Original Article

ACDJ Volume 4, Issue1, January, 2025

Bonding of Resin Cement to Monolithic Zirconia Ceramic after Different Artificial Aging

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Abstract:

Objectives: This invitro research investigated shear bond strength (SBS) of resin cement to zirconia ceramics after different artificial aging.

Methods: 60 zirconia discs were CAD-CAM fabricated. Composite discs were bonded to conditioned zirconia discs using adhesive resin cement. Specimens were divided into 6 test groups according to artificial aging condition: 30000 cycles of cyclic loading test group (1), 5 months of water storage +30000 cycles of cyclic loading test group (2), 5 months of Water storage +5000 cycle of thermocycling test group (3), 5000 cycle of Thermocycling test group (4), 5 months of Water storage test group (5) and 5 months of water storage +5000 cycles of thermocycling+ 30000 cycles of cyclic loading test group (6).A universal testing machine was used for SBStest to all specimens**.**One-way ANOVA and post-hoc Tukey tests were used for statistical analysis.

Results: Group 1 recorded the highestshear bond strength showed means ± SD record (9.35 ± 2.25) followed by group 4 which means ± SD record (8.53 ±4.92) followed by group 2 which means ± SD record (6.92 ± 1.04) followed by group 3 and group 5 respectively which means ± SD records (5.97 ± 2.57) and (4.72 ± 1.30) while the lowest shear bond strength was for group 6 with means ± SD record (3.75±1.19).

Conclusions: The influence of water storage in combination with thermocycling and cyclic loading on SBS is higher than the effect of thermocycling alone and cyclic loading alone.

Keywords: Ceramics, Resin Cement, Shear Bond Strength.

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Introduction:

Adhesive cementation of zirconia crowns is preferred than conventional methods [1,2].The main disadvantage of zirconia-based restorations is poor bonding with resin cements[3].Conventional adhesive cementation processes, including acid etching followed by salinization of the ceramic surface, are ineffective in generating a strong connection between zirconia ceramics and resin cements because zirconia has a glass-free structure that cannot be erased by hydrofluoric acid[4]. The advancement of micromechanical interlocking is essential for establishing a bond and enduring adhesion between resin cement and zirconia [5]. Following the cementation of zirconia-based restorations with resin cement, a sandwich-like configuration with two interfaces is established: a zirconia/resin interface and a resin/dentin contact[6]. Both interfaces are significant, and several assessments can be employed to ascertain the bond strength at the zirconia/resin contact, including micro tensile, tensile, micro shear, and shear bond strength tests[2,7].

Restorations in the oral cavity are susceptible to diverse thermal and mechanical stresses resulting from intraoral masticatory forces[8]. Various artificial aging methods, including prolonged water immersion and thermal cycling, can replicate intraoral conditions[9]. They play a crucial role in establishing how long the resin cement-zirconia-based ceramic bond will last [10].

A range of artificial aging approaches, including as thermocycling, cyclic fatigue, and long-term water storage, can simulate intraoral conditions [11].

Artificial aging can mimic conditions in oral cavity, which is crucial for assessing the integrity of the resin cement-Y-TZP ceramic bond [12].In vitro research has made use of laboratory-induced thermal ageing techniques to evaluate the bond interface's longevity. These techniques are crucial for ascertaining the longevity of the bond formed between zirconia-based ceramics and resin cements [13]. Bond failure may ultimately occur from strains impacting the bond interface brought on by thermal fatigue brought on by thermal expansion and contraction [14].By exposing the tested specimens to thermal cycling, which replicates the temperature characteristics of the oral environment, laboratory-induced thermal ageing has been established as a means of testing the endurance of the bond interface in in-vitro research[15].

With respect to variations in the coefficient of thermal expansion of materials, thermocycling has been widely employed to mimic thermal stresses that are frequently encountered in the oral environment [14]. That being said, thermocycling by itself does not accurately replicate oral circumstances[16]. Incorporating fatigue cyclic loading might potentially enhance the evaluation of adhesive systems' clinical performance[17,18].

The null hypothesis of this study was that the different artificial aging regimes will have the same effect on shear bond strength.

2. MATERIALS AND METHODS:

2.1. Ethical approval

Prior to the project, the Mansoura University Faculty of Dentistry's institutional ethical committee accepted the study protocol with code number (A03023Fp).

