

Original Article

Shear Bond Strength of Zirconia Versus Zirconium Silicate Filled Indirect Composite Resin (In Vitro Study).

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Abstract

The aim of this study was to determine the shear bond strength of zirconium dioxide and indirect zirconium silicate filled composite resin materials bonded to dentin using self-adhesive resin cement. According to the materials used, a total of 20-disc samples with dimensions of 10 mm in diameter and 2 mm thickness were equally divided into two groups (n=10). Group I: Zirconia ML (Katana, Kurary Noritake, Tokyo, Japan) and Group II: Ceramage “indirect zirconium silicate filled resin composite material (Shofu, Tokyo, Japan). Samples of both groups were bonded with self-adhesive resin cement to dentin. Universal testing machine (Instron) was used to measure the shear bond strength. Results: A higher mean shear bond strength value (5.67 + 3.36 MPa) was recorded with Ceramage (Group II), while a lower mean value (3.44 + 1.57 MPa) with Zirconia ML (Group I). Statistical analysis revealed no statistically significant difference between both tested groups. Conclusions: Indirect composite resin material filled restorative material did not show any significant increase in shear bond strength when compared to Zirconia ML restoration.

Keywords: monolithic zirconia, zirconium silicate filled resin, shear bond strength

I. INTRODUCTION:

The increased demands and high esthetic expectations of patients lead to an increase in the use of metal-free tooth-colored materials, including ceramic restorations and indirect resin restorations. Although all-ceramic restorations generally meet these esthetic expectations, a major drawback is their brittleness, which results from the low tensile stress and fracture toughness of the material.¹ Currently, ceramic restorations are frequently milled out of industrially made computer aided designed and manufactured ceramic blocks.²

Besides different types of ceramic blocks (Feldspathic ceramic, reinforced glass ceramics, zirconia, etc.), new materials including resin-ceramic hybrid materials, have been developed for computer-aided design and computer aided manufacturing (CAD/CAM) technique.^{2,3} These materials combine the advantageous properties of ceramics, such as durability and color stability, with those of composite resins, such as improved flexural properties and low abrasiveness. In addition to category of indirect composite resin materials which are highly filled with ceramic fillers to provide the same previous advantages.

During the last few years, zirconia restorations established themselves in conservative dentistry, thus allowing proper biocompatibility and great mechanical qualities. These qualities make zirconia optimal for high-stress-bearing areas.⁴ Despite these advantages, bonding between zirconia and tooth structure is problematic due to its inertness. The surface cannot be activated with hydrofluoric acid etching like other reinforced glass ceramics, as acid etching does not act on the crystalline component.⁵

Clinical success and longevity of indirect ceramic restorations depend on the cementation and bonding procedures.⁶ Adhesive cementation (AC) to zirconium oxide ceramics (Zr_2O) is desirable since it improves marginal adaptation, fracture resistance, and enables more conservative preparations. In order to enhance adhesion between the resin cement and zirconia different methods have been adopted: among which is the use of a phosphate-modified monomer (MDP) in resin cement, laboratory or chairside air-abrasion with 110 or 30 μ m Silica-coated aluminum particles. These methods produced controversial bond strength results for Zirconia.⁷⁻⁹

The cohesion of the tooth–restoration complex with a bonding technique involves the establishment of a durable link even within the substructures through using resin cement. Several experimental pretreatments of zirconia were proposed, including air-borne particle abrasion (ABPA) with alumina.¹⁰

Continuous evolution of polymeric materials has led to materials with the advantage of improved esthetic appearance, high abrasion resistance and color stability⁶, as well as lower abrasive impact on the opposing dentition.^{11,12} “Ceramage” one of the polymeric highly ceramic filled restorative materials has been introduced for dental application¹³. The special composition of this micro-hybrid composite system, with a zirconium silicate filler content of more than 70 %, allows the

fabrication of different esthetic indirect anterior and posterior restorations including veneers, crowns, occlusal veneers, and long term provisional restorations¹⁴. Another advantage is the low elastic modulus, which allows the material to absorb functional stresses produced under occlusal load which has a positive effect on the chewing behavior of patients with implant-supported restorations¹⁵.

