Surface Treatment Prior to Cementation of Y-TZP Based Ceramics

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Introduction

A progressive improvement in the mechanical properties of dental ceramics has led to an increase in all-ceramics restorations. The introduction of yttrium-stabilized tetragonal zirconia (Y-TZP) as a ceramic core material extended the design, and application limits of all-ceramic restorations [1-4]. The superior mechanical properties of this polycrystalline material, like high strength, fracture toughness, hardness, wear resistance, it combination with CAD/CAM technology have allow for fabrication of long-span and complex restorations with high accuracy and success rates [1,3,5,6].

In contrast to conventional dental ceramics (veneer ceramic), Y-TZP does not present the glassy phase in its crystallite structure [3,7]. In this way, the absence of a glassy phase impairs the effectiveness of conventional adhesive luting procedures, which include etching ceramic surfaces with hydrofluoric acid and applying silanes coats prior to the use of a resin cement [2,3,5,7-9].

Strong resin bonding relies on micromechanical interlocking and adhesive chemical bonding to the ceramic surface, requiring surface roughening for mechanical bonding and surface activation for chemical adhesion [10]. In some instances, high strength ceramic restorations do not require adhesive bonding to tooth structure and can be placed using conventional cements, which rely only on micromechanical retention. However, resin bonding is desirable in several clinical situations-e.g., when the prepared tooth structure is short or tapered. In addition, it is likely that strong chemical adhesion would lead to enhanced long-term fracture and fatigue resistance in the oral environment. Non-destructive methods for treating inert ceramics to produce an activated/functionalyzed surface are desirable in such cases.

The longevity of an indirect restoration is closely related to the retention quality. High retention is important to maintaining integrity of the cement at the margin (prevention of micro leakage) [11], and increasing the fracture/fatigue resistance [11,12]. A 3 years follow-up of zirconia single crowns related that 7% of all Y-TZP crowns placed in the posterior region de bonded [13]. This draw back may suggest that promoting a durable bond between the Y-TZP and tooth structure is still challenging the adhesive dentistry.

Although the loss of retention has never been a topic of interest with Y-TZP restorations (because it has never been reported), efforts have been undertaken to establish ways to achieve a reliable bond between Y-TZP crowns and luting agents [14-19]. Comparatively, data from a literature review [20] showed a 2.8% loss of retention rate in all-ceramic systems (except for zirconia) after five years, while for a metal-ceramic system the loss of retention rate decreases to 0.7% after 10 years [21]. Thus, the effective bond strength between the substrate and ceramic plays an important role in enhancing the longevity of restorative treatments [17,18,22] and in preventing micro leakage [19].

A great diversity of materials for luting ceramics restorations is commercially available. These include zinc phosphate cements, conventional and resin-modified glass ionomer cements, resin cements and self-adhesive resin cements [23]. Conventional cementation techniques do not provide sufficient bond strength for some clinical applications [10]. Resin cements possess some advantages compared with the other classes of materials, since they have lower solubility, higher resistance and better esthetic characteristics [23,24]. In addition, the adhesive interface between the resin cement and ceramic might increase the restoration’s resistance during occlusal loads [10,23].

The shear bond strength of 11 different types of cements to a Y-TZP ceramic was evaluated [23]. The results indicated that zinc phosphate and conventional and resin-modified glass ionomer cements were not able to form a durable bond to Y-TZP [23]. In another study, the authors stated that the bond strength of glass-ionomer cements and that of a conventional Bis-GMA-based resin-composite to Y-TZP ceramics is significantly lower, especially after thermal aging [24].

Due to the difficulties in choosing an adequate surface treatment and cementation for zirconia ceramic systems, the aim of this literature review was to investigate the treatment modalities for reinforced ceramic surfaces bonded to resin cements.

Mechanical Bonding - Roughening

Bonding to traditional silica-based ceramics, generally employing both mechanical and adhesive retention, has been well reported and bond strengths are predictable [10]. A strong resin bond relies on micromechanical interlocking created by surface roughening and chemical adhesion between the cement and ceramic [10]. Current
Chemical / Mechanical Bonding

Other types of micromechanical adhesion is the association of silica particles in sandblasting, which promote a silica coating on acid-resistant ceramic structure by interlocking silica particles in the ceramic surfaces. Thus, the silane is able to act on the treated surface and promote adhesion chemistry enhancing micromechanical bond [12].

