

2017

## 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

Reem Gamal  
dr.reem.gamal@gmail.com

Yasser F. Goma  
yaserfh@gmail.com

Mostafa A. Abdellatif  
mostafa.eldessouky@fue.edu.eg

Follow this and additional works at: <https://digitalcommons.aaru.edu.jo/fdj>



Part of the [Medicine and Health Sciences Commons](#)

---

### Recommended Citation

Gamal, Reem; F. Goma, Yasser; and A. Abdellatif, Mostafa (2017) "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," *Future Dental Journal*: Vol. 3 : Iss. 2 , Article 3.  
Available at: <https://digitalcommons.aaru.edu.jo/fdj/vol3/iss2/3>

This Article is brought to you for free and open access by Arab Journals Platform. It has been accepted for inclusion in Future Dental Journal by an authorized editor. The journal is hosted on [Digital Commons](#), an Elsevier platform. For more information, please contact [rakan@aarj.edu.jo](mailto:rakan@aarj.edu.jo), [marah@aarj.edu.jo](mailto:marah@aarj.edu.jo), [u.murad@aarj.edu.jo](mailto:u.murad@aarj.edu.jo).



# 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



Reem Gamal <sup>a</sup>, Yasser F. Gomaa <sup>a</sup>, Mostafa A. Abdellatif <sup>b,\*</sup>

<sup>a</sup> Biomaterials Department, Faculty of Dentistry, Minia University, Egypt

<sup>b</sup> Faculty of Oral and Dental Medicine, Future University in Egypt (FUE), Egypt

## ARTICLE INFO

### Article history:

Received 6 May 2017

Accepted 16 July 2017

Available online 19 July 2017

### Keywords:

Dual – cured self adhesive resin cements

Zirconia

Microtensile bond strength

Airborne abrasion

Aging

## ABSTRACT

**Aim of the study:** This *in vitro* study was conducted to evaluate the microtensile bond strength ( $\mu$ TBS) 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 flat 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 7days followed by thermocycling; subgroups 1, 2 and 3 respectively. After aging, the assemblies were sectioned into beams approximately 1 mm<sup>2</sup> in cross section resulting in 25 beams for each subgroup; 20 of them were selected for  $\mu$ TBS (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  $\mu$ TBS 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  $\mu$ TBS of both cements adhered to dentin while aging had an adverse effect. MS showed higher insignificant  $\mu$ TBS.

© 2017 Faculty of Oral & Dental Medicine, Future University. Production and hosting by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## 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

\* Corresponding author. Faculty of Oral and Dental Medicine, Future University in Egypt (FUE), End of 90th St., Fifth Settlement, New Cairo, Egypt.

E-mail addresses: [dr.reem.gamal@gmail.com](mailto:dr.reem.gamal@gmail.com) (R. Gamal), [yaserfh@gmail.com](mailto:yaserfh@gmail.com) (Y.F. Gomaa), [mostafa.eldessouky@fue.edu.eg](mailto:mostafa.eldessouky@fue.edu.eg) (M.A. Abdellatif).

Peer review under responsibility of Faculty of Oral & Dental Medicine, Future University.

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 ZrO<sub>2</sub> 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 magnesia (MgO), yttria (Y<sub>2</sub>O<sub>3</sub>), and ceria (CeO) [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, laser treatments 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 (Al<sub>2</sub>O<sub>3</sub>). 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 Al<sub>2</sub>O<sub>3</sub>. This *in-vitro* study was conducted to evaluate microtensile bond strength ( $\mu$ TBS) 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 × 4 × 5mm) 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 × 5 mm) was airborne abraded with 100  $\mu$ m Al<sub>2</sub>O<sub>3</sub> 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 Al<sub>2</sub>O<sub>3</sub> 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 (COXO BD-686-Ib, China) at intensity of 1600 mW/cm<sup>2</sup>. 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 ( $\mu$ TBS)

The assemblies of each subgroup were bonded from their zirconia sides by epoxy resin (4 Minutes Steel Epoxy, Boossil, 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 mm<sup>2</sup>. Another horizontal section parallel to the adhesive cement interface, at the junction between zirconia and lead base, was done to separate the beams

from the lead bases. Only five beams were chosen from each assembly, so a total of 25 beams were collected from each subgroup; 20 beams were tested for  $\mu$ TBS ( $n = 20$ ) and 5 beams were kept for scanning electron microscope (SEM). Any beam that failed during specimen preparation was replaced by a similar one.

