Microtensile bond strength and scanning electron microscopic evaluation of zirconia bonded to dentin using two self-adhesive resin cements; effect of airborne abrasion and aging

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Aim of the study: This in vitro study was conducted to evaluate the microtensile bond strength (μTBS) of surface treated zirconia bonded to dentin specimens using two aged contemporary dual cured self-adhesive resin cements.

Materials and methods: Sixty cuboidal-shaped zirconia ceramic specimens were obtained using CAD/CAM system. Specimens were divided into two equal main groups; 30 specimens each, gp A in which specimens did not receive any further surface treatment & gp B in which only one surface of each specimen was airborne abraded. Each group was then divided into two equal groups; 15 each, according to the type of adhesive resin cement used for bonding zirconia specimens to ground dentine surfaces; RelyX™ U200 (cement I) and Multilink® Speed (cement II). The assemblies were further subdivided into 3 equal subgroups; 5 assemblies each, according to aging protocol. The aging protocols were storage in distilled water for 1 day, for 7 days without thermocycling and for 7 dayses followed by thermocycling; subgroups 1, 2 and 3 respectively. After aging, the assemblies were sectioned into beams approximately 1mm² in cross section resulting in 25 beams for each subgroup; 20 of them were selected for μTBS (n = 20) and 5 were kept for SEM examination.

Results: Group B showed statistically significantly higher mean micro tensile bond strength value than group A. The type of cement had statistically insignificant effect on mean micro tensile bond strength. Thermocycling significantly reduced μTBS of both cements bonded to untreated zirconia ceramic; IA3 and IIA3 subgroups.

For SEM, cement I showed gaps at its interface with zirconia groups A and B regardless of aging protocol. Cement II showed only gaps at its interface with zirconia ceramic group A only but good adaptation appeared at its interface with zirconia ceramic group B for aged for 1 day (subgroup IIB1) and 7 days without thermocycling (subgroup IIB2). However, cement II bonding air abraded zirconia ceramic followed by thermocycling (subgroup IIB3) showed both gap free as well as gap containing areas at high magnification only.

Conclusions: Airborne abrasion-surface treatment of zirconia significantly enhanced the μTBS of both cements adhered to dentin while aging had an adverse effect. MS showed higher insignificant μTBS.

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1. Introduction

The use of all-ceramic materials has been increasing due to their high biocompatibility and improved esthetics. There are many types of all ceramic materials; zirconia and lithium disilicate are the most popular types used [1].

Zirconia is a polycrystalline material which can exhibit structural polymorphism. Pure zirconia is monoclinic at room
temperature and stable up to 1179 °C. Above this temperature, it transforms to a denser tetragonal phase with 5% volume decrease. The tetragonal form is stable between 1170 and 2370 °C, while at higher temperatures ZrO₂ transforms to cubic structure. During cooling, tetragonal turns back to monoclinic, accompanied with 3–4% volume expansion [2]. Several different oxides are added to zirconia to stabilize the tetragonal phases at room temperature as magnesium (MgO), yttrium (Y₂O₃), and ceria (CeO₂) [3]. Stabilizing the tetragonal phases at room temperature is of prime importance to reinforce the material through phase transformation toughening [4].

Establishing a strong and stable bond to zirconia surface is difficult, as the material is acid resistant and does not respond to common etching and silanization procedures used with other glass containing ceramic materials [5]. To obtain a strong bond between zirconia and cement, zirconia surface could be treated with several methods such as plasma, hot chemical etching solution, treatment with erbium: yttrium aluminum-garnet (Er: YAG) or neodymium: yttrium-aluminum-garnet (Nd: YAG) laser, using functional adhesive monomers, zirconia ceramic powder coating, nano-alumina coating and air-abrasion with aluminum oxide particles (Al₂O₃). The later could be used with a wide range of particle size, pressure, working time, impact angle and distance between the nozzle and zirconia surface [3,4,6,7].

