

EFFECT OF CURING METHODS OF RESIN CEMENT BONDED TO CERAMICS MATERIALS ON BOND STRENGTH

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ABSTRACT

Objectives: The purpose of this study was to investigate the curing method-dependent bond strength of resin cement to high translucent monolithic zirconia and lithium disilicate glass ceramic.

Methods: Two ceramic materials were used in this study: high translucent zirconia (HTZ) from DD BioZx2 A3-HT and lithium di-silicate (IPS e-max CAD LT A3, C14). Based on the kind of ceramic utilized, forty ceramic discs (10 x 12 mm) were built and categorized into two groups (20 discs each). Each group was then further divided into two subgroups (10 discs each) based on the curing process (dual-cured and light-cured). Following their bonding to a resin cement cylinder of 5 mm in diameter and 2 mm in height, all samples were subjected to micro-shear bond strength test (μ SBS).

Results: There was no statistically significant difference between the light-cured and dual-cured groupings, in the E-max group ($P=0.785$). As for the zirconia subgroups, there was no statistically significant difference between light-cured and dual-cured subgroups ($P=0.057$). There was a statistically significant difference between the two materials' samples, with the E-max samples demonstrated a higher bond strength in both the light and dual-cured subgroups ($p=0.001$).

Conclusions: There is no difference in light-curing nor dual-curing regarding the micro-shear bond strength of E-max or zirconia. E-max has higher bond strength in both modes of curing than zirconia.

KEYWORDS: CAD/CAM, E-max, Zirconia, Curing mode, Dual-cure, Light-cure, Resin Cement, Micro-shear bond strength.

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INTRODUCTION

In order to give better aesthetics and the best possible bond strength, a variety of resin cements have been developed as a result of the growing usage of ceramic restorations. Luting agent types & properties used have a direct impact on how well ceramic restorations function in clinical settings. The effectiveness of ceramic bonding depends critically on the resin cement's superior adherence to the tooth structure and restorative surface.⁽¹⁾

Over the past few decades, zirconia has emerged as one of the most popular ceramics in prosthetic dentistry. Because of its crystal content and transformation toughening from crystal transformation, the material possesses a high flexural strength. Because of these properties, it may be used as a monolithic restoration or as the main component of bi-layered restorations.⁽²⁾

Resin-based composite luting agents are now a crucial part of restorative practice due to the growing usage of ceramic restorations. Fiber and metal posts, ceramic crowns, porcelain veneers, ceramic inlays and onlays, adhesive (bonded) bridges, orthodontic brackets, and other materials are all cemented using composite luting agents.⁽³⁾

Many clinicians have lost faith in bonded zirconia restorations due to the problem of debonding in recent years. One of the main causes of debonding was contamination during the try-in phase because of zirconia's strong affinity for proteins, lipids, and saliva. Conventional techniques for glass ceramics, such as phosphoric acid, heating, ultrasonic bathing, and so on, cannot be utilized to decontaminate them.⁽⁴⁾

Using aluminum oxide particles for airborne abrasion, 10 mm from the fitting surface with 50 µm particles is the most used technique. This method is frequently used in conjunction with primer that contains 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) to form a chemical connection.⁽⁵⁾

The null hypothesis of this study postulates that neither the light-cured resin cement nor the dual-cured resin cement will have a significant effect on bond strength of glass or zirconia ceramics.

MATERIAL AND METHODS

The materials used in this study are illustrated in the following table (Table1):