Silanes are compounds that contain silicon (Si) atom or atoms, are similar to orthoesters in structure, and display dual reactivity. Their use in clinical dentistry and effect on adhesive bonding has been described in detail in the scientific literature [15,25,35-37]. One end of a silane molecule is organically functional (e.g., vinyl–CH₂, amino–NH₂), and can polymerize with an organic matrix (e.g., a methacrylate). The other end is generally comprised of alkoxide groups (e.g., methoxy–OCH₃, ethoxy–OCH₂CH₃), which can react with a hydroxylated 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, and with certain ceramic or ceramic-containing composite posts for endodontic applications employing resin-based filling materials. Silanes are also believed to promote surface wetting, which enhances potential micromechanical retention with low viscosity resin cements [37,38]. Traditional silane chemistry is not truly effective with Y-TZP, as it possesses a relatively non-polar surface, is more chemically stable than silica-containing ceramics, and not easily hydrolyzed.

Aboushelib et al. [39] showed that application of silane alone on Y-TZP resulted in low bond strength. The use of five silanes (MPS (3-methacryloyloxypropyl-trimethoxysilane)ACPS (3-acryloyloxypropyl-trimethoxysilane) and ICS (3-isocyanatopropyl-triethoxysilane) along with styrylthyltrimethoxysilane and 3-(N-allylamino propyltrimethoxysilane) to aid in luting of as-received Y-TZP resulted in bond strengths that were significantly less than when using the silanes on SIE (selective infiltration etching) Y-TZP. It was shown that MPS produced greater bond strength when used on SIE Y-TZP compared to the other silanes. However, bond strength of SIE Y-TZP using the silanes decreased significantly after long-term storage [15]. This decrease in bond strength demonstrates that use of silanes does not aid in producing a hydrolytically stable bond with Y-TZP. Although bond strength decreased after time, SIE does create a retentive surface for mechanical bonding. It is also possible that SIE could chemically modify the surface to improve bonding between the silane and Y-TZP.

Due to the absence of silica in Y-TZP, 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 [10,23,26,27,40-42] with the Co Jet and Rocatec systems (3M ESPE, Seefeld, Germany) being the most heavily favored commercial products utilized for applying the coating. The tribochemical technique air-abrades the ceramic surface with alumina particles that have been coated with silica, embedding/coating the surface with silica [43,44]. It results not only in preparing the surface for silanization, but also to creates the micromechanical retention [10]. Research has shown that the application of a tribochemical
coating before silanization significantly enhances bond strength between a treated substrate and resin cement [10,23,26,27,40-42]. The bound silica particles serve as reactive sites for conventional organo-silane monomer primers, however residual bond strengths tend to be lower than those obtained on conventional dental porcelain surfaces [10]. Furthermore, there can be significant loss in bond strength over the long-term when using traditional resin cements used for silica-based ceramics. This might be a result of a low concentration of silica on the surface due to difficulty in particle abrasion caused by the high hardness of Y-TZP [12].

The cleaning of a tribochemical coated Y-TZP before resin bonding can be deleterious to bond strength. Ultrasonic cleaning of tribochemical coated Y-TZP for 2 or 5 min before silanization and resin bonding significantly decreases bond strength to statistically similar values of air-abrading alone before bonding [45]. It was shown because ultrasonic cleaning results in decreased silica content on the surface. This decrease, along surface morphology changes, is thought to cause the loss of bond strength. Air pressure has also been shown to affect bond strength when applying a tribochemical coating. Heikkinen et al. [46] determined that increasing the air pressure at which tribochemical coating is applied significantly increases the bond strength of resin cements to zirconia ceramics. The application of the MDP monomer with a silane agent to YPSZ ceramic coated with silica in the tribochemical system showed the highest results.

