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Review Article

A Review of Surface Treatment Methods to Improve the Adhesive Cementation of Zirconia-Based Ceramics

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In spite of high mechanical strength, zirconia-based ceramics (ZrO_2) has poor bond strength after conventional bond cementation procedures, requiring different surface treatment methods (STMs). This review gathered information about the STM for adhesive cementation (AC) to ZrO_2 in the PubMed database, considering *in vitro* studies pertaining to AC for acid-resistant ceramics (ZrO_2) limited to peer-reviewed papers published in English between 1965 and 2013 in dental journals. Different STMs have been proposed for ZrO_2: air-abrasion (laboratory or chairside) with silica- (Si-) coated aluminum particles, the use of materials containing phosphate monomers, primer or silane application, laser irradiation, Si vapor phase deposition, and selective infiltration etching. In conclusion, STMs improve bond strength of resin luting cement to ZrO_2 mainly when tested in short time. STMs must be correlated to the type of ZrO_2 and the resin cement.

1. Introduction

Recent developments in ceramic materials science for dental applications have led to a class of high fracture strength materials represented by alumina (Al_2O_3) and zirconia-based ceramics (ZrO_2) that potentially enable long-term durability [1, 2]. These improved properties allowed the use of all-ceramic materials in situations of high mechanical stress, such as framework materials, crowns, bridges, core, and post systems [3].

The increase of mechanical properties by ZrO_2 addition is accompanied by a reduction in the glassy matrix and Si content [3, 4] resulting in acid-resistant ceramics [5]. In the Si-based ceramics the glassy matrix is selectively removed by hydrofluoric acid (HF) etching, increasing the surface roughness (Ra) for micromechanical bonding [6–8]. This procedure is generally followed by application of a silane coupling agent, that is able to bond with the silicone dioxide (SiO_2) and copolymerizes with the organic matrix of the resin cement [2]. The lack of a glassy matrix and the absence of SiO_2 make acid etching plus silane application incapable of modifying and treating the zirconia surface [9–12], with no apparent improvement in bond strength [7, 8, 13].

The clinical success of ceramic restorations depends on the cementation process [7]. Adhesive cementation (AC) to ZrO_2 ceramics is desirable [14] since it improves retention [2, 15], marginal adaptation, and fracture resistance [16], reduces the possibility of recurrent decay [8, 17], and enables more conservative cavity preparations [15]. Different methods to

Table 1: Criteria for paper selection for this study.

Topics	Criteria
Database	PubMed
Date range	1965 to 2013
Keywords	Ceramic surface treatment AND zirconia, "zirconia adhesion," zirconia ceramics, and "bond strength"
Language	English
Type of paper	*In vitro* laboratory research and literature reviews
Subset (journal group)	Dental journals

Table 2: Number of selected papers according to keywords.

Keywords	Total retrieved papers	Number of selected papers	Total*
Ceramic surface treatment and zirconia	120	30	79
"Zirconia adhesion"	79	12	
Zirconia ceramics and "bond strength"	118	37	

*Excluding repeated papers.

promote the adequate adhesion between the resin cement and ZrO_2 have been proposed: use of a phosphate-modified monomer (MDP) in resin cement [2, 18–23], laboratory or chairside air-abrasion with 110 and 30 μm Si-coated aluminum particles [22–26], the use of zirconate coupler primers [27], tetraethoxysilane flame-treat device usage [3], the use of organofunctional silanes [28, 29], laser irradiation [17, 30], the Si vapor phase deposition method [1], and the selective infiltration etching procedure [31–33].

Nevertheless, even with all these surface treatment methods (STMs) for increasing bond strength to ZrO_2 there are several controversial results, especially due to different bond strength testing methodologies [3]. The aim of this study was to discuss the STM for increasing adhesion capability of ZrO_2 by means of reviewing the literature, establishing a protocol for clinical procedures.

2. Methods

This review of the literature was based on a PubMed databases search following the criteria listed in Table 1. Papers were selected since 1965 due to the first cited method to strengthen dental ceramics by the addition of reinforcement oxides [34]. However, since it was not the aim of the present work to identify ZrO_2 ceramics introduction and development into dentistry, the search was limited to find different STMs and their results in relation to bond strength.

