-adhesive). In general, within the same group, the cement had great variability due to both the percentage of the different components and the viscosity of the cement, which can interfere with micromechanical interpenetration [59]. There is no consensus on which cement is above another, except for Bis-GMA, which showed lower adhesion values than the other two groups. However, this molecule better withstands hydrolytic degradation [31][59]. The relationship to the addition of a primer containing 10-MDP is unclear. Different studies have reported an increase in adhesion when previously applying a 10-MDP primer, especially with self-adhesive cement [39][51][52]. Conversely, another study reported the opposite in cement with 10-MDP due to the saturation of this molecule [52]. Nevertheless, there is consensus on the need for previous mechanical surface conditioning to increase their adhesive values [50][57][59][69]. Regarding the degradation of cement after artificial aging, no consensus exists. Thus, more studies are needed to demonstrate the ideal resin cement [30][59].

## 2.3. Test

Different types of tests have been used to assess the bond strength between zirconia and composite cement that can be explained by the lack of an international standard. The most used was the macroshear test, probably due to its simplicity of use. Otani et al. [73] 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 surface. 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. However, the number of premature failures in the specimen preparation step was higher. Nevertheless, the variability of the tests and their influence on the results make it very difficult to compare the results among the studies.

## 2.4. Artificial Aging

The most used method for artificial aging was liquid storage and thermocycling. Liquid storage allows the evaluation of hydrolytic degradation, and thermocycling reproduces in vitro hydrothermal aging [5][74]. The most frequently used liquid was distilled/deionized water, but other types of solutions were used, such as esterase, acetic acid, alcohol, phosphoric acid or artificial saliva, to reproduce different clinical scenarios [32][44][61]. Studies concluded that storage in a liquid medium significantly reduced adhesion compared to control groups. Acetic acid, phosphoric acid and esterase were the solutions that caused a greater effect [34][32][40][44][63][64]. The number of cycles showed a great variation among the studies with thermocycled groups, which makes it impossible to compare the results. In this systematic review, the ISO 10477 standard was followed concerning metal–resin bond, which established the minimum number of cycles at 5000 [75]. Thermocycling decreased adhesion values due to hydrothermal aging [45][50][51][67]. However, it has been reported that the number of cycles above 5000 does not decrease the values significantly [32][45][76]. Other studies used a combination of storage in liquid medium and thermocycling, which caused a significant decrease in the adhesive values [57][63][66]. This combination may be the one that causes greater degradation at the interface but requires much more time to complete [57][63][66].

### 3. Conclusions

The following conclusions were drawn:

- There are a great variety of zirconia surface pretreatments, cement, artificial aging method and tests used in the studies that make it difficult to compare the results.
- Zirconia surface cleaning must be performed before pretreatment methods to adhesion.
- Mechanicochemical surface pretreatments offered the best adhesive results. Tribochemical silica coating at a pressure of 1.8–2.8 bar has proved to achieve a significant increase in adhesion to zirconia.
- New methods as feldspathic ceramic sandblasting and silane application or YAG laser combined with silane seem to be promising alternatives in adhesion to zirconia.
- There is great variability in the percentage of components and the viscosity of the resin cement. Self-adhesive cement and those containing 10-MDP obtained the best results in adhering to zirconia, without clarification of which is the best.
- The use of a 10-MDP primer is still controversial.
- Standardization of test to evaluate the bond strength between zirconia and resin cement is needed
- Artificial aging decreased adhesion; therefore, storage in water for 30 days or thermocycling for 5000 cycles must be performed in laboratory studies.
- A clinical protocol for adhesive cementation to zirconia has not yet been performed.

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