THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Durability of resin bonding to translucent zirconia materials: An in vitro study

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ABSTRACT

Statement of problem. How the yttria content (3Y-PSZ, 4Y-PSZ and 5Y-PSZ) affects the tensile bond strength of luting resin to translucent zirconia ceramics is unclear.

Purpose. The purpose of this in vitro study was to evaluate whether the bond strength of resin to translucent zirconia ceramics and its durability are influenced by the yttria content (3Y-PSZ, 4Y-PSZ and 5Y-PSZ).

Material and methods. Sixty-four disk-shaped specimens of zirconia ceramics were randomly allocated to 4 groups (n=16) with respect to the yttria concentration: GHT: 3 mol% yttria-partially stabilized zirconia 3Y-PSZ; GHTML: 3 mol% yttria-partially stabilized multilayered zirconia 3Y-PSZ; GSTML: 4 mol% yttria-partially stabilized zirconia 4Y-PSZ; and GUTML: 5 mol% yttria-partially stabilized zirconia 5Y-PSZ. For bonding, acrylic resin tubes were filled with composite resin and bonded to the zirconia disks using adhesive luting resin by following manufacturer's instructions. Half of each main group (n=8) was stored in water for 3 days at 37 °C. The other half was stored in water for 150 days with 37 500 thermocycles (5 to 55 °C). The tensile bond strength test was determined with a universal testing machine at a crosshead speed of 2 mm/minute using a chain loop for alignment.

Results. The tensile bond strength was not significantly affected by the yttria concentration of different zirconia materials (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ, P=.17). The mean ±standard deviation failure loads for groups ranged from 24.1 ±2.6 MPa for the ultratranslucent multilayered zirconia (with thermocycling) to 52.7 ±5.4 MPa for the high translucent multilayered zirconia (without thermocycling). However, artificial aging adversely affected the tensile bond strength values for all tested zirconia systems (P<.001). Scanning electron microscope evaluation showed that cohesive fracture was the predominant failure mode, while few specimens exhibited adhesive failure at the interface.

Conclusions. The yttria concentration (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) had no significant effect on the bond strength of different zirconia ceramics (P=.17), whereas the artificial aging procedures led to a statistically significant decrease in tensile bond strength (P<.001). (J Prosthet Dent 2025;133:1583.e1-e8)

Supported by the Deanship of Postgraduate Studies and Scientific Research, Dar Al Uloom University, Riyadh, Saudi Arabia; materials used in study provided in part by Kuraray Noritake Dental Inc. (Osaka, Japan).
The authors report no conflicts of interests.

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Clinical Implications

Artificial aging with thermocycling significantly affected the tensile bond strength of luting resin to translucent zirconia ceramics; however, yttria content (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) had no statistically significant effect.

Restoration fabrication has become more automated, efficient, and precise with the introduction of digital milling combined with innovative rapid-sintering technologies.[1–4] Advances in computer-aided design and computer-aided manufacturing (CAD-CAM) technology, along with excellent mechanical and biocompatibility properties, have made zirconia ceramics popular for dental applications.[5] Monolithic zirconia eliminates mechanical problems and the necessity for extensive tooth preparation, resulting in a prosthetic restoration that preserves as much of the natural tooth structure as possible.[6]

Monolithic zirconia restorations have a number of advantages, including improved flexural strength, the ability to prepare teeth conservatively, reduced antagonist wear, improved esthetics, fewer clinical sessions, reduced laboratory time, and lower risk of chipping.[7–9] Therefore, zirconia ceramic is nowadays one of the most utilized indirect restorative materials in the field of prosthodontics.[10]