The exact dimensions of each beam was evaluated using a digital caliper (digital caliper 150 mm (6"), FG-900125-CS-013, China) and the exact bonded area was considered in the calculations. A specially designed stainless steel attachment consisted of 2 halves freely moving along their axial direction was constructed. One half had 2 pins to be secured in place in 2 opposing pin halls in the corresponding half. The test bars were glued into a slot engraved axially along the 2 halves with a cyanoacrylate adhesive (Amir A2000, oc-cyanoacrylate adhesive, china).

The attachment was then fixed to a universal testing machine (3345, Instron, 2519–104, 3345, Canton, MA, USA) and stressed to failure under tension at a crosshead speed of 0.5 mm/min. Microtensile bond strength was calculated in MPa by the machine computer software using the equation:  $\sigma = F/A$ ; where  $\sigma$  is the  $\mu$ TBS,  $F$  is the maximum force at failure in Newton exerted on the beam and  $A$  is the area of bonding in  $\text{mm}^2$ .

#### 2.4. Scanning electron microscope (SEM)

Beams were inserted in high vacuum machine for dehydration and subjected to gold sputtering for higher resolution imaging. Afterwards, they were examined by SEM (JEOL, JXA-840A, Electron probe microanalyzer, Japan) at different magnification powers to detect intimacy of contact at the zirconia/cement interface.

#### 2.5. Statistical analysis

The obtained values were statically analyzed by statistical Package for Social Sciences IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 22 for Windows. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. ANOVA and Independent  $t$ -test were used to study the effect of different variables on mean  $\mu$ TBS. The significance level was set at  $P \leq 0.05$ .

### 3. Results

Results revealed that cement II produced stronger micro tensile bond ( $5.21 \pm 3.55$  MPa) than cement I ( $4.54 \pm 2.74$  MPa), however this difference was statistically insignificant, Fig. 1.

Air abraded specimens (gp B) showed significantly higher mean  $\mu$ TBS values ( $6.73 \pm 2.9$  MPa) compared to non-treated (gp A) ones ( $2.42 \pm 1.46$  MPa), Fig. 2.

Thermocycling of subgroup 3 significantly reduced  $\mu$ TBS values compared to aging for 1 and 7 days without thermocycling for

subgroups 1 and 2. The difference in the mean  $\mu$ TBS between 1 and 7 days aging was statistically insignificant. The mean values for subgroups 1, 2 and 3 were  $5.78 \pm 3.54$  MPa,  $5.29 \pm 3.13$  MPa and  $3.58 \pm 2.59$  MPa respectively, Fig. 3.

Generally, there was reduction in  $\mu$ TBS with progressing aging conditions. The reduction in  $\mu$ TBS 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 interfacial zone as in shear bond strength test [13–16] and macrotensile bond strength test [13,14].

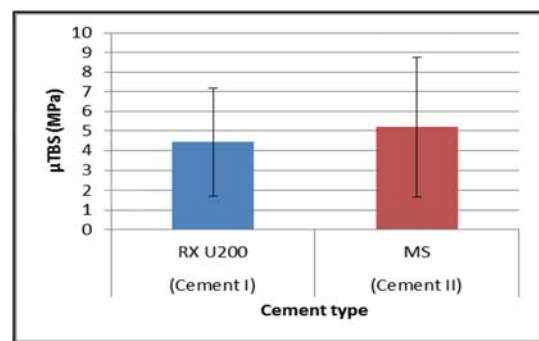


Fig. 1. Histogram showing mean ( $\pm$ SD)  $\mu$ TBS values of both cements.

Table 1

Factorial experimental design.