The success of an indirect restoration largely depends of the luting agent utilized [8]. Resin cements are the luting agents of choice for zirconia because of their ability to reduce fracture of the ceramic structure and the range of shade available to produce optimal esthetic appearance [9]. Self-adhesive cements are the latest introduced subgroup of resin cements. They simplified the luting procedures by being directly applied on the tooth structure and the ceramic substrate without need to previous treatment. In addition, they are claimed to reduce post-operative sensitivity that produced by total etch resin cements. The bonding mechanism of the self-adhesive resin cements is based on a micromechanical retention and chemical interaction. The chemical reaction is established between multifunctional phosphate based monomers of the cement to the hydroxyapatite crystals of the teeth [10]. Reactions may also occur between the zirconium oxide and the phosphate monomer present in the self-adhesive resin cements [11–13].

Self-adhesive resin cements can make adequate bond with zirconia surface treated with Al₂O₃. This in-vitro study was conducted to evaluate microtensile bond strength (μTBS) and intimacy of contact of two aged contemporary dual cured self-adhesive resin cements bonding airborne abraded zirconia to dentin.

2. Materials and methods

2.1. Teeth preparation

Sixty caries and crack — free human maxillary first premolars extracted for orthodontic purposes from patients 18–20 years old were collected. Following the ethical protocol of the Faculty of Dentistry, Minia University, Minia, Egypt. They were then immersed in distilled water with 0.1% Thymol solution and stored at 4 °C to inhibit microbial growth, for maximum one month. Later, the roots of the extracted teeth were embedded in acrylic resin blocks, (Acrostone, Egypt). The mesial surfaces of the teeth were ground parallel to their longitudinal axis by a diamond disk (BesQual Diamond Disk, DIA #6, Korea) under copious amounts of water coolant till the underlying flat dentin surface was exposed. The diamond disk was changed every 10 teeth. The exposed surfaces were finished and polished by silicon carbide papers (E.C MOORE Company, 48126, USA). Afterwards, the ground teeth were sectioned horizontally through their cemento — enamel junctions and their coronal portions were collected.

2.2. Zirconia specimens’ preparation and grouping

A specially constructed cuboidal Teflon block (3 x 4 x 5 mm) was constructed. The block was then laser scanned to cut 60 standardized zirconia specimens (ICE Zirkon Translucent ZirkonZhan, Italy) by computer aided design/computer aided manufacturing (CAD/CAM). Half of the zirconia specimens; 30 specimens each, were kept untreated (no treatment; gp A), while in the other half of the specimens only one surface (4 x 5 mm) was airborne abraded with 100 μm Al₂O₃ particles (airborne abraded; gp B). In gp B, abrasive particles were applied for 20 s at a pressure of 0.4 MPa, perpendicular to the selected surface of each specimen. The distance between the nozzle and the surface was fixed at 10 mm. Separately, the specimens of each group were then ultrasonically cleaned in distilled water for 10 min to remove loosely attached Al₂O₃ particles in gp B and surface contaminants in gps A and B. Afterwards, gentle air drying was performed using oil — free air spray. Two types of dual cured self — adhesive resin cements were used in the current study; RelyX™ U200 (cement I) (3 M ESPE, Germany, LOT 561723) and Multilink™ Speed (cement II) (Ivoclar Vivadent, Liechtenstein, LOT S05050).

Fifteen specimens from gp A as well as another 15 specimens from gp B were cemented to dentin surfaces of the teeth using cement I; designated as assemblies IA and IB respectively. The remaining specimens were cemented to dentin using cement II, designated as assemblies IIA and IIB respectively as well. Cements were used according to the manufacturers’ instructions. Both cements were mixed in 1:1 base to catalyst ratio through the auto-mixing tips and light cured by LED light-curing unit (CIXO BD-686-Ib, China) at intensity of 1600 mW/cm². Initially, curing was done for 4 s, to allow for removal of excess cement by a scaler. Additional curing for 20 s was performed from the buccal as well as from the palatal sides of the bonded assemblies to obtain optimal polymerization. Luting procedures were carried out under a constant load of 0.5kg at room temperature.