Depending on the temperature, zirconia can be available in 3 crystalized forms: monoclinic (room temperature to 1170 °C), tetragonal (1170 °C to 2370 °C), or cubic (2370 °C to melting point).[11] Y-TZP comes in a variety of forms depending on the additives and dopants used, as well as the sintering profiles and heat treatments used.[12] Monolithic zirconia restorations have become popular because of advancements in the manufacturing procedures with improved optical qualities and excellent mechanical features. When compared with veneered framework, monolithic zirconia has the advantage of requiring less tooth structure removal, a shorter workflow, and hence lower costs, as well as eliminating the complications associated with veneering ceramic.[13,14] Recently, and with great clinical success, zirconia has been used for a wide range of clinical applications, from single crowns to complete mouth rehabilitations.[15–19]

The chemical inertness and the polycrystalline structure of zirconia ceramic, as well as the absence of silica phase, renders it resistant to etching with acids and, consequently, prevents it from establishing satisfactory bond strength to adhesive luting resin.[20] Airborne-particle abrasion (APA) with alumina particles has proven to be an effective approach and is nowadays a widely accepted method of preparing the bonding surfaces of zirconia ceramics.[21] Adequate cleaning of the bonding surface, the use of pure Al_2O_3 for APA to create micromechanical retention, and the use of phosphate monomers such as 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) found in primers or luting composites to create chemical adhesion can all result in a strong, long-lasting bond to dental zirconia.[21–25] 10-MDP is now considered the standard monomer for promoting adhesion to zirconia substrates.[26] Earlier investigations demonstrated that luting resins and primers containing 10-MPD monomer produced reliable and durable chemical bonds to airborne-particle abraded zirconia.[27] Kern and Wegner[28] used a variety of bonding systems to evaluate the bonding of conventional zirconia ceramic, reporting that luting agents which contain a phosphate monomer group (10-MDP) had the highest bond strength to the zirconia ceramic after airborne-particle abrasion of their intaglio surface with alumina particles.

Recently, attempts have been made to improve the translucence of zirconia so that it can be used as a monolithic restoration, including increasing the content of yttrium oxide to produce 4Y-PSZ and 5Y-PSZ zirconia, in which a cubic phase is added to the metastable tetragonal phase to increase translucency. However, the reduction in the tetragonal phase results in reduced mechanical properties.[29,30] Therefore, the aim of this in vitro study was to compare the bond strength of zirconia ceramics based on its yttria mol% concentration (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) after different aging procedures. The null hypotheses were that bonding to zirconia ceramics would not be affected by the yttrium oxide content or by artificial aging.

MATERIAL AND METHODS

Four types of zirconia ceramics based on yttria concentration were included in the study (Table 1). A total of 64 disk-shaped specimens of different concentration yttria-partially stabilized zirconia ceramics (Katana High Translucent; Kuraray Noritake Dental Inc) were allocated to 4 groups (N=16) according to the yttria concentration. Specimens of each group were fabricated by milling the respective zirconia blank into cylinders (Fig. 1). Then, the cylinders were sectioned at the level of the uppermost layer with a rotary saw (Isomet 1000 precision saw; Buehler GmbH) under water cooling to obtain disk-shaped specimens. The specimens were sintered according to the manufacturers' instructions to Ø8.0×3.4-mm specimens and polished in an automated grinder polisher (EcoMet/AutoMet 250 Pro; Buehler GmbH) using 600-grit rotating silicon carbide paper (SiC grinding paper; Buehler GmbH). Subsequently, all zirconia specimens were subjected to airborne-particle

Table 1. Properties of tested zirconia materials based on information from manufacturer (Kuraray Noritake Dental Inc)

Zirconia Material	Wt.-% Cubic Phase	Yttria Concentration (mol%)	Flexural Strength (MPa)	Translucency%[a]	Elastic Modulus (GPa)
Katana HT	<50	3 mol%	1125	45	214
Katana HTML	<50	3 mol%	1150	45	214
Katana STML	~65	4 mol%	750	49	217
Katana UTML	~75	5 mol%	550	51	217