3. Results

The number of papers retrieved and selected from the PubMed search are described in Table 2. From all retrieved papers (Table 2) the ones that described techniques developed to increase bond strength of ZrO_2 to resin cement and their relation with the material's composition were selected. Bond strength methods of selected papers were shear tests (29 papers), *micro*shear tests (3 papers), tensile tests (5 papers), *micro*tensile tests (11 papers), and pull-out tests (01 paper). Just one paper employed both *micro*-tensile and shear bond strength methods, observing similar results [35].

Table 3 shows retrieved papers from PubMed database search grouped in accordance with bond strength (BS) tests, surface treatment method (STM), and results for BS improvement. Only zirconia-based ceramics BS measurements were listed in the table.

The following sections are based on the types of STMs for ZrO_2. Treatments were divided into chemical surface treatments, mechanical surface treatments, and alternative treatments.

4. Chemical Surface Treatments

4.1. Hydrofluoric Acid Etching. The most common STM for AC to ceramic restorations is based either on micromechanical bond obtained with HF etching, particles sandblasting or on chemical bond, obtained by the application of a silane coupling agent [15]. HF removes the glassy matrix of glass ceramics creating a high surface energy substrate with microporosities for the penetration and polymerization of resin composites, that is, enabling a micromechanical interlocking [7]. However, HF etching does not produce any change in arithmetic roughness (Ra) of ZrO_2 [36]. The negligible effect of the HF on the ZrO_2 surface occurs due to the absence of glassy matrix, resulting in low bond strength values [2, 35, 37, 38].

4.2. Functional Monomers. Special functional monomers have been used to improve the adhesion to ZrO_2. These materials present a chemical affinity for metal oxides and can be included both in resin cement and adhesives or applied directly over the ceramic surface [17]. Phosphate ester monomers, such as 10-methacryloyloxyidecyl-dihyidrogenphosphate (MDP), chemically react with ZrO_2, promoting a water-resistant bond to densely sintered zirconia ceramics [20]. MDP-based resin cement is advocated as mandatory for better adhesion to ZrO_2 [21, 22, 39], but some studies do not show bond advantages over conventional BIS-GMA based resin cement [17, 40]. A phosphonic acid monomer, 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA), showed some form of chemical bonding to zirconia surface [9], but there is no data available regarding the effect of 6-MHPA on the resin bond strength to zirconia ceramics after severe aging conditions [9]. Another monomer commonly used in ceramic primer materials, such as MEPS (thiophosphoric methacrylate), have been tested but with no clear advantages [17, 40]. Some nonphosphate metal primers were tested such as 6-[4-vinylbenzyl-n-propyl]amino-1,3,5-triazine-2,4-dithione (VBATDT), 6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate (MTU-6), 4-methacryloxyethyl trimellitic anhydride (4-META), and

Table 3: Retrieved papers from PubMed database search are, grouped in accordance with bond strength (BS) tests, type of ceramics, surface treatment method (STM), resin cements and results for BS improvement.