Self-adhesive resin cements are an intermediate category between simple application of conventional and adhesive resin cements with their higher physical bond strength properties. It could be a reliable choice for efficient bonding with zirconia and zirconium silicate indirect composite resin hybrids¹⁶. In vitro studies among which are the shear bond strength (SBS) test are essential for understanding the laboratory performances of materials that could be a prediction of their clinical efficacy. Accordingly, this study aimed to determine the shear bond strength of zirconium dioxide and indirect zirconium silicate filled composite resin materials bonded to dentin using self-adhesive resin cement. The null hypothesis of this study is that there is no difference in shear bond strength between zirconia and a highly ceramic filled indirect composite resin when bonded to dentin.

II. MATERIALS AND METHODS

A. Sample size calculation

This power analysis used shear bond strength as the primary outcome. Based upon the results of Sari F et al.⁽¹⁷⁾; the mean and standard deviation (SD) values were 12.49 (2.7) and 18.41 (3.99) MPa for the two groups, respectively. The effect size (d) was 1.738. Using alpha (α) level of (5%) and Beta (β) level of (20%) i.e., power = 80%; the minimum estimated sample size was 7 specimens per group. Sample size calculation was performed using G*Power Version 3.1.9.2.

B. Samples grouping

Discs of two indirect zirconia-containing restorative materials of shade A2 were used in the current study (Table 1). A total of twenty-disc samples were equally divided into two groups, each of ten discs (n=10) according to the material used as follows: **Group I** (control): Zirconia ML (Multi Layered) (Katana, Kuraray Noritake Dental, Tokyo, Japan); **Group II**: Indirect Zirconium silicate filled composite resin (Ceramage, Shofu, Kyoto, Japan).

C. Sample preparation

For the fabrication of Group I Zirconia ML (Katana) disc samples, a specially crafted Teflon mold of 10 mm internal diameter and 2 mm thickness was scanned with an extra-oral scanner (D/R2000, 3 shape, Copenhagen, Denmark). The 3D shape design of the disc was saved in a Standard Tessellation Language (STL) file format. The produced (STL) file was then exported to a milling machine (K5+, VHF, Ammerbuch, Germany). The milling process was performed under a copious amount of water irrigation. After completion of the milling process the produced Zirconia ML discs were glazed and sintered in the zirconia-sintering furnace (Noritake KATANA F-1; Kuraray Noritake, Tokyo, Japan) using the recommended 7-hour sintering schedule with maximum temperature=1550°C and hold time = 2 hours according to the manufacturer's instructions^{10,18}. While, for Group II Indirect Zirconium silicate filled composite resin (Ceramage) samples, discs were prepared in a specially designed Teflon mold (10 mm internal diameter × 2mm thickness). The indirect resin composite material was condensed into the mold using a load transfer device at the force of 1 kg and then two microscopic glass slides with smooth polished surfaces were used to pack it. Discs were initially cured with light curing device

(dentmax, 470-570 nm, LED, Korea), complete curing of the material was done in special curing unit (Solidilite V Light-Curing Unit, Shofu, Kyoto, Japan)¹⁹ then finished and polished as recommended by the manufacturer. Finally, the prepared samples for both groups were carefully inspected then stored for 24 hours at 37°C in an incubator prior to bonding procedures

D. Bonding procedures:

Freshly extracted human molar teeth for orthodontic purposes were collected from teeth bank MIU University, Obour City. All teeth were selected to be free of caries or any cracks. Molars were further cleaned from any debris or blood remnants, then each tooth was embedded individually in an acrylic mold. The occlusal surfaces of the selected teeth were wet ground using 600 grit silicon carbide (SiC) paper to expose dentin. Teeth were stored in distilled water for 24 hours to be ready for cementation procedures. Discs of both groups were bonded to tooth structure. Cementation was carried out according to the manufacturers' recommendations. First, the surfaces of both studied materials were sandblasted with 110 µm aluminum oxide Al₂O₃ particles using a micro-sandblaster (Oxyker duet, Fili Manfredi, Italy) at pressure 0.25 MPa at an angle of 45° from a distance of 10 mm for 5 seconds. All discs were then cleaned in an ultrasonic cleaner using distilled water for 5 minutes, then left to dry. After rinsing the exposed dentin surface under a stream of distilled water and dried with compressed air, discs of both groups (Katana & Ceramage) were cemented by self-adhesive dual cured resin cement (Rely X200 Uni-cem 3M dental company) to dentin²⁰. Firstly, resin cements were applied to the dentin surface, for both groups disc samples were applied on top with a steady pressure by microscopic glass slabs. Excess resin cement was removed after soft light curing (5 seconds). Then, they were fully polymerized using an Elipar LED lamp (3M ESPE, Maplewood, Minnesota, USA) for 20 seconds. Before performing shear bond

strength test, all polymerized samples were stored in an incubator with 100% humidity at 37°C for 24 hours²¹.