Katana HT, 3 mol% yttria-partially stabilized zirconia 3Y-PSZ; Katana HTML, 3 mol% yttria-partially stabilized multi-layered zirconia 3Y-PSZ; Katana STML, 4 mol% yttria-partially stabilized zirconia 4Y-PSZ; Katana UTML, 5 mol% yttria-partially stabilized zirconia 5Y-PSZ.
[a] Translucency defined as light transmission: 100% (transparent) and 0% (opaque).

abrasion (APA) with 50-μm alumina at 0.1 MPa for 15 seconds at 10 mm. Prior to APA, the bonding surfaces were colored with a black permanent marker.[23] Then, APA was applied until the color was completely erased. The APA was performed in diverse horizontal and vertical directions to ensure that the bonding surface was entirely abraded. After that, all specimens were blasted with oil-free compressed air and ultrasonically cleaned in a 99% isopropanol bath for 3 minutes. After retrieving the specimens, alcohol residue was removed by applying a gentle oil- and water-free compressed air stream.

Acrylic resin tubes with an inner diameter of 3.2 mm[31] were filled with an autopolymerizing foundation composite resin (Clearfil Core New Bond; Kuraray Noritake Dental Inc) and left undisturbed to polymerize for 10 minutes before bonding them to the zirconia specimens. The bonding surfaces of zirconia specimens were primed (Clearfill Ceramic Primer Plus; Kuraray Noritake Dental Inc), allowed to dry for 20 seconds, and then gently dried with oil- and water-free airflow. Subsequently, the filled tubes were bonded with a dual-polymerizing adhesive luting resin (Panavia V5; Kuraray Noritake Dental Inc) to the primed zirconia specimens. To ensure that the long axis of the filled tube was perpendicular to the bonding surface of the specimen, an alignment apparatus which applied a

