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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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A B S T R A C T

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 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 7dayes followed by thermocycling; subgroups 1, 2 and 3 respectively. After aging, the assemblies were sectioned into beams approximately 1 mm^2 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.

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

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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_2 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_2O_3), 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_2O_3). 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_2O_3. 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 × 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 μm Al_2O_3 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_2O_3 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 SO5050).

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 automixing tips and light cured by LED light-curing unit (COXO BD-686-Ib, China) at intensity of 1600 mW/cm^2. 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, 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^2. Another horizontal section parallel to the adhesive cement interface, at the junction between zirconia and lead base, was done to separate the beams