TABLE (1) List of materials used

	Brand Name	Material's Description	Manufacturer	Lot Number	Composition
1	IPS E.max CAD	Lithium disilicate glass-ceramic	Ivoclar Vivadent, Amhrst, USA	#65701	SiO Additional contents: Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅
2	Zirconia (HTZ) DD BioZx2	Monolithic zirconia	Spence, Germany	#667	Zirconium Oxide ZrO ₂ Yttrium Oxide Y ₂ O ₃ Hafnium Oxide HfO ₂ Aluminum Oxide Al ₂ O ₃
3	G-Cem One	Dual cure self-adhesive resin cement (translucent)	GC America Inc	#2311151	Dimethacrylate, urethane dimethacrylate (UDMA), titanium dioxide, Monomer, synergist, photoinitiator, stabilizer, initiator
4	Bisco Choice 2	Light-cured resin cement (translucent)	bisco, U.S.A	#C-411A1	Triethylene Glycol Dimethacrylate Tetrahydrofurfuryl Methacrylate Trimethylolpropane Trimethacrylate
5	Porcelain Etchant	9.5% Hydrofluoric acid gel	bisco, U.S.A	#230000105	Hydrofluoric Acid, aqueous solutions
6	Porcelain primer	Silane coupling agent	bisco, U.S.A	#2200007072	Acetone, (Trimethoxysilyl)propyl-2-Methyl-2-Propenoic Acid
7	One Coat 7	Light-cured universal adhesive	coltene, Switzerland	#N02531	diurethane dimethacrylate, 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, ethanol, diphenyliodonium chloride, diphenyl (2,4,6- trimethylbenzoyl) phosphine

To determine the right sample size required to detect a statistically significant difference in micro-shear bond strength between the groups under this study, a power analysis was conducted. With a power of 80% ($\beta = 0.2$) and a significance level set at 0.05, the analysis indicated that 40 samples in total—10 for each group would be required.

These calculations were made using information from a previous study ⁽⁶⁾. Using a fixed effects model and a one-way ANOVA F-test in an omnibus style, the sample size was calculated using G Power software version 3.1.9.4 (Heinrich-Heine University, Düsseldorf, Germany).

Sample Grouping:

Forty ceramic discs measuring 10 x 12 mm were made, and they were divided into two groups of twenty each based on the type of material tested. Each group was then subdivided into two subgroups, each consisting of 10 discs, based on the resin cement's curing method (dual-cured or light-cured).

Ceramic Discs fabrication

With the use of CAD software (exocad GmbH, Darmstadt, Germany), a ceramic cylinder with dimensions of 10 mm in diameter and 12 mm in length was created for both type of ceramic materials high translucent zirconia (HTZ) from DD BioZx2 A3-HT and lithium di-silicate (IPS e-max CAD LT A3, C14).

Since the shrinkage ratio of the utilized zirconia blocks was 0.25, the size of the zirconia cylinders was made 12.5 x 15 mm to compensate for the shrinkage that would happen after sintering. After being verified, the cylinder's form was sent to the CAM system (VHF, Ammerbuch, Germany). Following sintering, cylinders were cut with an IsoMet 4000 micro saw (Buehler Germany precision cutting, Germany) to produce discs 2 mm thick and 10 mm in diameter. Each disc's thickness was then measured using a digital caliber (Mitutoyo, Mitutoyo America Corporation, California).

E-max Partially crystallized blocks were milled in a CAD/CAM machine (VHF, Ammerbuch, Germany) into the designed cylinders. Each block was inserted in the milling machine and secured in place using the latch driver provided by CAM 5-S1 impression milling unit. The milling order was sent from the digital software to the milling machine to mill the cylinder according to the desired design. The milling process included the use of diamond burs (VHF, Ammerbuch, Germany) under copious water irrigation to prepare the ceramic block to the desired dimensions. It took around 40 minutes for each milling process after which the cylinders were cut to shape but still attached to the metal handle of the block. The milled cylinders were removed from the machine with the same latch driver and a diamond disc (Dental Diamond Disc, Henry Schein Dental, USA) was used to cut the handle off. All specimens were then cleaned ultrasonically in a distilled water bath for 10 min, compressed-air-dried, then crystallization was done in a sintering oven (MIHM-VOGT GmbH & Co. KG, Stutensee-Blankenloch, Germany) according to the manufacturer's instructions (850°C for 25 minutes).

Finishing of the Samples

E-max CAD finishing and polishing: The specimens underwent finishing and polishing procedures using Eve Diapol, EvE Ernst Vetter GmbH (Rastatter Str, Pforzheim, Germany). According to manufacturer instructions, initially, finishing with the green discs (medium) with approximately 35 microns was done. Subsequently, the grey wheel (fine) with a particle size of 4–8 microns was used for pre-polishing and smoothing. Finally, for high-luster polishing, the pink wheel (extra fine) with a particle size of about 1-2 microns was used. Every step of the process took one minute, and the recommended speed was 7000 rpm. Polishing was executed using a straight handpiece (NSK EX-6B, Japan), mounted to a specialized device to ensure standardization of grinding pressure, direction, and rate applied to the samples.