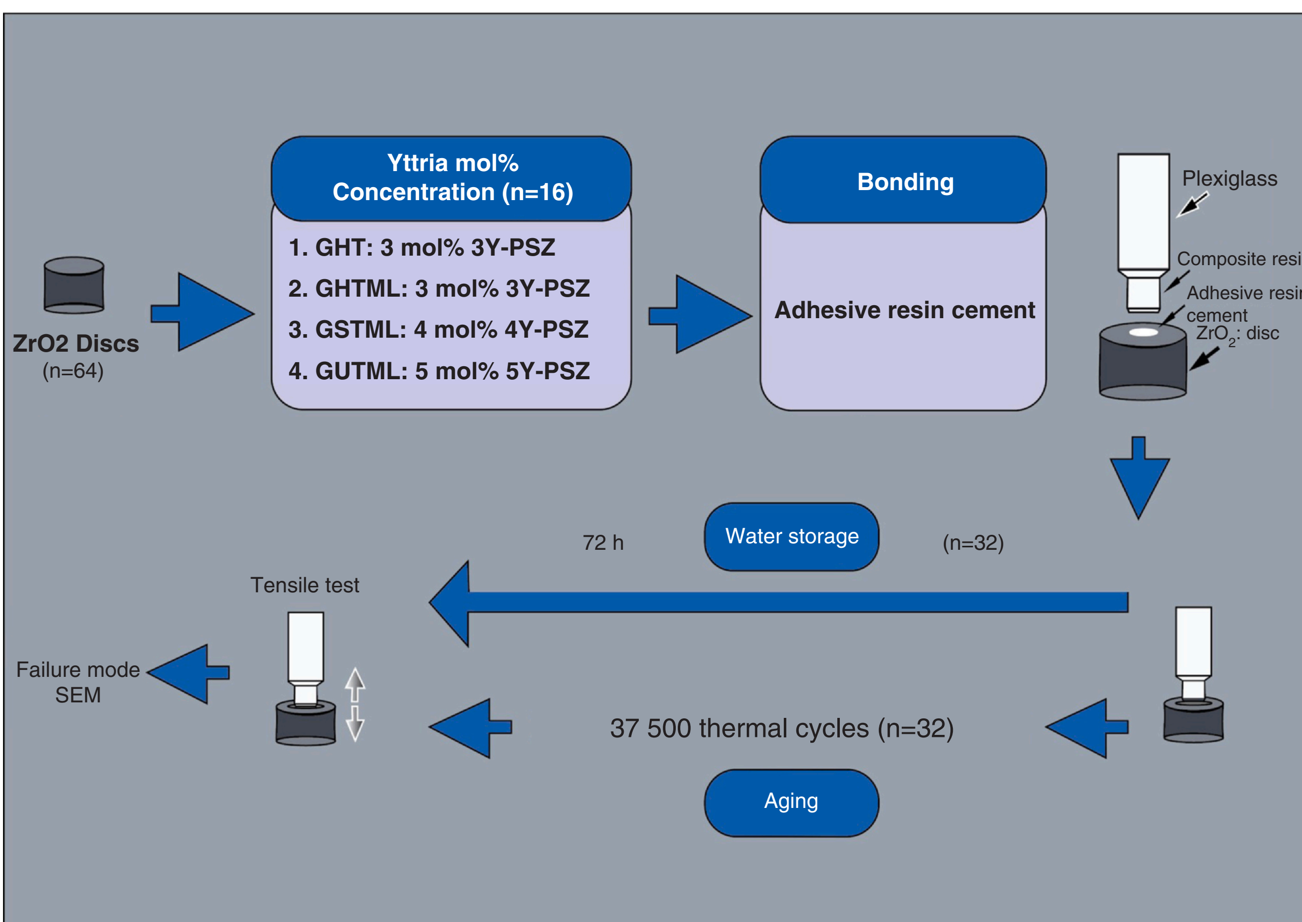

Figure 1. Flow chart of experimental procedures.

constant load of 7.4 N was used to hold the tubes in the vertical position.[22,24] Excess luting resin around the tube margins was cleaned by means of a sponge pellet, and an air-blocking gel (Oxyguard II; Kuraray Noritake Dental Inc) was injected around the bonding interface margins to prevent the formation of an oxygen-inhibited layer. Preliminary light polymerization was conducted from 2 opposite sides of the specimen for 20 seconds each (Radii-cal; SDI). Then, light polymerization was completed with a dental laboratory light-polymerizing unit (Dentacolor XS; Kulzer GmbH) for 90 seconds

Each bonded main group of zirconia specimens (n=16), representing one of the investigated yttria-partially stabilized zirconia (PSZ) ceramic materials, was randomly (www.randomizer.org) divided into 2 subgroups (n=8). The first subgroup was stored for 3 days (short-term storage) in 37 °C distilled water without undergoing any thermocycling to evaluate the initial tensile bond strength (TBS). The second subgroup was stored for 150 days (long-term storage) in 37 °C distilled water and underwent 37 500 thermocycling using temperatures of 5 °C and 55 °C and a dwell time of 30 seconds to examine the impact of hydrolytic aging on the TBS durability of the bonded specimens. Finally, the TBS of the luting resin to zirconia specimens was tested using a universal testing machine (Zwick Z010; ZwickRoell Group) at a crosshead speed of 2 mm/minutes using chain loop alignment to ensure a moment-free axial application of the tensile force.[22] The TBS value in MPa was calculated by recording the tensile force at failure in N and dividing it by the bonding surface area (A) in mm^2 ($A=\pi d^2/4$, d=3.2 mm).

The data obtained were statistically analyzed using a software program (IBM SPSS Statistics; v20.0 for Windows; IBM Corp). The Shapiro–Wilk test revealed a normal distribution of the data ($P>.05$). Therefore, a 2-way analysis of variance (ANOVA) was applied, followed by multiple pairwise comparisons of groups and subgroups with the Tukey HSD post hoc test ($\alpha=.05$).