60 zirconia cuboidal specimens												
Surface treatment	No surface treatment (Gp A, 30 specimens)						Air abrasion (Gp B, 30 specimens)					
Cement type	15 specimens were cemented with RelyX (cement I) (Assembly IA)			15 specimens were cemented with Multilink Speed (cement II) (Assembly IIA)			15 specimens were cemented with RelyX (cement I) (Assembly IB)			15 specimens were cemented with Multilink Speed (cement II) (Assembly IIB)		
Aging protocol	1 day (1)	7 days (2)	7 days followed by Thermocycling (3)	1 day (1)	7 days (2)	7 days followed by Thermocycling (3)	1 day (1)	7 days (2)	7 days followed by Thermocycling (3)	1 day (1)	7 days (2)	7 days followed by Thermocycling (3)
Labeling of the subgroups	IA1	IA2	IA3	IIA1	IIA2	IIA3	IB1	IB2	IB3	IIB1	IIB2	IIB3
Number of assemblies in each subgroup is 5 assemblies.												

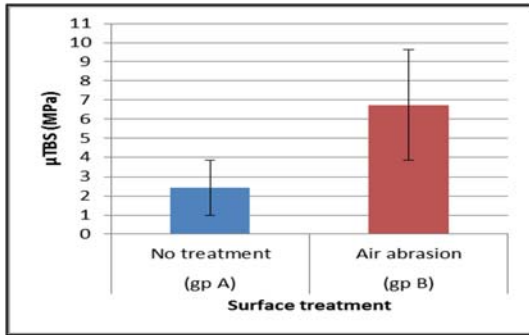


Fig. 2. Histogram representing the effect of surface treatment on μTBS values' means (±SD).

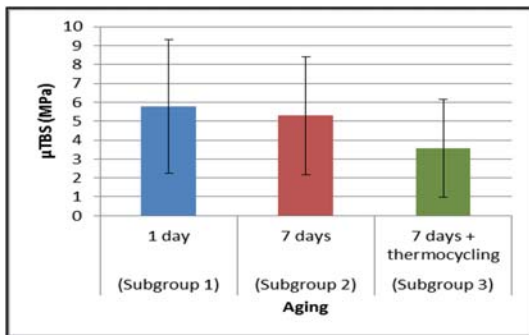


Fig. 3. Histogram revealing the effect of aging on μTBS values' means (±SD).

In contrary, microtensile bond strength test ( $\mu$ TBS) 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<sup>®</sup> 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  $Al_2O_3$  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  $\mu$ TBS 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 exposable 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  $\mu$ TBS results between the two tested cements could be attributed to their chemical