<table>
<thead>
<tr>
<th>Study</th>
<th>Ceramic</th>
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<th>Luting Agents</th>
<th>Test</th>
<th>Results</th>
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<td>Delta Bona A et al. (2007) [16]</td>
<td>ZrO2-In-Ceram Zirconia</td>
<td>Silica coating (Cojet, 3M-ESPE) and 9.5% hydrofluoric acid application.</td>
<td>Z100, 3M-ESPE, St. Paul, MN, USA</td>
<td>Traction and shear bond strength</td>
<td>The silica coating system (Cojet, 3M-ESPE) demonstrated a significant increase in the mean bond strength in all the tests.</td>
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<td>Derand T et al. (2008) [20]</td>
<td>Procera (alumina and zirconia)</td>
<td>Aluminium oxide blasting with 50 and 100μm particles.</td>
<td>Panavia 21 and Variolink II</td>
<td>Microtraction</td>
<td>Resin cement has a higher retention value when cementing zirconia, when compared with alumina.</td>
</tr>
<tr>
<td>Amaral R et al. (2006) [22]</td>
<td>In-ceram alumina, In-ceram zirconia and Procera</td>
<td>Aluminium oxide blasting and silica coating using Cojet System.</td>
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<td>Kitayama S et al. (2009) [21]</td>
<td>Zirconia (Cercon Base)</td>
<td>Al2O3 blasting with 70 μm particles, inner surface inc ceramic - INT</td>
<td>Panavia F and Superbond C&amp;B</td>
<td>Traction</td>
<td>The zirconia surface treatment with INT coating followed by silanation successfully increased the bond strength between resin cements and zirconia ceramics.</td>
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<td>Tanaka R et al. (2008) [22]</td>
<td>Zirconia YPSZ</td>
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<td>The application of the MDP monomer with a silane agent to YPSZ ceramic coated with silica in the tribochemical system showed the highest results.</td>
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Table 2: Results from studies performed with Primers.

<table>
<thead>
<tr>
<th>Study</th>
<th>Ceramic</th>
<th>Type of primer tested</th>
<th>Bonding System</th>
<th>Test</th>
<th>Results</th>
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<tr>
<td>Dias de Souza et al. (2011) [22]</td>
<td>Zirconia - LAVA</td>
<td>Alloy Primer, Epiguard Primer</td>
<td>RelyX Unicem and Panavia F</td>
<td>Traction</td>
<td>They showed that application of a primer containing MDP can increase the bond strength between the cementation system and the flat and smooth zirconia substrate.</td>
</tr>
<tr>
<td>Tsuo Y et al. (2006) [22]</td>
<td>Zirconia Y-TZP</td>
<td>Alloy Primer, Super Bond Monomer Liquid, Metal Primer II</td>
<td>Linkmax HV (GC Corp., Tokyo, Japan)</td>
<td>Shear bond strength</td>
<td>The results from this study suggest that surface treatment with a commercial adhesive primer following blasting with 50-μm particles of aluminium oxide was effective for a strong and lasting bond between the resin cement and the zirconia ceramic.</td>
</tr>
<tr>
<td>Yun JY et al. (2010) [25]</td>
<td>YTtria Tetragonal Zirconia Polycrystal (Y-TZP).</td>
<td>Alloy Primer, V primer, metalite</td>
<td>Panavia F 2.0, Superbond C&amp;B or M bond</td>
<td>Shear bond strength</td>
<td>The combination of blasting with 50-μm Al2O3 particles and primer for metal is reliable for a strong a lasting bond between the zirconia ceramic and the cements, especially Panavia F 2.0.</td>
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</table>

Table 3: Results from studies on Er/Nd: YAG.
on a Y-TZP surface and fired in a furnace using butane gas burned with atmospheric oxygen [48] or using plasma spray technique to deposit a siloxane coating on Y-TZP [8]. The fused glass film increased surface roughness of Y-TZP, allowing increased micro-retention. The silica-rich film also allows for silanization of Y-TZP before bonding, making it possible to form siloxane bonds to resin cement. Derand et al. [8] showed that use of this fused micro-pore film significantly increased the bond strength of Y-TZP (11.3–18.4 MPa) compared to untreated or silanized Y-TZP (0.5–1.5 MPa). However, it was expensive and too complex to be commercially viable for standard dental applications.