Each assembly category (IA, IIA, IB, IIB) was further subdivided into 3 equal subgroups; 5 assemblies each, according to aging procedures. The first and second subgroups (assigned as 1 & 2 respectively) were aged by storing assemblies in distilled water at room temperature for 1 day and 7 days respectively. The third subgroup (assigned as 3) was aged under same conditions as for subgroup 2 then followed by thermocycling for 500 cycles at temperatures 5 °C and 55 °C with a dwelling time of 30 s in each bath and transferring time of 4 s (ISO TR 11450). The factorial design of the current study is represented in Table 1.

2.3. Microtensile bond strength test (μTBS)

The assemblies of each subgroup were bonded from their zirconia sides by epoxy resin (4 Minutes Steel Epoxy, Boosill, Malaysia) to metallic lead bases which were then fixed into a linear precision saw (Isomet 4000, Buehler Ltd, Lake Bluff, IL, USA). Assemblies were then vertically sectioned into slabs, approximately 1 mm in thickness, perpendicular to the adhesive cement interface. Sectioning was done using a diamond disc (Isomet, Buehler, wafering blade, 20LC, 11–4225, USA) with 0.34 mm thickness under copious amounts of water coolant at speed of 600 rpm and feed rate of 3.3 mm/min. Further sectioning perpendicular to the first one was done to cut the slabs into about 12 beams with a cross-sectional bonded area of approximately 1 mm². Another horizontal section parallel to the adhesive cement interface, at the junction between zirconia and lead base, was done to separate the beams.
3. Results

Results revealed that cement II produced stronger microtensile bond bond (5.21 ± 3.55 MPa) than cement I (4.54 ± 2.74 MPa), however this difference was statistically insignificant, Fig. 1.

Air abrasion specimens (gp B) showed significantly higher mean μTBS values (6.73 ± 2.9 MPa) compared to non-treated (gp A) ones (2.42 ± 1.46 MPa), Fig. 2.

Thermocycling of subgroup 3 significantly reduced μTBS values compared to aging for 1 and 7 days without thermocycling for subgroups 1 and 2. The difference in the mean μTBS between 1 and 7 days aging was statistically insignificant. The mean values for subgroups 1, 2 and 3 were 5.78 ± 3.54 MPa, 5.29 ± 3.13 MPa and 3.58 ± 2.59 MPa respectively, Fig. 3.

Generally, there was reduction in μTBS with progressing aging conditions. The reduction in μTBS was generally statistically insignificant for both cements. However, the reduction of bond strength with cement II was more obvious than that with cement I, and the least mean bond strength values were recorded with subgroup IA3, Fig. 4.

SEM examination of all examined assemblies in gp A (no treatment group) showed gaps at zirconia/cement interface when examined at magnification 500X regardless of the aging method or the cement type.

In gp B (air abraded group), at magnification of 500X, gaps were detected at zirconia/cement I interface of all assemblies regardless of aging method, Fig. 5 (a, c and e). On the other hand, zirconia/ cement II interface of assemblies aged by the 1st two methods was gap free at 500X up to 2000X, Fig. 5 (b and d). Aging by storage followed by thermocycling of assemblies cemented by cement II, didn't reveal gaps at zirconia/cement II interface at magnification 500X, but increasing power of magnification to 1000X showed gap free as well as gap containing areas at zirconia/cement interface, Fig. 5 (f).

4. Discussion

For bond strength evaluation, many mechanical testing methods as shear, tensile, and microtensile tests have been suggested. For accurately measuring the bond strength between an adhesive and a substrate, the bonding interface should be the most stressed region. However, many studies reported that some bond strength tests do not appropriately stress the interfaceal zone as in shear bond strength test [13–16] and macrotensile bond strength test [13,14].