Monolithic zirconia polishing and finishing: light blue silicone points were used for initial finishing at 15,000 rpm, followed by dark blue silicone points for polishing at 15,000 rpm, and a nylon brush was used in conjunction with diamond polishing paste for final polishing at 10,000 rpm. The polishing was done using the OptraFine ceramic polishing kit (Ivoclar Vivadent). Every point will be utilized for 40 seconds.

Fixation of the ceramic specimen in an acrylic mold: Each ceramic disc was embedded and fixed in a 25 mm internal diameter polyvinyl chloride (PVC) tube (Misr El-Hegaz, Cairo, Egypt) that had been filled with a self cure acrylic resin (Acrostone, Cairo, Egypt) in order to serve as a mold for the fabrication and testing process. The upper surface of the PVC tube was flush with the upper surface of the acrylic.

Surface treatment of the ceramic surface was performed according to the manufacturer's instructions. Twenty E-max CAD cylinders were treated using a traditional surface treatment method. For 20 seconds, 9.6% HF acid was applied to etch the bonding surfaces. Followed by a 30-second air/water spray wash and a 10-second drying period ⁽⁷⁾. After applying a puddle coat of silane coupling agent (Porcelain primer Bisco, U.S.A.) to the surface, it was allowed to react for 60 seconds before being allowed to air dry without being rinsed.

Twenty monolithic zirconia CAD discs were spaced 10 mm from the sample surface and sandblasted with 50 μ m Al₂O₃ (Korox; Bego, Bremen, Germany) for 15 seconds at 2.5 bars of pressure perpendicular.

Bonding of resin cement samples to the ceramic specimens:

1. One coat seven universal bond was applied to all discs (E-max and Zirconia) then received a second layer of bond, which was applied with a microbrush (Meta-Biomed, Republic of Korea) and dried for ten seconds. The discs were then

light cured with curing unit wide-spectrum LED (Fanta, China) for twenty seconds.

2. To standardize the size of the composite resin, samples were created using Tygon tubes (Saint-Gobain, Paris, France) to hold resin cement with a height of 2 mm and an internal diameter of 5 mm. **(Fig 1).**⁽⁸⁾
3. Using G-cem one dual-cured translucent resin cement, a total of twenty resin cement samples were created, ten for each kind of ceramic material (E-max and zirconia).
4. The dual-cured resin cement was applied to the ceramic bonding region via mixing tips, to remove any bubbles in mixing tip of the dual-cure resin cement syringe a tiny amount of material was poured onto a mixing pad after resin cement was mixed in a 1:1 ratio per the manufacturer's directions to ensure there is no bubbles then it was applied directly into the tygon tube.
5. Using Bisco choice 2 light-cured translucent resin cement, a total of twenty resin cement samples were created, ten for each kind of ceramic material (E-max and zirconia).
6. Resin cement was dispensed directly into the tygon tube using the tip of syringe and ensured a good packing of resin cement by tapping with a metal spatula over celluloid matrix strip (TOR-VM, Moscow, Russia) that was placed above the tube.

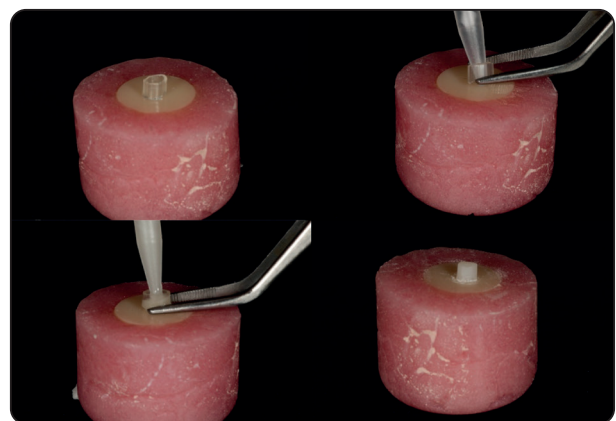


Fig (1) Tygon tube on every ceramic disk.