Reference number	BS method	Type of ceramics	STM	Resin cement	Results
[1]	MTBS	ZirCAD and ProCAD	50 μm Al_2O_3 AA + 2.6 nm Si_xO_y; 50 μm Al_2O_3 AA + 23 nm Si_xO_y; CoJet	C and B cement	Si_xO_y 2.6 nm > CoJet = Si_xO_y 23 nm > Ctrl
[3]	Shear	Empress II, In-Ceram, and ZrO_2	Flame treatment (2.5 s/cm^2), (5 s/cm^2), and (10 s/cm^2) + silane. Empress II- HF + silane	Variolink II	HF + silane yielded the best BS
[4, 5, 25]	MTBS	In-Ceram	110 μm Al_2O_3 AA, CoJet, and Rocatec; all silanated	Panavia F	CoJet = Rocatec > 110 μm Al_2O_3 AA
[8]	Shear	Y-TZP	HF; 50 μm Al_2O_3 AA; diamond abrasion	Enforce	AA > diamond abrasion > Ctrl > HF
[9]	TBS	GN-1 ceramic block	70 μm Al_2O_3 AA	Bistite II and Tokuso Ceramic Primer; Linkmax and GC Ceramic Primer; Rely-X ARC and Rely-X Ceramic Primer; Panavia F and Clearfil Ceramic Primer; Resicem and Shofu Porcelain Primer or AZ Primer	Primer enhanced BS except the AZ Primer
[10]	Microshear	Y-TZP	53 μm Al_2O_3 AA; YAG laser	Variolink II and Monobond-S; NAC-100 and SCP-100	Variolink II: control yielded the best BS NAC-100: AA = laser = ctrl
[11]	Shear	Y-TZP	Silica coating; 50 μm Al_2O_3 AA; MPS and/or 4-META silanes	Panavia F and SuperBonder	SuperBonder: MPS = 4-META Panavia F: MPS > 4-META
[12]	Shear	Y-TZP	TBC + primers	Rely-X ARC, epricord opaque primer; AZ Primer	All primers yelded BS
[13, 26]	Micro shear	Y-TZP In-Ceram	100 μm Al_2O_3 AA; HF glass pearls	Panavia F and Clearfil Porcelain Bond	In Ceram: HF = AA > ctrl Cercon: AA > Ctrl > HF
[17]	Shear	Y-TZP	53 μm Al_2O_3 AA; Er : YAG laser; primers	Calibra, Panavia F	AA + primers yelded high BS
[20]	TBS	Y-TZP	AA; silanated; silica coated; acrylized; MDP; polyacid-modified composite	Panavia	MDP > acrylized > polyacid-modified composite > silica coated > silanated = AA
[21]	Shear	Y-TZP	AA + MDP silane; AA + silane	Rely-X ARC, Panavia F	MDP silane assured higher BS for both RCs
[22]	MTBS	Y-TZP	125 μm Al_2O_3 AA, TBC	Calibra; Clearfil Esthetic; Rely-X Unicem	Clearfil Esthetic yelded the best BS
[23]	Shear	Y-TZP	125 μm Al_2O_3 AA, Clearfil silanes, MDP solution, and CoJet	Panavia F	CoJet + MDP + silane = CoJet + silane = CoJet > Ctrl = MDP + silane = silane
[27]	Shear	Y-TZP	MDP; zirconia primer	Clapearl DC	MDP = zirconia primer = MDP + zirconia primer
[29]	Shear	Y-TZP	Organo silane	Rely-X ARC, experimental resin	Only isocyanatopropyltri-ethoxysilane does not improve BS
[33, 74]	MTBS	Y-TZP	SIE and zirconia primer	Panavia F	SIE + primer > Ctrl. SIE + primer decreased BS with aging

Table 3: Continued.