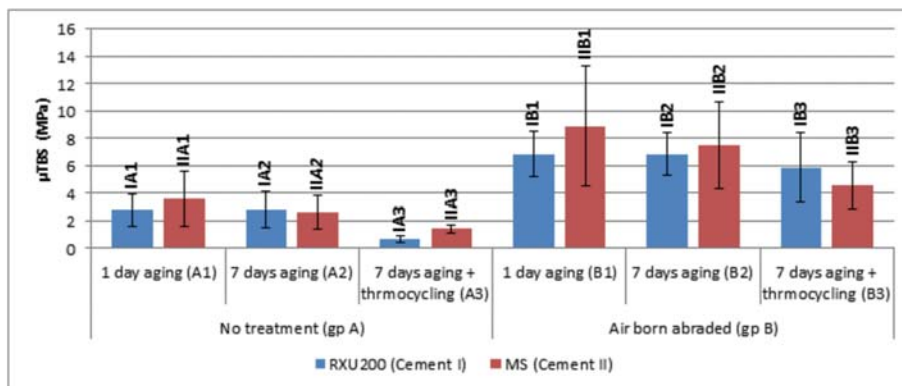
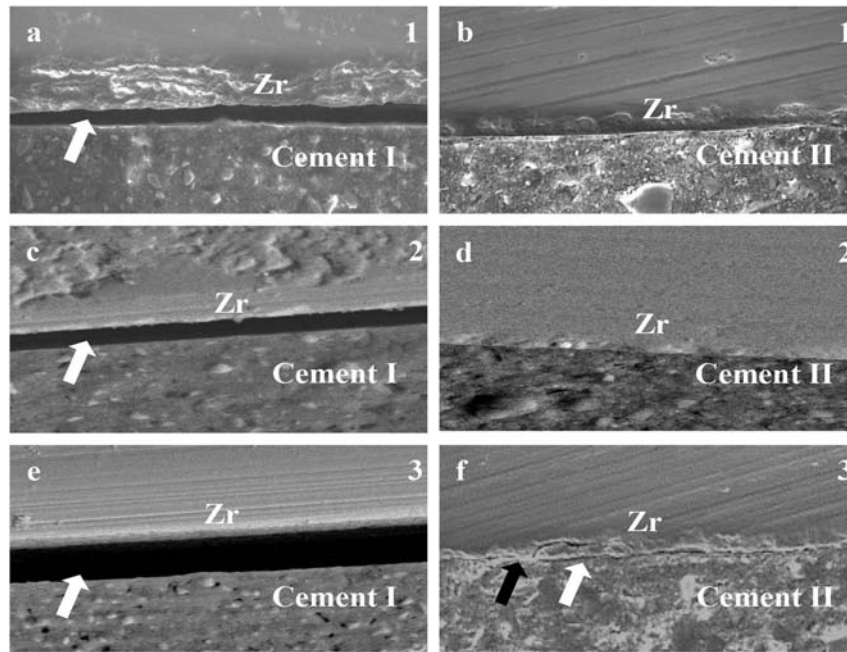


Fig. 4. Histogram showing means (±SD) μTBS of both cements bonded to zirconia with different surface treatments under different aging conditions.



**Fig. 5.** SEM for group B. Micrographs (a) and (b) represent subgroup 1, micrographs (c) and (d) represent subgroup 2, while micrographs (e) and (f) represent subgroup 3. Micrographs (a), (c) and (e) show gaps (arrows) at zirconia (Zr)/cement I interface, (500X). Micrographs (b) and (d) show gap free zirconia/cement II interface, (2000X). Micrograph (f) shows discontinuous gaps at zirconia/cement II interface; white arrow refers to gaps, while black one refers to gap free areas, (1000X).

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  $\mu$ TBS.

The reduction in  $\mu$ TBS 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 responsible for mechanical properties. [10].

The progressing reduction in  $\mu$ TBS 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 explicated 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 ( $\alpha$ ) are 10.5 ppm/°C and 8.3 ppm/°C for zirconia-based ceramics (3Y-TZP) and dentin respectively. For composite resin,  $\alpha$  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  $\mu$ TBS.

The significantly increased  $\mu$ TBS 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 Al<sub>2</sub>O<sub>3</sub> air abrasion than untreated zirconia. [16,36] Moreover, Al<sub>2</sub>O<sub>3</sub> 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 gapes, as demonstrated by SEM

micrographs at zirconia/cements interface at as low magnification as 500X, contributing to their lower  $\mu$ TBS.

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 spreadability 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  $\mu$ TBS of subgroup IA3 compared to all other subgroups.

## 5. Conclusions

Within the limitations of the current study, airborne abrasion had significantly improved  $\mu$ TBS 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