7. Light-cured and dual-cured samples were light-activated with curing unit wide-spectrum LED (Fanta, China) Curing Light, with a tip diameter of 9 mm and irradiance of 1200mw/cm². Held in direct contact with ceramic for 40 seconds with normal curing mode.
8. After twenty-four hours all tygon tubes were removed using scalpel and were ready to be tested.

Micro-shear bond strength testing.

An Instron universal testing machine (type 3345, USA) was used to perform micro-shear bond strength tests. Each sample (acrylic blocks containing the specimens) was positioned horizontally and separately on the machine's lower fixed head. Using 0.14-inch-diameter stainless steel wire connected to the testing machine's upper moveable head, which was positioned as close to the cement/slice interface as feasible, each tube was put through a micro-shear bond strength test (also known as a tugging test). Tensile tension was applied during the test up until specimen failure at a crosshead speed of 1.0mm/min (**Fig 2**).



Fig. (2) Micro shear bond testing using Universal testing machine.

An audible sound and indications of debonding between the cemented discs were signs of failure. The following formula was used to calculate micro-shear bond strength: The bond strength in MPa was calculated by dividing the load at failure (N) by the bonding area (mm²).

$$\tau = P / \pi r^2$$

where r is the disk's radius (mm), $\pi = 3.14$, P is the load at failure (N), and τ is the micro-shear bond strength (MPa). Software (Instron's Bluehill Lite Software) was used to conduct these tests. The program was used to record data. Following collection, the data were collated and subjected to statistical analysis.

Statistical analysis:

Was performed using a commercially available software program (SPSS 20-Statistical Package for Scientific Studies, SPSS, Inc., Chicago, IL, USA) for Windows. Numerical data were summarized using mean and standard deviation. Data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. Based on the parametric distribution of most data, comparisons between groups and between subgroups were performed using independent t test. All p-values are two-sided. P-values ≤ 0.05 were considered significant.

RESULTS

Means and standard deviations of the micro-shear bond strength (μ SBS) were measured for both material groups, lithium disilicate (E-max) and monolithic zirconia and among the subgroups according to curing technique (light-cure and dual-cure). The results were presented in tables (2-5) and figures (3-6) as follows:

i- Regarding values of μ SBS of (E-max samples)

Light-cure and dual-cure subgroups, results showed that there was no statistically significant difference between the subgroups P-value=**0.785**. (Table 2, fig.3).

ii- Regarding values of μ SBS of (Zirconia samples)

Light-cure and dual-cure subgroups, results showed that there was no statistically significant difference between the subgroups P-value=**0.057**.

Light-cure subgroups showed more bond strength than -cured ones. (Table 3, fig.4).

iii- Regarding values of μ SBS of (Light-cure subgroups)

Light -cure of E-max vs light-cure of zirconia groups, results showed that there was statistically significant difference between the subgroups P-value=**0.001** Light-cure of E-max subgroup showed more bond strength than zirconia one. (Table 4, fig. 5).

iv- Regarding values of μ SBS of (Dual-cure subgroups)

Dual-cure of E-max vs dual-cure of zirconia groups, results showed that there was a statistically significant difference between the subgroups P-value=**0.001**

The dual-cure subgroup of E-max showed more bond strength than zirconia one. (Table 5, fig.6).

TABLE (2) Comparison of Micro-shear bond strength of E-max samples in light-cure & dual-cure Groups in (MPa):

SBS	Dual-cure	Light-cure	P-value
Mean \pm SD	30.13 \pm 4.74	30.95 \pm 3.32	0.785
Range	23.90-35.30	27.60-35.50	

TABLE (3) Comparison of Micro-shear bond strength of zirconia samples in light-cure & dual-cure groups in (MPa):

Strength	Dual-cure	Light-cure	P-value
Mean \pm SD	13.45 \pm 1.79	10.78 \pm 1.39	0.057
Range	11.46-15.45	9.12-12.31	

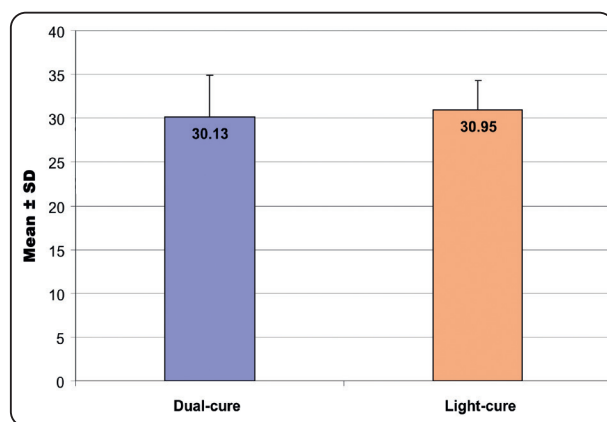


Fig. (3) Mean μ SBS between E-max samples in light-cure & dual-cure groups.