Reference number	BS method	Type of ceramics	STM	Resin cement	Results
[35]	TBS and Shear	In-Ceram	HF; CoJet; 25 μm Al_2O_3 AA. All silanated	Z-100 resin applied directly to ZrO_2 surface	CoJet > AA > HF
[37]	MTBS	In-Ceram	HF; CoJet, Silane; Al_2O_3 AA.	Resin block	CoJet + silane yelded the best BS
[38]	Shear		HF; 25 μm Al_2O_3 AA; bur grinding	Panavia 21, Twinlook, and Super-bond	Grinding + Superbond showed the best BS
[39]	MTBS	Y-TZP	125 μm Al_2O_3 AA, CoJet	Calibra, Clearfil cement, and Rely-X Unicem	Phosphate monomer-containing cement (Clearfil) > other RCs, irrespective of STMs
[40]	Shear	Y-TZP	50 μm Al_2O_3 AA; Korox; Rocatec; flame treatment	Panavia F	No significant differences
[41, 42, 44, 46, 58]	Shear	Y-TZP	Al_2O_3 AA	Alloy Primer; Super Bond Monomer; Metal Primer II; Panavia F, Superbonder	Primers improve BS
[45]	Shear	Y-TZP	Zirconia primer; zirconia primer heat treatment (45°C; 79°C; 100°C)	Panavia F	Heat treatment of zirconia primer improved the early bond strength
[52]	Shear	Y-TZP	Plasma; silanized glass pearls	Variolink II	Glass pearls > Plasma
[57]	TBS	Y-TZP	50 μm Al_2O_3 AA; air-powder-water spray	Variolink II; Panavia F, Heliobond	AA > air-powder-water spray
[61]	Shear	Y-TZP	Rocatec	Ketac-Cem; Nexus; Superbond; Panavia 21; Panavia F; Rely-X Unicem	Panavia 21 yelded the best BS
[62]	TBS	Y-TZP	AA; AA + HF; silica coating	Z100	AA = AA + HF > silica coating
[65]	Shear	Y-TZP	70 μm Al_2O_3 AA; SiC blasted	Zinc phosphate, glass ionomer, and Adhesive resin	Adhesive resin yelded the best BS
[66]	Shear	Digizon-A	Silica coating; 30 μm Al_2O_3 AA; silanized	Panavia EX; Panavia F 2.0; Rely-X Unicem; Bifix QM; Dual Cement; Duo Cement Plus; Multilink Automix; ParaCem; Rely-X ARC; Variolink Ultra; Variolink II	Bifix QM + silica coating yelded the best BS
[67]	Shear	Y-TZP	Rocatec; silanized (Espe Sil); Epricode; α-alumina	Rely-X ARC	The silane, Espe Sil yelded the best BS
[69]	Shear	Y-TZP	110 μm Al_2O_3 AA	Fuji I; Ketac Cem Easymix; Fuji Plus; RelyX Luting; Principle; Ionotite F; Panavia F; Rely-X Unicem	Panavia F = Principle > Fuji Plus= Rely-X Unicem > Rely-X Luting = Ketac Cem> FujiI > Ionotite F
[73]	Pull out	Y-TZP	110 μm Al_2O_3 AA; silanized glass pearls	Zinc phosphate, Panavia 21, and Variolink II	Glass pearls > AA
[76]	Shear	Y-TZP	50 μm Al_2O_3 AA	BisCem; Rely-X Unicem; G-Cem; Maxcem; Clearfil SA	AA > Ctrl. G-Cem + AA yielded the best BS
[77]	Shear	Y-TZP	AA; silica coating; diamond abrasion. HF; silane; zirconia primer	Resin cement	silicoated + silanated > diamond abrasion + zirconia primer > AA + silanated > zirconia primer > AA + zirconia primer
[60]	MTBS	Y-TZP	Silica coating; MDP; silane	Panavia F	Silica coating + silane > silica coating + MDP = Ctrl

Table 3: Continued.

Reference number	BS method	Type of ceramics	STM	Resin cement	Results
[71]	Shear	Y-TZP	AA; YAG laser-irradiated; glaze applied + HF	Clearfil Esthetic Cement	Laser irradiated > glaze applied + HF = AA = Ctrl
[75]	Shear	Y-TZP	CoJet; glaze + HF; glaze + CoJet. All silanized	Panavia F	Glaze + HF > glaze + CoJet = CoJet
[72]	Shear	Y-TZP	AA; CoJet; Er : YAG laser; AA + Er : YAG laser	Rely-X U100, Clearfil Esthetic Cement, and Panavia F	Er : YAG showed lower bond strengths irrespective of RC
[78]	MTBS	Y-TZP	TBC; TBC + zirconia primer	Clearfil Esthetic Cement and Panavia F	TBC + zirconia primer > TBC

MTBS: microtensile bond strength; TBS: tensile bond strength; SIE: selective infiltration etching; AA: air-abrasion; Ctrl: control groups with no STM; RC: resin cement; HF: hydrofluoric acid etching; TBC: tribochemical treatment.

phosphoric acid acrylate monomers, allowing additional chemical bond with zirconium/metal oxides [17, 41, 42].