After debonding of the zirconia ceramic specimens, the interface surface of each specimen was inspected via optical microscopy (Wild Makroskop M 420) at ×30 magnification to determine the ratio of adhesive to cohesive failure of each individual zirconia specimen. Adhesive failure was defined as a failure in the interface between the ceramic surface and the bonding material without visible resin remnants on the ceramic surface. Cohesive failure was defined as a failure within the bonding material with visible resin remnants on the ceramic surface. Debonded areas of all tested specimens were examined, and each failure mode was calculated and expressed as a percentage of the total bonding surface area for each tested subgroup. Thereafter, a representative specimen from each investigated subgroup was sputtered with a thin gold-alloy conductive layer of approximately 10 nm in thickness, and the representative specimens were examined by means of a scanning electron microscope (SEM) (XL 30 CP; Philips): images were captured at ×65 magnification with an acceleration voltage of 15 kV and a working distance of 10 mm.

RESULTS

The mean ±standard deviation TBS values after 3 days of water storage ranged from a minimum of 43.8 ±9.6 MPa for subgroup GHT to a maximum of 52.7 ±5.4 MPa for subgroup GHTML (Fig. 2). The 2-way ANOVA test did not reveal a statistically significant interaction ($P=.069$) between zirconia type and aging. No statistically significant differences were found among the initial mean TBS values of all investigated subgroups ($P>.05$). All bonded zirconia specimens survived the artificial aging for 150 days. The mean ±standard deviation TBS values after 150 days of water storage and thermocycling ranged from a minimum of 24.1 ±2.6 MPa for group GUTML to a maximum of 32.6 ±7.8 MPa for group GHTML. No statistically significant differences were found between the mean TBS values of all test subgroups after artificial aging ($P>.05$). However, artificial aging caused statistically significant deterioration ($P<.05$) in TBS values. All studied subgroups exhibited significantly lower TBS values after artificial aging in comparison with TBS values obtained after 3 days. The failure-mode distribution is illustrated in Figure 3 for each subgroup in percentages.

SEM evaluation showed that cohesive fracture was the predominant failure mode, while a few specimens exhibited adhesive failure at the interface (Fig. 4). The adhesive failure percentage for all nonaged or aged studied subgroups was less than 20%, except for the aged GUTML subgroup, which demonstrated up to 32% of adhesive failure.

DISCUSSION

Since translucent monolithic zirconia is a relatively new material, few studies have evaluated its resin bond strength and durability to translucent zirconia ceramics. Therefore, a study was needed to evaluate whether the resin bond strength and its durability to translucent zirconia ceramics is influenced by the yttria content (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ). Since adhesive interfaces and composite resins are influenced by chemical, mechanical, and thermal changes in the oral cavity, laboratory TBS tests using artificial aging were applied.[25,28] TBS testing was used because of its uniform stress distribution and ability to prevent inaccuracies