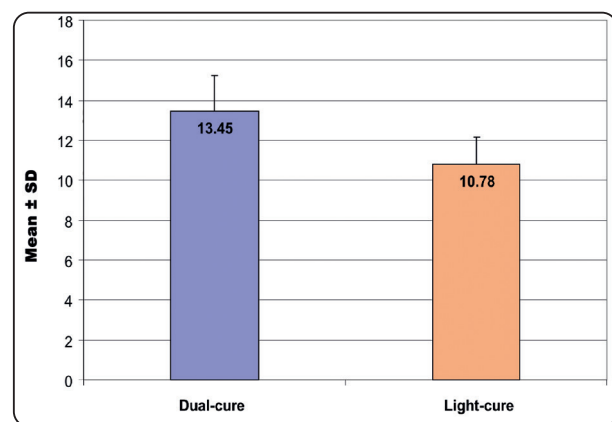


Fig. (4) Mean μ SBS between zirconia samples in light-cure & dual-cure groups

TABLE (4) Comparison of micro-shear bond strength of light-cure samples of E-max vs light-cure of zirconia in (MPa):

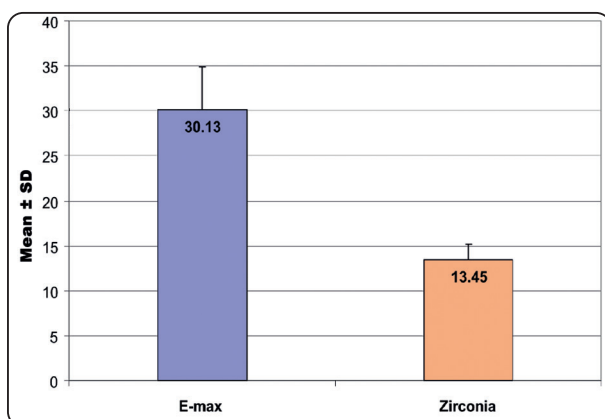
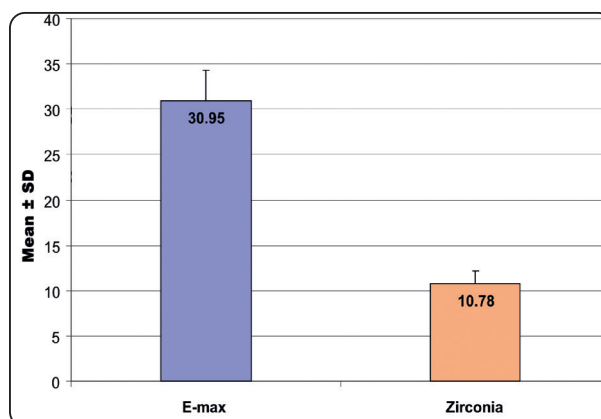
Strength	E-max	Zirconia	P-value
Mean \pm SD	30.13 \pm 4.74	13.45 \pm 1.79	0.001*
Range	23.90-35.30	11.46-15.45	

**Mean differences between Group Comparison with Post-hoc pairwise comparisons*

TABLE (5) Comparison of micro-shear bond strength of dual-cure samples of E-max vs dual-cure samples of zirconia in (MPa):

Strength	E-max	Zirconia	P-value
Mean \pm SD	30.95 \pm 3.32	10.78 \pm 1.39	<0.001*
Range	27.60-35.50	9.12-12.31	

**Mean differences between Group Comparison with Post-hoc pairwise comparisons*

Fig. (5) Mean μ SBS between light-cure samples of E-max vs light-cure of zirconia.Fig. (6) Mean μ SBS between dual-cure samples of E-max vs dual-cure of zirconia.