- Conrad HJ, Seong W-J, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. *J. Prosthet. Dent. Nov.* 2007;98(no. 5):389–404.
- Giordano R, Sabrosa CE. Zirconia: material background and clinical application. *Compend. Contin. Educ. Dent.* 2010;31(no. 9).
- Obradovic-Djuricic K, Medić V, Dodić S, Gavrilov D, Antonijević D, Zrilić M. Dilemmas in zirconia bonding: a review. *Srp. Arh. Celok. Lek.* 2013;141(no. 5–6):395–401.
- Saridag S, Tak O, Alniacik G. Basic properties and types of zirconia: an overview. *World J. Stomatol* 2013;2(no. 3):40–7.
- Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. *J. Prosthet. Dent.* 2007;98(no. 5):379–88.
- Chen L, In Suh B. Bonding of resin materials to all-ceramics: a review Liang Chen and Byoung In Suh Research and Development, BiscoInc. *Curr. Res. Dent.* 2012;3(no. 1):7–17.
- Mattiello RDL, Coelho TK, Insaurralde E, Coelho AAK, Terra GP, Kasuya AVB, et al. A review of surface treatment methods to improve the adhesive cementation of zirconia-based ceramics. *Int. Sch. Res. Not. Biomater* 2013;2013. p. 185376 (10 pp.)–185376 (10 pp.).
- Basso GR, Monteiro S, de Andrada MAC. Bond strength of resin cements to ceramic. *J. Mater. Sci. Eng.* 2013;11(no. 3):763–9.
- Anusavice KJ, Shen C, Rawls HR. *Phillips' science of dental materials*, 12th; 2013.
- Gatin E, Matei E, Pirvu DA, Galbinas BM, Iordache S. Comparative survey of the most used self adhesive dental cements based on resin composites. *Dig. J. Nanomater. Biostructures Mar.* 2012;7(no. 1):207–15.
- Griffin J, Suh B, Liang C, Brown D. Surface treatments for zirconia bonding: a clinical perspective. *Can. J. Restor. Dent. Prosthodont* 2010;3:23–9.
- Chen C, Kleverlaan CJ, Feilzer AJ. Effect of an experimental zirconia-silica coating technique on micro tensile bond strength of zirconia in different priming conditions. *Dent. Mater* 2012;28(no. 8):e127–34.
- Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. *Int. J. Prosthodont Jan.* 2005;18(no. 1):60–5.
- Valandro LF, Ozcan M, Amaral R, Vanderlei A, Bottino M a. Effect of testing methods on the bond strength of resin to zirconia-alumina ceramic: micro-tensile versus shear test. *Dent. Mater.* J 2008;27(no. 6):849–55.
- Aboushelib MN, Ghoniem M, Mirmohammadi H, Salameh Z. General principles for achieving adequate bond to all- ceramic restorations. *J. Dent. Oral Hyg* 2009;1(no. 3):36–41.
- Attia A. Bond strength of three luting agents to zirconia ceramic - influence of surface treatment and thermocycling. *J. Appl. Oral Sci.* 2011;19(no. 4):388–95.
- Valandro LF, Mallmann A, Della Bona A, Bottino MA. Bonding to densely sintered alumina- and glass infiltrated aluminum/zirconium-based ceramics. *J Appl Oral Sci* 2005;13(no. 1):47–52.
- Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al. Relationship between bond-strength tests and clinical outcomes. *Dent. Mater* 2010;26(no. 2):100–21.
- Osorio R, Castillo-de Oyagüe R, Monticelli F, Osorio E, Toledano M. Resistance to bond degradation between dual-cure resin cements and pre-treated sintered CAD-CAM dental ceramics. *Med. Oral Patol. Oral Cir. Bucal* 2012;17(no. 4):669–77.
- Sirisha K, Rambabu T, Ravishankar Y, Ravikumar P. Validity of bond strength tests: a critical review-Part II. *J. Conserv. Dent Sep.* 2014;17(no. 5):420–6.
- Aleisa K, Alwazzan K, Al-Dwairi ZN, Almoharib H, Alshabib A, Aleid A, et al. Retention of zirconium oxide copings using different types of luting agents. *J. Dent. Sci* 2013;8(no. 4):392–8.
- Scientific documentation multilink speed. IvoclarVivadent AG 2010.
- Della Bona A, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. *Braz. Oral Res* 2007;21(no. 1):10–5.
- Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. *Oper. Dent* 2009;34(no. 3):280–7.