E. Shear Bond Strength (SBS) test:

All samples were individually and horizontally mounted on a computer-controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a loadcell of 5 kN and data were recorded using computer software (Bluehill Lite; Instron Instruments). A circular interface shear test was designed to evaluate the bond strength. Jakobe's chuck was used as sample holder. The sample holder was secured to the lower fixed compartment of the testing machine by tightening screws. Shearing test was done by compressive mode of load applied at tooth-resin interface using a mono-beveled chisel shaped metallic rod attached to the upper movable compartment of the testing machine traveling at a crosshead speed of 0.5 mm/min (Figure 1). The load required to debonding was recorded in Newton²¹.

Shear bond strength (SBS) calculation.

The SBS was calculated from the following formula:

SBS (in MPa) = $P \times 9.8 / r^2 \times \pi$ where **P** is the maximum load (in kgF) when the disc samples were debonded, and **r** is the radius (in mm) of the disc samples. (Figure 2).

III. RESULTS

A. Statistical analysis:

Data management and statistical analysis were performed using the Statistical Package for Social Sciences (SPSS) version 18. Numerical data were summarized using median, means, standard deviations, minimum, maximum and confidence intervals. Data were

explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests.

Comparisons between the 2 groups with respect to non-normally distributed numeric variables were compared by Mann-Whitney test. All p-values are two-sided. P-values ≤ 0.05 were considered significant.

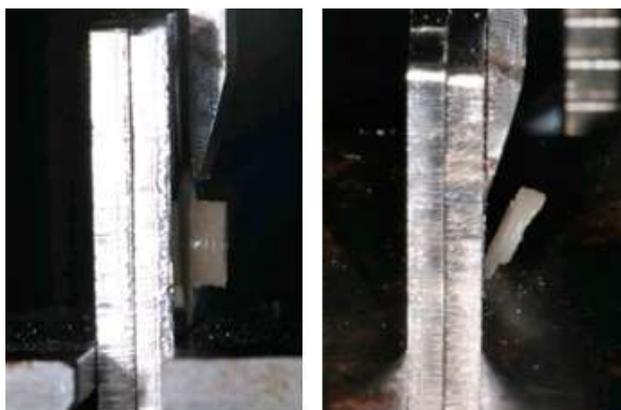
B. Shear Bond Strength test

A higher median (5.3) and mean value (5.67 ± 3.36) was recorded in Group II: Indirect zirconium silicate filled composite resin (Ceramage), in comparison to median (3.64) and mean value (3.44 ± 1.57) recorded in Group I: Zirconia ML, (Katana™). The difference was not statistically significant ($p=0.226$), results were presented numerically in (Table 2) and graphically in, (Figure. 3)

IV. DISCUSSION:

Since aesthetics is a key of success for dental materials, specific attention should be also given to the mechanical properties and bonding longevity of different restorations along with testing techniques and methodologies that determine the efficacy of bonded interfaces²²⁻²⁴. Despite the eminent advances achieved in adhesive dentistry, still the bonded interface is considered as the weakest point of an adhesive restoration^{25,26}.

Behavioral performance of dental materials should be assessed by different testing techniques. One of these methods is to examine the ability of a material to bond to a substrate through various bond strength tests²⁷. Although the relationship between bond strength test results and reliability of clinical performance for dental adhesives remains questionable, recent evidence proves that clinical reliability can, to some extent, be predestined based on laboratory results^{28,29}.



Figures (1) and (2): Shear bond strength testing Figure and Debonding of disc sample

Table 1: Materials used within this study.

Material	Filler by weight	Composition
Katana™ Zirconia, ML, Japan.	< 1 %	ZrO ₂ +HfO ₂ +Y ₂ O ₃ monolithic (Tokyo, Japan)
	> 99%	Yttria stabilized zirconia & Other oxides
Ceramage, Kyoto, Japan	73%	Zirconium silicate ceramic
	5-15%	Urethane dimethacrylate (UDMA)
	>12%	NP (not provided by the manufacturer)

Table (2): Descriptive statistics and comparison of shear strength (MPa) in both groups (Mann Whitney U test)

	Median	Mean	Std. Dev	95% Confidence Interval for Mean		Min	Max	P value
				Lower Bound	Upper Bound			
Katana™ Zirconia ML	3.64	3.44	1.57	1.61	5.28	.50	9.51	
Ceramage Zirconium silicate resin	5.3	5.67	3.36	3.27	8.07	.12	9.70	.226 ns