Table 1

<table>
<thead>
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<th>Factorial experimental design.</th>
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<tr>
<td><strong>Cement type</strong></td>
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<tr>
<td>No surface treatment (Gp A, 30 specimens)</td>
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<tr>
<td><strong>Aging protocol</strong></td>
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<tr>
<td><strong>Thermocycling</strong></td>
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<td><strong>Labeling of the subgroups</strong></td>
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<td><strong>Number of assemblies in each subgroup</strong></td>
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In contrary, microtensile bond strength test (μTBS) has several advantages over other bond strength testing methods. It allows appropriate alignment of the samples leading to more homogeneous distribution of stress. In addition, it provides better economic use of samples, better control of regional differences and gives the ability to test irregular surfaces. Therefore, it could be considered the most sensitive method used for evaluating and comparing of bond strengths [14-20].

It was claimed that the cut zirconia with inherent roughness would be adequate for bonding with luting cements through micromechanical interlocking, and further surface treatments to zirconia would be unnecessary [21]. According to the manufacturer, Multilink® Speed cement contains an adhesive monomer consisting of a long-chain methacrylate with a phosphoric acid group in its composition. This chain is able to establish a stable chemical bond to zirconium oxide [22].

On the other hand, many researchers, most zirconia ceramics’ manufactures as well as luting agents’ producers recommend surface treatment for zirconia, and they emphasize that airborne-particle abrasion would be the surface treatment of choice [16]. Air abrasion with Al2O3 produce surface roughness that provides a larger surface area for micromechanical retention [23-25], it improves surface energy and wettability to zirconia surface. [16] Furthermore, it is one of the best methods to remove organic contaminants from ceramic surface [26-28] and hence, the bonding surface could be activated [29]. It would be uncommon to use dentin as substrate onto which zirconia is cemented and sectioned to test μTBS due to great variations in dentin microstructure.

These variations would generate discrepancy in results [13,35,36]. However the clinical need for studying complex structures formed of ceramics cemented to tooth structure would be more interesting [24,37].

Ground proximal surfaces of premolars have the widest exposed flat superficial dentin. The superficial dentin surface is rich in collagen fibrils than deep dentin. Therefore, the bond strength was significantly higher to superficial dentin than to deep one due to the opportunity for stronger micromechanical bonding to collagen fibrils in dentin. [30] Adhesion procedures were done under constant load that ensured intimate adaptation of relatively viscous cement to the adherend surfaces. [31] Also the application of sustained seating pressure during luting procedures was to improve the final bond strength of the resin cement [32].

The most widely aging technique used in in-vitro studies is simple thermocycling [18,33], where repeated cycles of alternating temperatures result in mechanical stress [34]. Cutting of specimens at high speed reduces disk oscillation and consequently minimize specimen surface damage [38]. However, in the current study, speeds above 600 rpm resulted in premature debonding of assemblies. This might be due to converting of cutting energy into heat leading to thermal damage of the assemblies that affect the properties of its components. [39] So, 600 rpm was the chosen speed to cut the assemblies.

In comparison to previous studies, the bond strength data reported in the current one were generally different, and this difference would be due to lack of standardized testing methods across studies. [40] Also variations in results might be related to difference in marketed commercial products, the methods of specimens’ preparation or geometrical differences related to size and orientation of the specimen during testing. [27].