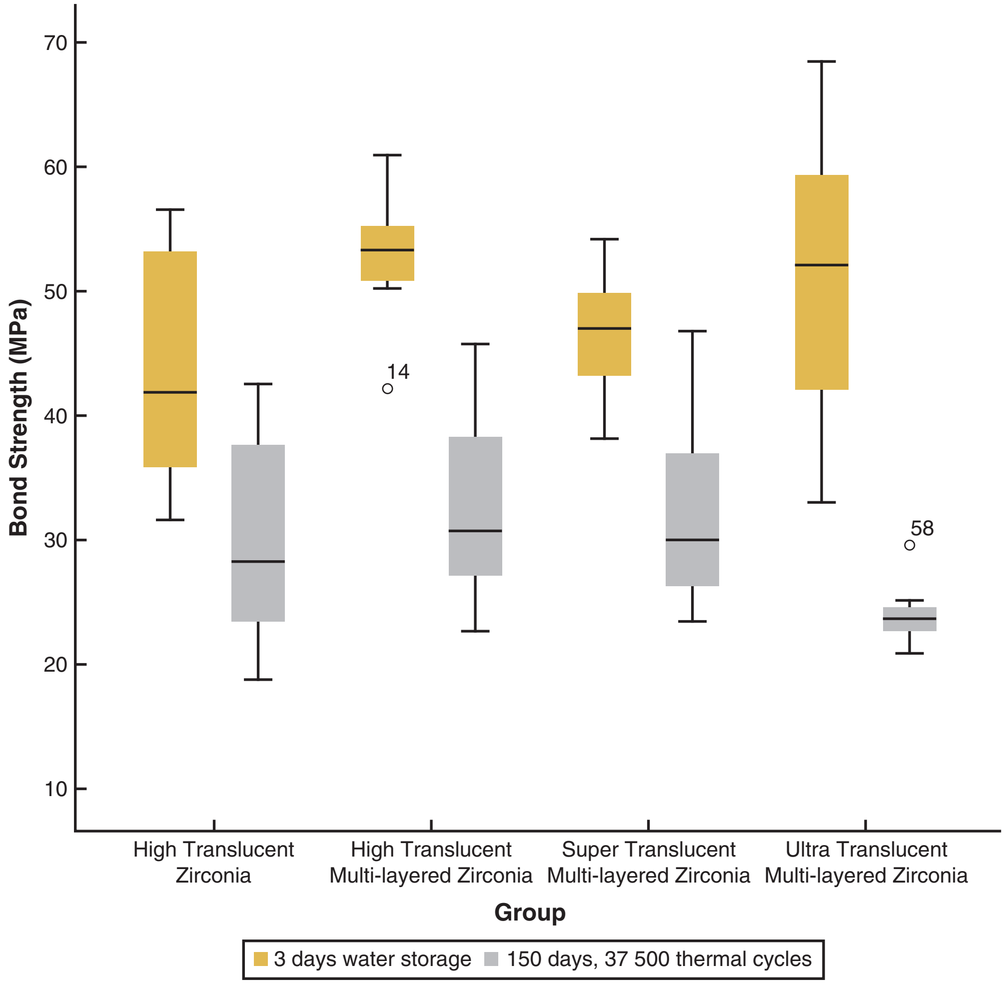

Figure 2. Mean bond strength values (MPa) and standard deviations for luting resin to different zirconia ceramics with and without aging.

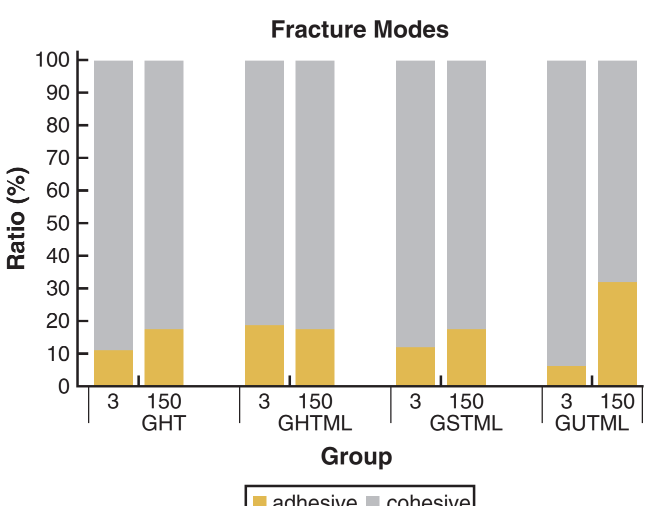

Figure 3. Distribution of failure modes in % for all test subgroups after tensile bond strength testing.

resulting from complex forces occurring in shear bond strength tests.[32] In addition, to minimize procedure sensitivity and eliminate the micromovements and vibrations generated during specimen preparation, a microtensile bond test was not selected.[33] A weak point of the TBS test, the alignment of the composite resin tubes on the ceramic specimens, was overcome using a paralleling bonding device that ensured vertical positioning over the ceramic bonding surface.[34]

Thermocycling has been used to simulate the in vivo aging of restorative materials by subjecting them to repeated cyclic exposure to hot- and cold-water baths in order to imitate the thermal changes occurring in the oral cavity.[35] According to the International Organization for Standardization (ISO) standard,[36] thermocycling should be conducted between 5 °C and 55 °C, with a minimum of 500 cycles and a dwell time of more than 20 seconds. In the current study, the selected thermocycling protocol was adopted by several earlier