The surface treatment with primers containing functional monomers such as MDP (e.g., Alloy Primer and Clearfil Ceramic Primer, Kuraray Medical Inc., Japan) or other phosphoric acid acrylate monomer (e.g., Metal/Zirconia Primer, Ivoclar-Vivadent) are often recommended to improve the bonding to ZrO_2. Since results are not always significant, the combination of primers and air-abrasion methods tend to produce better bond strength, especially in longterm [12, 42–45]. The use of new zirconia primers (a mixture of organophosphate and carboxylic acid monomers) or a phosphonic acid monomer (6-MHPA) has been tested showing good immediate results [9, 46].

4.3. Silane Coupling Agents. Silane coupling agents or more precisely trialkoxysilanes are hybrid inorganic-organic bifunctional molecules that are able to create a siloxane network with the hydroxyl (OH) of the Si in the ceramic surface and copolymerize with the resin matrix of composites; also, silanes lower the surface tension of a substrate, wet it, and make its surface energy higher [47]. Thus, a hydrophobic matrix (resin composite) can adhere to hydrophilic surfaces, such as silica, glass, and glassceramics [29]. Different types of silanes have been studied, but none of them were able to show high effectiveness in surfaces with absent or reduced Si content as the surface of ZrO_2 [20, 41, 48–52]. In addition, siloxane bonds may be sensitive to hydrolytic degradation, affecting the stability of the adhesive interface [20, 39, 53]. Organosilanes were also tested (3-methacryloyloxypropyltrimethoxysilane, 3-acryloyloxypropyltrimethoxysilane, or 3-isocyanatopropyltriethoxysilane) with better results for the two first ones [29]. The silane organofunctional groups are generally a methacrylate molecule, but acrylate groups are known to be more reactive than methacrylates [29]. The 3-isocyanatopropyltriethoxysilane is a rare silane, which has not been reported to be used as adhesion promoters in dental materials research.

5. Mechanical Surface Treatments

5.1. Air-Abrasion with Aluminum Oxide Particles. Air-abrasion with aluminum oxide particles (Al_2O_3) has been studied since the nineties and its effectiveness is closely related to the sandblasted ceramic surface and the air abrasion method. Through a scanning electron microscopy (SEM) evaluation in 2003, Borges et al. [7] showed that the air-abrasion with 50 μm Al_2O_3 during 5 s at 4-bar pressure is able to create irregularities on the surface of glass ceramics; however, the same procedure did not change the surface of In-Ceram Alumina, In-Ceram Zirconia, and Procera.

During an evaluation with an optical profilometer, Della Bona et al. [36] showed an increase in the arithmetic roughness (Ra) of In-Ceram Zirconia (from 207 nm to 1000 nm) after the use of 25 μm Al_2O_3 air-abrasion at a distance of 10 mm for 15 s, at a pressure of 2.8 bars. de Oyagüe et al. [22] employed 125 μm Al_2O_3 air-abrasion for 10 s at approximately 5-bar pressure, which resulted in 45.77 nm for Ra, against 9.39 nm of the control group (notreatment), on a yttrium-stabilized tetragonal zirconia (Y-TZP) material (Cercon Zirconia, Dentsply). On the other hand, working with similar ceramics (Lava, 3 M-ESPE), Casucci et al. [54] observed just 7.11 nm for Ra against 6.94 nm on the control group (notreatment).

Since greater roughness was produced over different ZrO_2 based materials, the air-abrasion method must take this fact into consideration. In-Ceram Zirconia should not be classified as pure zirconia ceramics since it is composed of 63% of alumina, 32% of zirconia, and 4% of glass matrix [36]. In addition, alumina is less ductile than zirconia, with larger grains and higher surface hardness, which makes air-abrasion more effective [55]. Contradictory results from the studies of de Oyagüe et al. [22] and Casucci et al. [54] may be possibly related to the different ceramic brands they used.