2.2. Specimen preparation

Sixty discs (n = 60) of monolithic Y-TZP ceramic were fabricated utilizing CAD/CAM technology as follow:
A design was made in Exocad to get the dimension of disc (8 mm diameter × 3 mm thickness) The zirconia discs were dry milled from zirconia blanks (IPS e.max ZirCAD) by using DWX-52D (DGSHAPE) with 5 axes dry milling machine in Mansoura University lab.

2.3.Composite resin discs preparation

A metal ring with a central Teflon hole (4 mm in diameter × 3 mm in thickness) was used to fabricate sixty composite resin discs.
The composite resin discs were fabricated through the central hole which was filled by layering technique in two layers, each layer was 2mm of Ruby-Fill resin composite (universal nano composite), which was cured with a light curing unit (I led, woodpecker, China) for 20 seconds for each layer at intensity of 1000mW/cm^2 with 20mm distance.

Following milling, the zirconia discs were sintered in a high temperature furnace (Nabertherm, Germany) according to manufacturer specifications. Conventional sintering cycle of IPS e.max ZirCAD: discs were sintered according to the recommended sintering parameters of IPS e.max ZirCAD was 1500ºC for 2 hours, rate of temperature increased 10 C/min, holding time: 2.5 hours, and cooling rate: -10 C/min.

2.4. Surface treatment of zirconia specimens

Renfert Basic sandblaster (Renfert, Germany)was used forSandblasting the bonding surfaces of zirconia discs by using 110 µm Al2O3 particles under a pressure of .2 MPa which might equal (2 bars) for 15 seconds with a 10 mm distance perpendicular to the bonding surfaces of discs[19,20].

2.5. Bonding of specimens

A layer of (Monobond N,Ivoclar Vivadent, Schaan, Liechtenstein) universal primer was applied to bonding surface of each disc firstly with micro brush and left for 60 seconds then it was displaced with oil-free air stream for 5 seconds according to manufacturing instructions.

Bonding composite resin discs and previously treated zirconia discs was done using adhesive resin cement (Multilink N,Ivoclar Vivadent, Schaan, Liechtenstein) as follows:

An auto-mix tip was used to apply adhesive resin cement to the bonding surface of the zirconia discs.

zirconia discs were topped with composite resin discs in a specifically constructed device to apply a steady load of 5 Kg to the composite/zirconia disc assembly during cementation.

Excess resin cement was cleaned by a micro brush, then utilized a light curing unit (I led, Woodpecker, China) in four directions for 20 seconds at intensity of 1000mW/cm^2 with 20mm distance.

2.6.Artificial aging

One hour after cementation, specimens were divided into six test groups according to artificial aging regime:

Group 1:were de-bonded after cyclic loading (30,000 cycles) (equal 1.5 month clinically approximately)

Group 2:were de-bonded after 5 months of water storage (long term storage) + cyclic loading (30,000 cycles) (equal 1,5 month clinically approximately)

Group 3:were de-bonded after 5 months of water storage (long term storage) + thermal cycling (5000 cycles) (equal 6 months clinically approximately).

Group 4:were de-bonded after thermal cycling (5000 cycles) (equal 6 months clinically approximately).

Group 5:were de-bonded after 5 months of water storage (long term storage).

Group 6:were de-bonded after 5 months of water storage + thermal cycling (5000 cycles) + cyclic loading (30,000 cycles)

2.7. Recording bond strength

A universal testing machine (Instron 3345, USA) was used to apply compressive load at the zirconia/ substrate interface at a crosshead speed 0.5mm per minute. The load to failure was recorded in Newton. To convert the SBS to MPa using the equation: failure load / surface area.

2.8. Mode of failure analysis

the mode of failure was detected by Stereo microscope. The recorded patterns of failure were adhesive or mixed failure with no occurrences of cohesive failure patterns. Electron microscope (SEM) imaging was used to make further investigation.

3.RESULTS

Statistical analysis of variance was conducted using version 26.0 of the IBM Social program for Statistical Sciences (SPSS) software. IBM Corporation, New York, United States.

3.1. Shear bond strength results:

Group 1 recorded the highest shear bond strength showed means ± SD record (9.35 ± 2.25) followed by group 4 which means ± SD record (8.53 ±4.92) followed by group 2 which means ± SD record (6.92 ± 1.04) followed by group 3 and group 5 respectively which means ± SD records (5.97 ± 2.57) and (4.72 ± 1.30) while the lowest shear bond strength was for group 6 with means ± SD record (3.75±1.19).