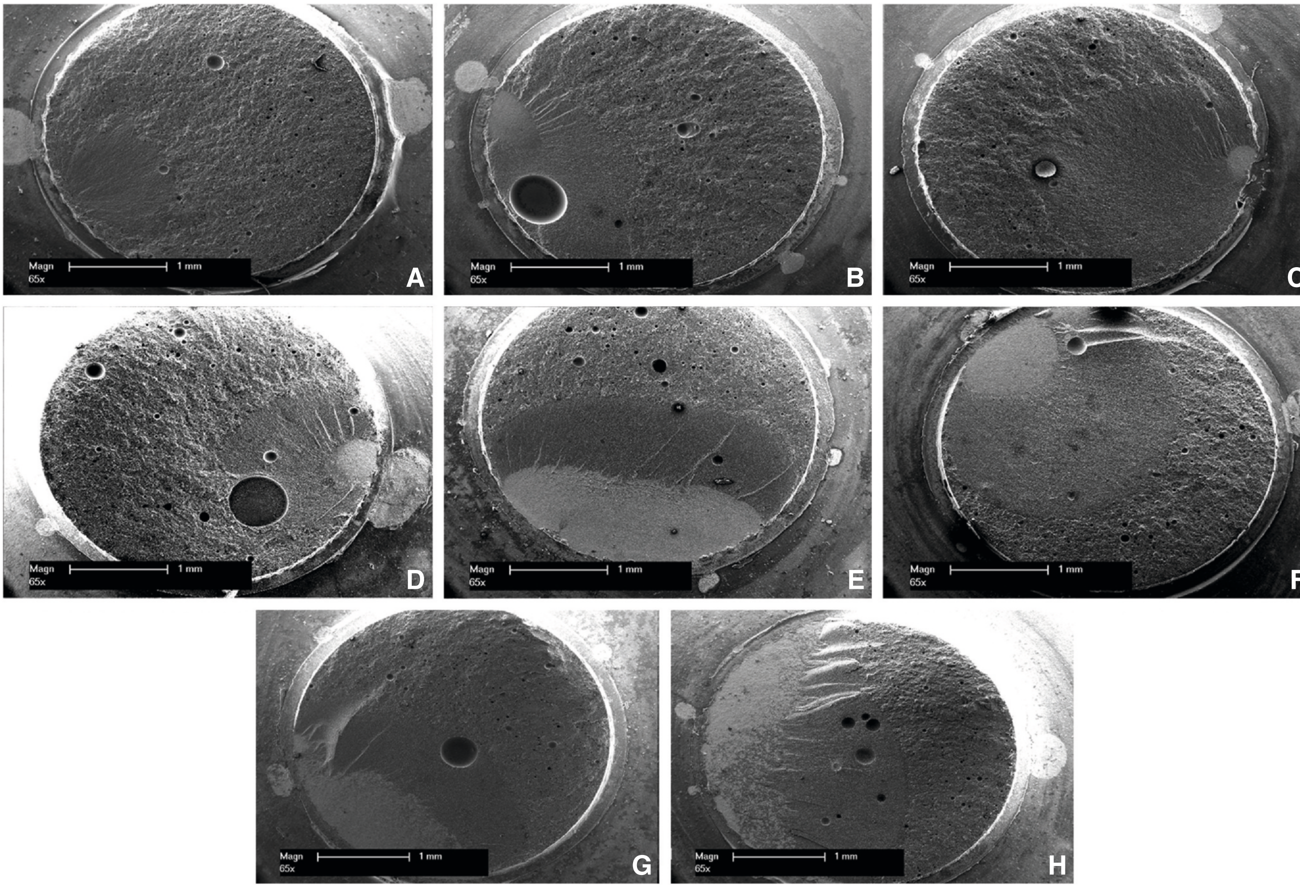

Figure 4. Scanning electron microscope images of typical failure modes after tensile bond strength testing (original magnification ×65). A-D, Nonaged specimens of subgroups, GHT, GHTML, GSTML, GUTML, respectively. E-H, Aged specimens of subgroups, GHT, GHTML, GSTML, GUTML, respectively. GHT, Katana High Translucent; GHTML, Katana High Translucent Multi Layered PLUS; GSTML, Katana Super Translucent Multi Layered; GUTML, Katana Ultra Translucent Multi Layered.

studies before testing the TBS of luting resin to zirconia.[22,24,25] This protocol was applied as it used the largest number of cycles (37 500 cycles) reported in the literature, thereby simulating the conditions of the oral cavity and ensuring the complete water saturation of the bonded specimens.[37,38]

APA plays an important role in improving the bonding strength of resins to the surfaces of zirconia ceramics, as shown in previous studies.[21,23,25] Therefore, all zirconia surfaces were abraded with 50-μm alumina at 0.1 MPa for 15 seconds at a distance of 10 mm.[39] The low pressure and the small particle size aimed to minimize surface defects and to create an effective micromechanical retentive surface.[40]

The success of zirconia-based ceramic restorations depends largely on the proper pretreatment of the bonding surfaces and the luting protocols.[41] Therefore, in the current study, MDP-containing primer (Clearfill Ceramic Primer Plus) with dual-polymerized luting resin (Panavia V5) were used to establish a strong bond between the luting resin and zirconia ceramics.[42]

The null hypothesis that bonding to zirconia would not be affected by the yttrium oxide content was not rejected. TBS was not significantly affected by the yttria concentration (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) of the different tested zirconia materials ($P>.05$). The similarity may be because the investigated materials had relatively similar composition with slight modifications in the grain size and quantity of aluminum oxide. In addition, the bond strength at the interface between zirconia and the adhesive material may be more influenced by the type of surface treatment and the chemistry of the bonding agent, as revealed by several studies,[22,24,27,40,41,43] than by the yttria content. All tested zirconia specimens (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) were treated similarly to enhance surface roughness and increase the effective bonding area.