A novel surface roughening technique that has been explored for Y-TZP is selective infiltration etching (SIE) [14]. SIE uses a heat-induced maturation process to pre-stress surface grain boundaries in Y-TZP to allow infiltration of boundaries with molten glass. The glass is then etched out using hydrofluoric acid, creating a 3D network of inter-granular porosity that allows nano-mechanical interlocking of resin cement. The advantage of SIE is that it only involves grains that are exposed to molten glass, allowing control of the area to be etched. Aboushelig et al. [14] showed that using SIE on Y-TZP resulted in increased micro tensile bond strength (49.8 ± 2.7 MPa) when compared to particle air-abraded Y-TZP (33.4 ± 2.1 MPa). The use of SIE improved nano-mechanical retention of zirconia by increasing the surface area available for bonding. This was confirmed by AFM work done by Cascucci et al. [34] showing that the surface roughness of Y-TZP is significantly greater after SIE, when compared to particle air-abrasion or hydrofluoric etching.

Nevertheless, efforts should be directed to achieving a chemical protocol that does not require any special equipment or produce mechanical damage in the Y-TZP surface. As such, the use of MDP-based primers and alkaline solution seem to contribute to this approach [22].

**Chemical Treatment**

Y-TZP ceramics are not easily etched or chemically functionalized using conventional treatments, and require very aggressive mechanical abrasion methods to increase surface roughness, possibly creating strength-reducing surface flaws as a result [4, 10, 30, 31]. Therefore, in order to achieve acceptable cementation with less surfaces damage, chemical treatments for better attachment methods are required for Y-TZP ceramics.

The use of phosphoric acid primers or phosphate-modified resin cements has been shown to produce silane-like adhesion, through similar types of hydrolyzation-driven chemistry. However, bond strength values reported in the literature through use of these agents are generally lower that the values reported for tribochemical silica coating, coupling with silane and resin cement [38]. More recently, Aboushelig et al. [16] reported on increased bond strength utilizing selective infiltration etching and novel silane-based zirconia primers. The available approaches for adhesive bonding of zirconia ceramics are not adequate for all clinical applications and their long-term efficacy is currently unknown.

The chemical adhesion potential of zirconia is low as a result of its inertness (i.e., it presents a non polar surface) [49] which hampers its union with cements [50]. However, it has been discussed [51] that an increased availability of hydroxylic groups was found at the implant surface of a zirconia/alumina nano-composite after 15 M sodium hydroxide solution (NaOH) treatment. In addition, durable bond strength may be achieved by employing acid monomers, such as 10-methacryloxydecyl dihydrogen phosphate (MDP)-based primers [19].

As previously mentioned, the non-silica composition of Y-TZP makes it difficult to bond Y-TZP to tooth structures using traditional resin composite cements. The application of an MDP-containing bonding/silane coupling agent is the key factor for a reliable resin bond to Y-TZP ceramics and is not influenced by the resin luting agent used [22,25]. Currently, in the dental market, priming agents that contain special adhesive monomers are available to improve adhesive bonding to metal alloys.

Kern and Wegner [26] were the first to report the long-term bond strength of phosphate monomer-containing resin-based composite cements to Y-TZP. The use of phosphate monomer primers (MDP) into Y-TZP surface has been proposed to increase the bond strengths between cements and zirconia [28]; however, the composition of these primers can result in different bond strength values because they contain different monomers (eg, MDP, 4-methacryloxy-ethyl trimellitate ashy-dride, thiophosphate methacryloylalkyl derivates, and zirconate coupler) [52]. The phosphate ester group presents in MDP might chemically bond to metal oxides, such as zirconium dioxide [24,38,53]. Therefore, as a result of its chemical composition, the Alloy Primer (Kuraray Dental, Japan) can result in an improved performance when used in metal alloys. This primer presents two active monomers, MDP and 6-4-vinylbenzyl-n-propyl amino-1,3,5-triazine-2,4-dithione (VBATDT), that set up bonds to precious and non precious metal oxides, respectively. MDP has been considered an important monomer in bonding to Y-TZP [53]. The MDP-based material presented higher bond strength to zirconia surfaces air abraded with alumina particles and this bond could survive 150 days of water storage [53].

Other studies also stated that resin cements with phosphate ester groups increase the bond strength of air abraded and tri biochemically-coated surfaces [22,24 26 38].