The results ofpost hoc test indicated that group 1 was significantly different from group 5 (p=0.009) and group 6 (p=0.002), as well as group 4 being significantly different from group 5 (p=0.032) and group 6 (p=0.007).

So, from tests cycling load test group followed by thermocycling test group had the lowest effect on SBS which showed in their (mean) records.But according to spread of data which could consider them equally, in another word the cyclic loading and thermocycling effect were on SBS nearly equal in SBS, and the impact of combination between cyclic loading and thermocycling with long term water storage was more risky than other groups on SBS with lowest mean record.

3.2. Failure pattern analysis

After applying artificial aging techniques and subsequent de-bonding for testing, the failure patterns of all de-bonded specimens primarily displayed adhesive failure patterns (41 specimens), then by mixed failure patterns (12 specimens), with no occurrences of cohesive failure patterns noted.

3.3. Scan electron microscope (SEM).

Scanning electron microscopy (SEM) was used to analyze representative samples of the adhesive and mixed failure patterns shown in Figure (1,2). Zirconia surfaces included remnants of restorative materials such as resin cement or composite resin in specimens with a mixed failure pattern.

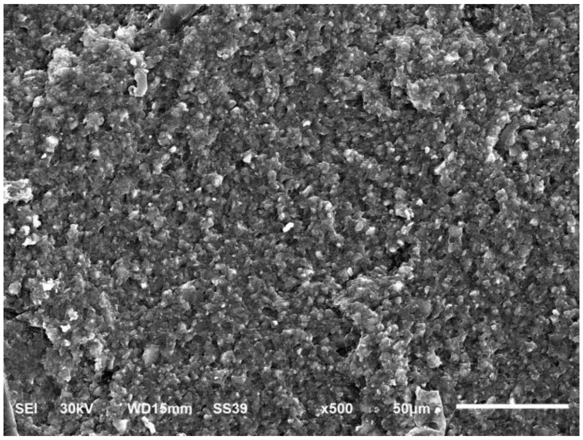

Figure 1: Showing adhesive failure at x500 of (water storage+ cyclic loading) group.

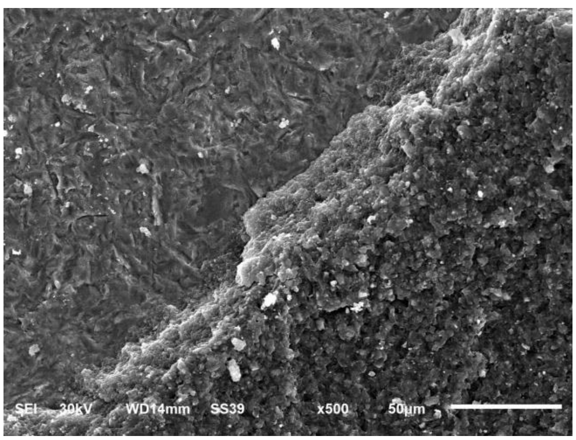

Figure 2: Showing mixed failure at x500 of (water storage + thermocycling +cyclic l

3. <u>DISCUSSION:</u>

The main goal of our study was to show the combination of water storage and thermocycling and cyclic loading as aging methods for evaluation bond durability of zirconia to resin cement. According to the present findings, the null hypothesis rejected partially due to the result of test groups which showed that some groups were nearly equal but other test groups were significant different.

In this study the combination of water storage, thermocycling and cyclic loading not mentioned before in other PubMed researches, just the combination between them mentioned in fracture resistance not SBS[21].**Shehri et al. 2017** [18] mentioned the combination of thermocycling with cyclic fatigue only which decreased SBS.

Instead of using dental tissues, this study used composite resin discs to attach zirconia specimens. Due to dentin's microstructure, which is heterogeneous, the homogeneity of the composite resin discs would eliminate the possibility of interpreting bond strength data incorrectly[22].

Y-TZP ceramic restorations were cemented using conventional cements. As opposed to conventional cements, resin cements have a number of benefits, such as enhanced retention, marginal flexibility, and fracture resistance for the restored tooth and the restoration itself [23]. This study utilized adhesive resin cement to bond the composite discs to zirconia ones. Multilink-N's formulation excludes acidic phosphate monomers; it consists of Di-methacrylate, HEMA, and silica fillers, which enhance its superior mechanical properties [21]. The manufacturer reports that two-fold curing increases flexural strength to around 110 MPa. The motivation for adopting Multilink® N resin cement is based on its outstanding mechanical qualities [24].