- Moezzyzadeh M, Nojehdehyan H, Haghi HV. The effect of different coating methods on resin band strength to zirconia, vol 31; 2013. p. 95–103. no. 1.
- Yang B, Barloi a, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. *Dent. Mater* 2010;26(no. 1):44–50.
- Aboushelib MN. Evaluation of zirconia/resin bond strength and interface quality using a new technique. *J. Adhes. Dent Jun.* 2011;13(no. 3):255–60.
- Thomas D. An in vitro evaluation of the effect of zirconia surface treatment on shear bond strength to a resin cement. *Ann. Essences Dent* 2013;5(no. 1):1–5.
- Komine F, Fushiki R, Koizuka M, Taguchi K, Kamio S, Matsumura H. Effect of surface treatment on bond strength between an indirect composite material and a zirconia framework. *J. Oral Sci* 2012;54(no. 1):39–46.
- Yang B, Ludwig K, Adelung R, Kern M. Micro-tensile bond strength of three luting resins to human regional dentin. *Dent. Mater* 2006;22(no. 1):45–56.
- De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. *Dent. Mater* 2004;20(no. 10):963–71.
- Chieffi N, Chersoni S, Papacchini F, Vano M, Goracci C, Davidson CL, et al. The effect of application sustained seating pressure on adhesive luting procedure. *Dent. Mater* 2007;23(no. 2):159–64.
- Sarmiento HR, Campos F, Sousa RS, Machado JPB, Souza ROA, Bottino MA, et al. Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia. *ActaOdontol. Scand* 2014;72(no. 5):346–53.
- El-Badrawy W, Hafez RM, El Naga AIA, Ahmed DR. Nanoleakage for self-adhesive resin cements used in bonding CAD/CAD ceramic material to dentin. *Eur. J. Dent Jul.* 2011;5(no. 3):281–90.
- Valandro LF, Leite FPP, Scotti R, Bottino MA, Niesser MP. Effect of ceramic surface treatment on the microtensile bond strength between a resin cement and an alumina-based ceramic. *J. Adhes. Dent* 2004;6(no. 4):327–32.
- Casucci A, Monticelli F, Goracci C, Mazzitelli C, Cantoro A, Papacchini F, et al. Effect of surface pre-treatments on the zirconia ceramic-resin cement microtensile bond strength. *Dent. Mater* 2011;27(no. 10):1024–30.
- Anand S, Ebenezer AV, Anand N, Rajkumar K, Mahalaxmi S, Srinivasan N. Microshear bond strength evaluation of surface pretreated zirconia ceramics bonded to dentin. *Eur J Dent* 2015;9(no. 2):224–7.
- de O. LULA EC, LEITE THM, ALVES CMC, SANTANA IL, ALMEIDA AML, COSTA JF. Parameters that influence microtensile bond testing of adhesive systems. *RGO - Rev. GaúchaOdontol* 2014;62(no. 1):65–70.
- Shihab SK, Khan Z a, Mohammad A, Siddiquee AN. F turning of hard steels used in Bearing and automotive applications. *Prod. Manuf. Res. Open Access J* 2014;2(no. 1):24–49.
- Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. *Quintessence Int. Berl* 2007;38(no. 9):745–53.
- Youm SH, Jung KH, Son S, Kwon YH, Park J-K. Effect of dentin pretreatment and curing mode on the microtensile bond strength of self-adhesive resin cements. *J. Adv. Prosthodont* 2015;7(no. 4):317–22.
- RelyX U2000 adhesive resin technical data Sheet. 3M ESPE 2011.
- Manu S, Usha K, Jyothi S, Krishnan VK, Pal SN. Effect of silanation of filler particles on the physical properties of a dental composite, vol. 5; 1998. p. 28–32. no. 1.
- Gasgoos SS, Sa'id RJ. The effect of thermocycling on shear bond strength of two types of self-etch primers. *Al-Rafidain Dent J* 2009;9(no. 2):246–53.
- Sakaguchi RL, Powers JM. *Craig's restorative dental materials*. thirteenth ed. Elsevier; 2012.
- Aboushelib MN, Feilzer AJ, Kleverlaan CJ. Bonding to zirconia using a new surface treatment. *J. Prosthodont* 2010;19(no. 5):340–6.
- Zhao J, Wang X, Si W, Shen Z. Effect of resin cement selection on the micro-tensile bond strength of adhesively veneered 3Y-TZP. *ActaOdontol. Scand* 2013;71(no. 5):1105–11.