DISCUSSION

Nowadays, ceramic restorations are very esthetic restorative material that can mimic the look of natural teeth. Dental ceramics are brittle under tensile strain and prone to breaking under chewing pressures, despite their many advantages.⁽⁹⁾

A strong resin connection between a ceramic restoration and the tooth structure transfers functional loads across the bonded interface and gives the restoration good support.⁽¹⁰⁾

This study examined whether the curing process (light vs. dual) significantly alters the bonding strength of resin cement to lithium disilicate glass ceramic and highly translucent monolithic zirconia. Dual-cure resin cement is widely used in practice because there can be a section of the restoration where the light can't reach. Additionally, the resin cement may not get enough light energy due to the opacity of restoration.⁽¹¹⁾

In this work, monolithic zirconia and IPS E.max CAD were utilized. Additionally, the samples

measurement were carried out in accordance with ISO standard. ⁽¹²⁾

Dental computer-assisted design and computer-assisted manufacturing, or CAD-CAM technology, is widely used because it reduces the number of clinical visits and production time needed to create ceramic restorations. Clinicians use ceramic restorations because they are more chemically stable and biocompatible than conventional metal-ceramic restorations. ^{(13), (14)}

For robust and long-lasting adhesive bonding, resin cement adhesion requires surface pretreatment of restorative materials. ⁽¹⁵⁾

When proper preparation techniques are used, silica-based ceramics have demonstrated a high bonding strength (up to 71.5 MPa) to resin cement ^{(16), (17)}. Since air abrasion has been shown to have a greater failure rate and problems when applied to thin veneers, HF etching and silane application are preferable methods ^{(18), (19)}.

Researches revealed that an extra priming step strengthened the bonding between adhesive resin cement and zirconia ^{(20), (21)}.

MDP primer was selected because it increases the substrate surface's wettability for resin bonding, and the physio-mechanical interaction that occurs within the adhesive interface produces dependable adhesion and advantageous chemical stability, facilitating the formation of a chemical bond with zirconia that is resistant to water ⁽²²⁾. Additionally, a previous evaluation found that the combination of air abrasion and agents based on 10-MDP produced the strongest bond and longest-lasting adherence to zirconia ⁽²³⁾ and this is what was employed in this present investigation.

Stresses at the ceramic-tooth contact are complicated and can be classified as shear or tensile stresses, which are produced by forces acting parallel or perpendicular to the tooth surface ⁽²⁴⁾. Furthermore, the forces of displacement of the crown tend to be closer to shear stresses than to

tensile stresses, according to *Holderegger et al.* (2008) ⁽²⁵⁾. μ SBS was the most straightforward and widely used testing procedure, according to *Oilo's* 1993 discussion of the clinical significance and accuracy of several testing techniques ^{(26), (27)}.

Therefore, the resin cement's binding strength has been measured in this study using the μ SBS test.

The micro-shear bond strength of dual-cured and light-cured resin cement in E-max CAD are comparable, according to the findings of the μ SBS test. These results are consistent with earlier research. ^{(28), (29)}

This result contradicts that of *Lühns A. K. et al.* (2014) ⁽³⁰⁾, who found that dual cure resin cement with light curing provides ceramic with a stronger bond. Methodological differences across research, such as sample preparation, irradiation through the restorations, and resin cement type, may be the cause of the disparities in the outcomes.

In zirconia group, the light-cure subgroup exhibited a higher micro shear bond values than the dual-cured one, although zirconia groups revealed no statistically significant difference in micro-shear bond strength between dual-cured and light-cured resin cement. These results are consistent with earlier research and can be explained by the increased degree of resin cement conversion following light irradiation. ^{(31), (32)}

In accordance with earlier research ^{(33), (34)}, the results of μ SBS also showed that resin cement (light-cured or dual-cured) with E-max CAD and zirconia differed in micro-shear bond strength, with the E-max samples having a greater bond strength than zirconia.

Because only controlled variables are taken into account in this in-vitro approach, the current study has inherent limitations. Since the various materials used in the study have larger thermal contraction/expansion coefficients than teeth, intraoral temperature variations may have an impact on the indirect restoration's long-term results.

CONCLUSIONS

1. There is no difference in light-curing nor dual-curing regarding the micro-shear bond strength of E-max or zirconia.
2. E-max has higher bond strength with both modes of curing than zirconia.

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