When it comes to abrasion with Al_2O_3, a wide range of particle size, pressure, distance from ceramic surface, working time, and impact angle have been studied. These differences can help explain contradictory results. Although studies consider the previous factors important [56], the type of the ZrO_2 ceramics may be mandatory. On a yttrium-stabilized tetragonal zirconia (Y-TZP) material, the use of greater particle size (from 50 μm to 150 μm) results in a rougher surface but no significant alteration in bond strength [41]. Evaluating a Y-TZP, Cavalcanti et al. [17] showed an increase in bond strength after air-abrasion with 50 μm Al_2O_3 for 15 seconds at 2.5 bars. With similar ceramics de Oyagüe et al. [22] showed that air-abrasion did not produce higher bond strength, even though the substrate surface became rougher than the control group (as shown in the previous paragraph). Several studies have shown low bond strength values with air-abrasion [20, 39, 54, 57] or even spontaneous debonding after artificial aging (150 days of water storage and repeated thermocycling) on the association of airborne particle abrasion, silane application, and Bis-GMA resin cement [20]. According to Kern et al. [58] air-abrasion without primers can result in higher initial bond strength to zirconia ceramics that will be reduced to zero in long-term evaluation, independent of air-abrasion application pressure.

5.2. Si Deposition Methods. Si deposition methods started in 1984 with the silicoater technology [13], and in 1989 the Rocatec system, a laboratory device, was developed and later the CoJet system, a chairside device was introduced into the market [59]. These systems are based on the use of 110 μm (Rocatec) or 30 μm (CoJet) Si-coated alumina particles that are blasted onto the ceramic surface. Sandblasted ceramics acquires a reactive Si-rich outer surface prone to silanization and the following AC with suitable resin composites. Its use requires silane application before cementation [28, 60]. The tribochemical Si-coating on ceramic surfaces increases the bond strength of resin cement to glass-infiltrated ZrO_2 [5, 24, 59] or Y-TZP [61–64]. Usually, 2.5–2.8-bar air-abrasion pressures are used [4, 23]; however, higher pressure results in higher bond strength with CoJet [55]. In spite of that, some studies still show similar shear bond strength with and without Si-coating by air-abrasion methods [65].

Si deposition by air-abrasion might produce a more silane reactive surface [66], but it also tends to produce a surface with lower roughness and consequently lower possibility of mechanical interlocking with resin cement [22, 29]. Some authors do not show lower roughness [67], but considering this might be a true observation; the enabled chemical interaction to resin cement or coupling agents would justify its use [68].

6. Alternative Treatments

Different alternative methods to treat ZrO_2 surfaces have been proposed and evaluated in order to produce a reliable adhesion, especially in long term. A large range of mechanical, chemical, or both approaches have been tried to modify the ZrO_2 surface to increase the surface bond area, surface energy, or wettability [69].

Plasma spraying (hexamethyldisiloxane) using a reactor (Plasma Electronic, Germany), proposed in a previous study [52], increased the bond strength of resin cement to ZrO_2. The authors related that plasma is a partially ionized gas containing ions, electrons, atoms, and neutral species. However, the mechanism of surface modification and rise of the bond strength remain unclear, and the authors suggested that the improvement in bond strength might be explained by covalent bonds [52].

Some studies have suggested the use of erbium-doped yttrium aluminum garnet (Er:YAG) or CO_2 laser to enhance the bond strength to resin cement [17, 30, 70, 71]; therefore, the effect of laser on the ZrO_2 could be tested with the same aim. Laser application removed particles by microexplosions and by vaporization, a process called ablation. However, bond strength results indicated that the effect of laser irradiation is contradictory. While some studies concluded that lasers are not effective to improve the bond strength between ZrO_2 and resin cement [17, 30, 72], recent research shows the improvement of adhesion after CO_2 laser application in comparison to conventional STM and indicates this technique as an alternative method for bonding to ZrO_2 surfaces.

The applications of micropearls of low fusing porcelain or vapor deposition of silicon tetrachloride ($SiCl_4$) are other types of silicatization methods that have been used, showing improved bond strength [52, 73].