The association of MDP-based metal primer with a zirconate agent (2,2-di [allyloxymethyl] butyl trimethacrylol zirconate) strengthened the bond between resin cement and zirconia ceramic [38]. High bond strength between Alloy Primer and Rely X U100 has been reported, even after aging [19].

MDP resins cements are hydrolytically stable, and therefore, do not decrease in bond strength over time. The addition of a MDP-containing bonding/silane coupling agent to enhance bonding of MDP resin cements has produced positive results. It was shown that particle air-abrasion or tribochemical coating, followed by the application of MDP-containing bonding/silane coupling agent, resulted in increased bond strength compared to MDP-containing cements only [25,27,33,54]. It is known that acidic monomers rapidly hydrolyze silane coupling agents, producing the siloxane bonds necessary for chemical bonding [55]. It is thought that the acidic nature of MDP enhances the polysiloxane bonding produced by silane coupling agents and results in improved retention of resin cements to Y-TZP [54].
Other phosphate monomer-containing cements like RelyX Unicem (3M ESPE, Seefeld, Germany), a universal self-adhesive resin cement, and non-phosphate monomer-containing cements like RelyX ARC and Bifix QM (VOCO GmbH, Cuxhaven, Germany), Bis-GMA resin cements, and Multilink Auto mix (Ivoclar Vivadent, Amherst, NY, USA), a phosphoric acid-based cement, have exhibited statistically comparable bond strength to MDP-containing resin cements in laboratory studies [23-25,33]. Although these resin cements have shown good mechanical retention, MDP-containing resin cement continues to be the popular choice for luting Y-TZP prosthetics in clinical applications due to their low incidence of failure and loss of retention [13,56,57].

Considering that strong chemical bonds with zirconia have been proven to be difficult as a result of zirconia nonreactive surface [10]. The addition of NaOH in the surface of Y-TZP prior the cementation may activate the zirconia surface due to the increased availability of hydroxyl groups (OH). This factor may have favored the acid-base reaction between the metal oxides present on the zirconia surface with both cement and primer agents, which are admittedly acidic. Moreover, the surface energy may have been raised (mainly the polar component), which could increase the wet ability of the zirconia surface, and, as a result, it may have favored the bond reacting between the metal oxides on the zirconia surface and the functional monomer present in the composition of both primer agents and the resin cement [22]. Lorenzoni et al. [22] found better shear bond strength result when the 0.5 M NaOH solution was associated with Alloy Primer (AP) (Kuraray Dental, Japan). However, this outcome may be more significantly related to the composition of the primer than to the benefits provided by the NaOH solution, since the AP and NaOH-AP results were statistically similar.

Minimal values of 10-13 MPa has been established for shear bond strength [58], only application of NaOH prior cementation do not reaches this cited values, but the association of NaOH and MDP primers can exhibit a ideal behavior [22].

Other monomers present in resin cements might also have a chemical affinity for metal oxides [23,24,59]. For example, the anhydride group present in 4-META monomer and the phosphoric methacrylate ester can also chemically bond to zirconia ceramics [24,59]. It was observed that the bond strength of a polymethylmethacrylate (PMMA) resin cement containing 4- META was initially high; however, this bond was not strong enough to resist thermal aging [24]. Water absorption by the PMMA during thermal cycling may have weakened the chemical bond [24]. On the other hand, the use of self-adhesive cement containing phosphoric methacrylate ester resulted in bond strengths to Y-TZP similar to that of MDP-based resin cements after 14 days of thermal cycling and water storage [23]. In another study, this cement provided similar coping retention compared to MDP-based resin cement and resin-modified glass ionomer cement [60]. The mean coping removal stresses for the axial surface ranged from 6.7 MPa to 8.5 MPa, which is similar to the range of removal stress observed for gold castings when using zinc phosphate and glass ionomer cements. The authors concluded that the three cements tested are capable of retaining zirconium oxide crowns successfully, requiring no additional internal surface treatment other than airborne-particle abrasion with 50-μm aluminum oxide followed by appropriate cleaning of the crown prior to cementation [60].