Masticatory forces cause thermal and cyclic stresses on restorations intraorally, which can alter the strength of bond between resin cement and zirconia-based restorations. This study showed long-term water storage, thermocycling, and cyclic loading, both alone and in combination, to simulate intraoral conditions and determine which element had the highest impact on shear bond strength [12].

Regarding cycles of thermocycling, **Gale and Darvell 1999** [17],have suggested that 10,000 cycles of thermocycling is roughly equivalent to one year clinically, so in the current study 5000 thermal cycles were used and during thermocycling, the mismatch in coefficient of thermal expansion between composite resin restorative material, resin cement, and zirconia ceramic could produce mechanical stresses that contribute to bond degradation while **Lu, Zhi-Cen et al (2023)** [25]stated that, Thermal aging by 10,000 thermocycles may be insufficient to interrupt the bond of composite cement to zirconia. So, the impact of thermocycling on bonding strength remains ambiguous that supporting with the findings of this study which showed minimal effect of thermocycling alone on SBS [26].

On the other hand **, Alrabeah et al(2023)**[12]informed that , the shear bond strength between the zirconia and the self-adhesive resin cements significantly decreased by thermocycling which was not in agreement with our study. This might be due to that, the influence of thermocycling on shear bond strength was affected by several factors such as type of resin cement adhesive or self-adhesive where was adhesive resin cementin this study that provided stronger and more durable bond strength than self-adhesive one [12].

Regarding cyclic loading**,** the fatigue after 1.2 million cycles may equivalent to 5 years of practical experience in dental restorations. [(21)] These strokes may compromise the restoration's interfacial bonds and the tooth surface, causing the restoration to fail [17,27].In the current investigation, cyclic loading was also employed to simulate chewing conditions (30000 cycles). Different investigations have reported varying mechanical loads, according to **Nemli et al2012**[28] Cyclic fatigues can cause tetragonal Y-TZP crystals to change into monoclinic phase. Cyclic fatigue is expected to cause some phase transition. However, the minimal stress level applied may not have resulted in considerable change, corroborating the results of this study.

When it comes to water storage alone, significant decrease in bond strength seen in the present study which, could be attributed to hydrolysis of the polysiloxane network that connects the ceramic substrate with the polymerized intermediate resin suggesting that long-term water storage had a substantial impact on SBS. [9,11,14]

Regarding to combination of both thermocycling and cyclic load, **Al-Shehri et al. 2017** [18]stated that, the bond strength of zirconia was significantly influenced by the combination of thermocycling and cyclic loading that supporting results of this study, when combination occurred between thermocycling and cyclic loading and long-term water storage and caused significant decrease in SBS.

Significant decrease in SBS occurred when the two bond-weakening processes (hydrolysis and fatigue) were combined in relation to thermocycling, cyclic loading and water storage.In clinical settings, different kinds of FDPs may lose as a result of gradually lowering bonding levels [18].

After applying artificial aging techniques and subsequent de-bonding for testing, the failure patterns of all de-bonded specimens primarily displayed adhesive failure patterns (41 specimens), then by mixed failure patterns (12 specimens), with no occurrences of cohesive failure patterns noted.

The limitations of this in-vitro study might be that, more researches are needed to validate this testing approach, including examining the effect of increasing the load, fatigue cycle and thermal cycles numbers, as well as testing other adhesive cements and surface treatments.

oading) group**.**

4. CONCLUSIONS:

Based on the results of this study, can conclude the following:

1. Shear bond strength was decreased after all aging regime
2. Thermocycling alone and cyclic loading alone had a minimal effect on shear bond strength compared to long term water storage alone.

5. Declaration

Acknowledgements:

Not applicable.

Funding:

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Availability of data and materials:

The data sets that were utilized and/or analyzed for this investigation can be obtained from the relevant author at any reasonable request.

Ethical approval

Prior to the project, the Mansoura University Faculty of Dentistry's institutional ethical committee accepted the study protocol with code number (A03023Fp).

Conflict of interests:

The authors declare that they have no conflict of interests or financial disclosure.

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