The null hypothesis that the bond strength to zirconia would not be affected by the aging process was rejected. The aging process influenced the TBS of the evaluated specimens ($P<.05$), regardless of the type of translucent zirconia material used. This result was consistent with the findings of studies that tested TBS both directly after bonding and after thermocycling with long-term water storage.[24,44,45] The significant reduction in TBS to zirconia subsequent to long-term water storage and thermocycling might be associated with the plasticizing effect of water on the matrix of the luting resin,[45] which, in turn, leads to hydrolytic degradation within the resin.[46] It has been reported that artificial aging might not significantly affect the bond strength durability to zirconia.[47] This controversy could be related to the differences in the various types of zirconia ceramics, the mechanical and chemical pretreatment methods, the composition of the primers, and the composite resin luting agents used.[48,49]

The failure modes of the tested groups were investigated using optical microscopy and SEM (Fig. 3). All groups showed a higher rate of cohesive failures in the luting resin, which was consistent with the obtained bond strength values. Higher TBS values were associated with a higher ratio of cohesive failure, possibly because of the hydrolytic stability of MDP; this is linked to the presence of long carbonyl chains which promote the formation of a water-resistant chemical bond with zirconia.[50,51]

Limitations of the study included the in vitro design, the relatively low number of specimens in each group, and that no mechanical fatigue was applied during the artificial aging process. Clinical investigations with the long-term assessment of translucent zirconia ceramics with different yttria content (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) regarding the durability of the bond strength are still required.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The TBS of luting resin to zirconia ceramics was not influenced by the yttria content (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ).
2. Aging with the thermocycling process significantly reduced the TBS of luting resin to different zirconia ceramics.

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https://doi.org/10.1016/j.prosdent.2025.03.017