However, the most innovative STM for ZrO_2 was introduced by Aboushelib et al. in 2006 and tested with respect to microtensile bond strength in 2007 [31]. This method was named selective infiltration etching (SIE) and uses principles of heat-induced maturation and grain boundary diffusion to transform the relatively smooth nonretentive surface of Y-TZP into a highly retentive surface. A low temperature molting glass is applied on selected ZrO_2 surfaces and submitted to a heat-induced infiltration process, determining zirconia crystal rearrangements. After that, the glass is removed with a 5% hydrofluoric acid solution bath, leaving intergrain nanoporosities where low-viscosity resin materials may flow and interlock after polymerization [31]. This method was tested in association with MDP-based resin cement, providing high and durable bond strength [31, 32], and with previous application of zirconia primers, providing increased initial bond strength [74] but not a stable bond with artificial aging [33].

Recent studies has shown promising results on bond strengths of Y-TZP/resin cement after glazed ceramic surface is subjected to air-particle abrasion with aluminum oxide and silanization [1] or etching with hydrofluoric acid [75], but a stable bond promoted by these methods is questionable and needs more studies.

Irrespective of the possibility of producing a rough surface with air-abrasion or SIE, these methods still do not completely assure better or durable bond strength, as it could be seen. To overcome this issue, it is clear that micromechanical plus chemical adhesion strategies should be used.

7. Discussion

Several surface treatment methods have been proposed to overcome intrinsic acid resistance of ZrO_2; however, these methods have presented controversial results about their effectiveness on bond strength improvement. Nonetheless it seems important to select multifunctional methods, which mix the ability to create a rough surface for micromechanical interlocking and increase the surface area to establish chemical bond with reactive substances. When testing self-adhesive luting resin cement containing a functional phosphate monomer, Yang et al. [42] showed reliable bond strength after air-abrasion at 2.5 bars or the combination of low pressure air-abrasion and priming with MDP-containing primers. Therefore, air-abrasion seems to be important even

Table 4: Guide to suggested STM for ZrO_2 ceramics.

Ceramics type	Commercial brands	STM	Primers/silane	Resin cement (RC)
Y-TZP ZrO_2	Lava, Cercon, Emax ZIR-CAD, Procera Zirconia	50–150 μm Al_2O_3 air-abrasion, with 2.5 bars, 10 mm distance, for 15 seconds	—	Phosphate monomer containing RC (Rely-X U200, Panavia F, and Panavia 2.1)
			Phosphate monomer containing solutions: Alloy Primer, Clearfil Ceramic Primer, or Kuraray's Clearfil Repair Kit	Dual or chemical cured RC (check compatibility with previous applied solutions)
Glass infiltrated ZrO_2	In-Ceram Zirconia	25 μm Al_2O_3 air-abrasion with 2.8 bars, 10 mm distance, for 15 s.	—	Phosphate monomer containing RC (Rely-X U200, Panavia F, and Panavia 2.1)
		Air-abrasion with Si-coating Al_2O_3 particles (CoJet or Rocatec system), at 4.5 bars, 10 mm distance, for 10 s.	Phosphate monomer containing solutions: Alloy Primer, Clearfil Ceramic Primer, or Kuraray's Clearfil Repair Kit	Dual or chemical cured RC (check compatibility with previous applied solutions)

when working with phosphate monomer resin cement [44] or with conventional self-adhesive resin cement [76]. If conventional resin cement is to be used, primer application seems mandatory for durable adhesion [58].

STM development shows the need for producing high roughness and chemical interaction on ZrO_2 ceramic surfaces. According to the analysis of the selected papers it could be seen that the use of Al_2O_3 air-abrasion followed by application of phosphate monomers-based primers or resin cement tends to produce more reliable results [13, 39, 42, 57, 61]. However, SIE also produces a rough surface, with optimal surface energy and interaction with resin cement [31], with the advantage of being user-controlled and depth-limited.