Further potential method for improving adhesion to Y-TZP is a unique vapor-phase deposition technique whereby chlorosilane is combined with water vapor to form a more reactive, SiOx-functionalized surface. The process utilizes a molecular vapor deposition (MVD) tool, developed specifically to deposit conformal, thin films to serve as hydrophobic, hydrophilic, biocompatible, protective, ordering, or otherwise reactive coatings [61]. This flexible system allows deposition of numerous materials from simple liquid precursors. Deposition conditions and precursor chemistry can also be modified to produce a range of surface characteristics. As the silicon tetrachloride (SiCl4) and water react with the substrate surface, active hydroxyl groups are formed on the surface subsequently forming a silicon oxide layer on the substrate surface. This treatment serves as a primer step for subsequent reactions with organo-silanes, used as adhesion promoters in conventional resin bonding applications. Piascik [10] showed that only a thinner SiOx seed layer treatment (2.3 nm) plus silane application had bond strengths better than tribiochemical silica coating treatment (Cojet Sand - 3M) and were the same as the bond iteration of Y-TZP/porcelain specimens. By using a chloro-silane-based vapor-phase pretreatment, a silica-like surface layer was created on Y-TZP and used to increase the binding sites for the subsequent organo-silane primer for conventional dental adhesive applications.

Discussion

Despite of several advantages provided by high crystalline ceramics, they carry a significant disadvantage with regards to cementation. On the other hand, glass ceramics show a well-established bonding protocol by means of fluoride acid conditioning combined to aluminium oxide blasting followed by the application of a silane agent [62, 63]. Despite the effectiveness of hydrofluoric acid on glass ceramics, the same cannot be applied to alumina or zirconia reinforced ceramic due to their low glass content, which is necessary to create surface porosity, thus making the latter an “acid-resistant”material [64].

There is no consensus amongst the reviewed works in this paper regarding to high crystalline ceramic cementation protocol. In this way, it is difficult to provide a well-defined protocol for surface treatment and cement/adhesive agents, which would provide high and durable bond strength between ceramic and substrate. Several treatments have shown reasonable results, yet not quite promising. Consequently, the choice of surface treatment for zirconia and alumina ceramic cementation is hard to be defined.

The literature analysis revealed that the procedures involved in ceramic surface preparation prior to cementation are essential for indirect restorations long-term success. It increases the surface energy and, consequently, the wet ability for the cementing agents [65]. The surface energy modification is the main objective to bond the high crystalline ceramics to substrate. It can be modified by means of several ways; however, there are literature suggestions that point out when it is modified by chemical agents and not by mechanical approaches (like sandblasting) advantages may be acquired due to it’s the non-invasive approach.
Amongst the papers reviewed, those on silica coating systems, Cojet and Rocatec reported high bond strengths [66-68]. A reasonable explanation to this outcome is related to the fact that when the surface of the ceramic is blasted with silica a chemical bond is created between the deposited silica and the cementing agent. Another factor would be the application of a silane agent, which is a bifunctional molecule, linking silicium dioxide to the hydroxyl groups from the surface of silica ceramic, and also shows a degradable functional group that co-polymerizes with the organic matrix from the resin cement. It is therefore applied to promote a chemical bond between the ceramic and the bonding system [69].

Sandblasting surfaces with aluminium oxide particles aims to change the surface topography of the ceramic, allowing a mechanical bond with the resin cement by means of mechanical interlocking. Besides to the abovementioned topography modification, the sandblasting method increases the surface energy and wet ability [66] allowing for an optimized surface to bonding to the cement agent. On the other hand, sandblasting may initiate micro cracks at surface cementation that may compromise the long-term stability and reliability of the ceramic [31, 70]. Finally, the size of the aluminium oxide particles, the distance and pressure for sandblasting application are not standardized, as per reported in the several studies reviewed [64,66,68]. There is no consensus on a clinical protocol for ceramic sandblasting, leading clinicians to considerable confusion.

**Conclusion**

The number of articles identified on this subject revealed contradictory results, considering the lack of clear guidelines for high crystalline ceramic surface treatment prior to cementation. However, the surface treatment prior to cement step is of extreme importance in order to achieve reliable bond strength between high crystalline ceramics and the resin cement. The surface treatments that are regarded as non-invasive may be regarded as the first choice for getting enough bond strength without decrease the outstanding mechanical properties of high crystalline contents ceramics.

**References**