The statistically insignificant difference in μTBS results between the two tested cements could be attributed to their chemical
composition where both cements are based on the presence of methacrylate monomers containing phosphoric acid groups responsible for bonding to both zirconia and dentin. [22,42] However the difference in presence of interfacial gaps as demonstrated by the SEM is might be attributed to the difference in their rheological properties. Thank to the new rheological modifiers added to the composition of cement I, its viscosity has been reduced compared to its predecessors; RelyX™ Unicem Aplicap™/Maxicap™ and RelyX™ U100, however, its laboratory use showed still higher viscosity and less spreadability onto substrate surface compared to cement II reducing its ability to infiltrate into surface irregularities. This was demonstrated by the SEM examination; where gaps were clearly seen at zirconia/cement I interface at as low magnification as 500X, Fig. 5 (a, c and e), indicating higher viscosity of cement I and hence lack of intimacy to zirconia. Some researchers reported that cements’ bonding capacities are related to combined effect of their ability to infiltrate into substrate’s surface irregularities hand in hand with their mechanical properties that is greatly affected by the amount of fillers content. [16,30,41] According to the manufacturers, the amount of silanized fillers in cement I was 72 wt% [42], while their amount in cement II was only 61 wt% [22]. The presence of silanized fillers in the resin matrix increases its mechanical properties and decreases its solubility. [43] Hence, cement I could be mechanically stronger than cement II due to higher content of silanized filler, however, cement II is less viscous and has more penetrating ability than cement I. Accordingly, the effect of both cements could be balanced and this might explain the insignificant difference in μTBS.

The reduction in μTBS with progressing aging conditions with cement II was more obvious than that with cement I. This could be attributed to the higher solubility, higher water sorption and less flexural strength of cement II [22] compared to cement I [42]. Flexural strength is an indicator for the mechanical properties of luting agent. Luting cements with high mechanical properties are more resistant to aging conditions. [16] Bonding performance is more related to the organic matrix than to the inorganic fillers. The latter is more related to the organic matrix than to the inorganic fillers. [10].

The progressing reduction in μTBS with aging conditions could be explained by deterioration of the mechanical properties of resin cements with water storage. [27] The reduction in bond strength after thermocycling might be explained by three major mechanisms. The first mechanism would be the great mismatch in the coefficient of thermal expansion between different surfaces at the adhesive junction, leading to mechanical stresses at dentin/cement and cement/zirconia interfaces resulting in degradation of the bond. [16,17,44] The linear coefficients of thermal expansion (α) are 10.5 ppm/°C and 8.3 ppm/°C for zirconia-based ceramics (3Y-TZP) and dentin respectively. For composite resin, α ranges from 25 to 68 ppm/°C according to the fillers content [45]. The second mechanism by which thermocycling would affect bond strength could be the degradation of the resin cement itself, which might be due to interfacial failure at fillers/matrix interface. [16,17] The third one might be generated by the effect of hot water which would accelerate extraction of poorly polymerized resin monomers. [44]

Negative effect of thermocycling on bond strength in the current study came in contradiction with a study that demonstrated insignificant increase in shear bond strength for indirect composite material cemented to zirconia. [29] This conflict could result from cutting of assemblies in the current study in the form of microbars with much higher exposed total surface area to thermocycling than do shear test specimens. [46] In addition, cutting of microbars had already stressed adhesive junction after thermocycling. Combination of both cutting and thermocycling stresses would be responsible for significant reduction in μTBS.

The significantly increased μTBS of both cements bonded to gp (B) than to gp (A) could have resulted from the higher surface energy and better wettability of zirconia surfaces treated with Al2O3 air abrasion than untreated zirconia. [16,36] Moreover, Al2O3 air abrasion is one of the best methods to remove any organic contaminants from ceramic surface. [27,28] Hence, the poor spreadability of cements onto untreated zirconia might have been the reason to the presence of the gaps, as demonstrated by SEM.
micrographs at zirconia/cements interface at as low magnification as 500X, contributing to their lower µTBS.

For gp (B), the presence of gaps in SEM at zirconia/cement I interface of all assemblies at 500X under magnification 500X might be due to the poorer spreading ability of cement I emerging from its higher viscosity that hindered its flow and adhesion to zirconia surfaces reducing its micromechanical retention. [47]

The synergistic effect of lacking of zirconia surface treatment, high viscosity of cement I and thermocycling could explain the marked reduction in µTBS of subgroup Iα3 compared to all other subgroups.

5. Conclusions
Within the limitations of the current study, airborne abrasion had significantly improved µTBS of zirconia adhesively cemented to dentin, especially under thermocycling aging condition, than did the cement type.

Conflict of interest
The authors declare no conflict of interest to disclose.

References


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