Similar to phosphate monomer solutions/cements, the use of metal primers can also establish chemical bond with zirconia [17]. Magne et al. [46] showed promising results with a special designed zirconia primer solution. An important point is that a functional monomer is not necessary in the resin luting agent if it is contained in the primer [21]. Optimal results were found with MDP-based resin cement [32]. However, when working with Bis-GMA based resin cement, Si-coating plus silane application produces good results [61, 77]. In addition, according to Kern et al. [58] the use of primers for conventional resin cement will result in long-term bond strength after air-abrasion. Thereby, adhesion to ZrO_2 seems mainly promoted by chemical bonds, either through hydrogen bonds between the polar functional groups of the polymers or monomers in the cement and the polar hydroxyl groups available on ZrO_2 surface or between siloxane bonds on ZrO_2 Si-coated surfaces. Therefore the main function of air-abrasion is to clean and increase the surface area [12, 42, 58], creating the conditions to establish the chemical bonds.

Even with a significant increase of Si ratio (76%) at the surface after Si-coating via air-abrasion (CoJet system) [36], which may enhance bonding to resin by silane coupling, Si-coated surfaces of In-Ceram Zirconia remain almost similar to their original state since an increase from 1.25% to only 2.21% of Si could be noted on the ZrO_2 surface [36]. Y-TZP Si-coated surfaces showed 4.2% of Si (by weight) and 11.2% atomic concentration [29]. The presence of Si is a prerequisite for durable siloxane bonding, and so on, some authors considered the above mentioned Si concentrations at zirconia surfaces too silicon-poor for durable bonding [29]. However, Inokoshi et al. [78] showed that the combination of Si-coating via air-abrasion (CoJet system) and zirconia primers presents durable bond to ZrO_2.

Glass pearls or silicon tetrachloride ($SiCl_4$) vapor deposition is new methods that deserve more research considering the ability to increase Si-concentration [1, 73]. This information reinforces the importance of the chemical bonds for a durable and reliable adhesion between the substrates (resin cement and ZrO_2).

On a Si-coated surface, silane bonding could be complemented by the use of monomers with metal affinity, such as MDP, present in metal and ceramic primers. However, previous silane application would prevent the contact between these functional monomers and the ceramic surface avoiding the hydrogen bond formation, except when the silane solution is mixed with MDP (e.g., Kuraray's Clearfil Repair Kit) [29]. With MDP-resin cement, Atsu et al. [23] proved this assumption, showing that tribochemical Si-coating (CoJet System) and the application of an MDP-containing bonding/silane coupling agent mixture increased the shear bond strength. In addition, according to Tanaka et al. [67] stable shear bond strength can be achieved on Si-coated Y-TZP with the cooperative interaction of hydrogen bond formed between phosphate monomer and ZrO_2 and silane coupling to SiO_2 incorporated to ceramic surface. In this case, silane application will increase ceramic surface wettability for the resin cement rather than establishing siloxane bonds.

Collected observations from all analyzed studies are sometimes conflicting, and this may be specially caused by different testing methods. Table 3 summarized the testing methods, and although most of them employed microtensile tests (the most suitable one for bond strength testing [79]), results are still controversial; thus, more work is necessary to draw definitive conclusions. Based on state-of-the-art concepts, Table 4 contains suggested STM protocols for the AC of ZrO_2 ceramic restorations.

As a final consideration, it is always important to emphasize that the composition of the ZrO_2 to be used is critical when choosing the best surface treatment. In addition, distance, pressure, working time, and particle's composition of air abrasion must be carefully observed. The type of the resin cement may be defined by the type of STM, depending on the compatibility of the selected functional monomer or on the creation of a siloxane bond after considering the presence and quantity of Si at ZrO_2 surface.

8. Conclusions

According the reviewed literature it was possible to conclude that

(1) STMs must be correlated to the type of ZrO_2 and the selected resin cement;

(2) airborne-particle abrasion combined with a resin composite containing phosphate monomers or tribochemical Si-coating plus silane (with functional monomers) coating combined with conventional Bis-GMA resin cement could be considered the best STMs;

(3) primer development seems to be an effective method to improve bond strength to ZrO_2;

(4) alternative STMs (selective infiltration etching or Si-coating or plasma treatment) have presented considerable surface alterations and have shown promising results; however, further studies are necessary.

Disclosure

The authors disclose no commercial interest in products or companies mentioned in the paper.

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