Significance level $p \leq 0.05$, ns=non-significant

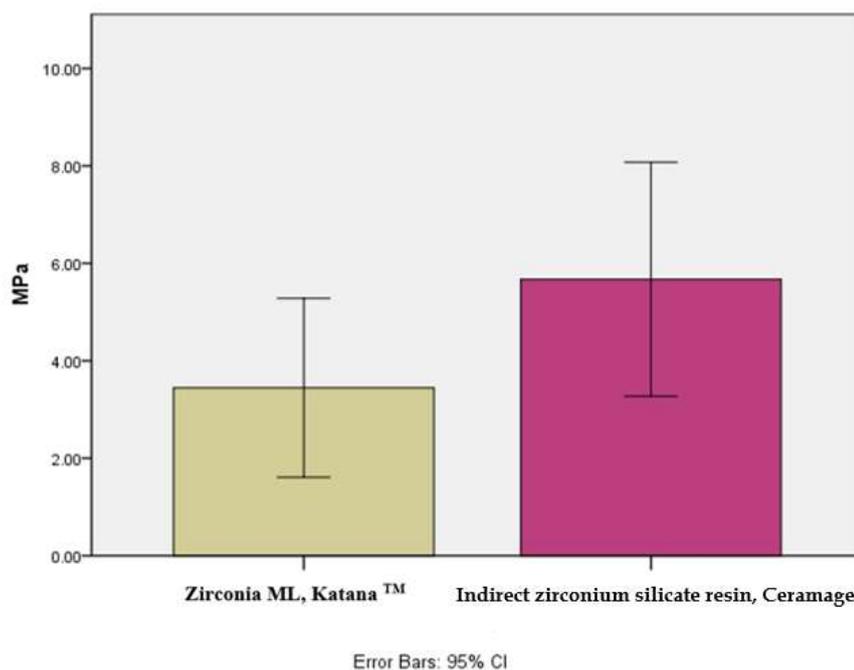


Figure (3): Bar chart illustrating mean shear strength (MPa) values in both groups

Accordingly, the aim of this study was to evaluate the shear bond strength of Zirconia ML (Katana) in comparison to indirect zirconium silicate filled composite material (Ceramage), to tooth structure.

Disc samples were fabricated according to the manufacturer instructions for each product. Zirconia ML (Katana) discs were fabricated using CAD/CAM system, sintered, and glazed to simulate the clinical condition.^{21,30} While, Ceramage composite disc samples had been initially cured, then were further subjected to an additional curing cycle to ensure total resin polymerization.

Bonding of indirect restorations to tooth structure can be either mechanical or chemical. As agreed with several researchers,^{18,19,20,31} attempts to enhance mechanical retention through creating surface roughness includes multiple approaches as, sandblasting, hydrofluoric acid application or roughening using carbide bur.

Sandblasting was the method adopted for surface treatment of materials under investigation. Prior to bonding procedures, disc

samples of both groups were subjected to sandblasting using 110 μm Al_2O_3 aluminium oxide particle size to enhance bonding³².

de Castro et al³³, suggested that surface treatment of highly crystalline Zirconia samples which lack silica and resist etching with hydrofluoric acid require creation of surface roughness prior to bonding procedures. This could be achieved by sandblasting with aluminium oxide particles. Whereas silica coating or adding of MDP Zirconia primer improve the bond chemically³⁴.

Regarding Ceramage, mechanical roughening of the restoration surface before bonding procedures is highly recommended by the manufacturer. Sandblasting of Ceramage discs help to increase the mechanical interlocking, as well as a larger surface amount of free-carbon residue left, is produced by this method that plays an important role in bonding³⁵. The bond between indirect ceramic filled composite “Ceramage” and tooth structure includes two different interfaces, one between the composite resin and resin cement

while the other, between resin cement and tooth structure³⁶.

Samples of both materials were cemented to tooth structure using dual cured self-adhesive resin cement. Adhesive resin cements are recommended for zirconia restorations to ensure their clinical success as glass ionomer cements have minimal bonding strengths to zirconia and are susceptible to water degradation due to their chemistry³⁷. Being less technique sensitive and in clinical situations it doesn't totally remove the smear layer from dentin surface hence, reducing postoperative hypersensitivity, adhesive resin cement is advisable for bonding of indirect micro-filled composite resin restorations³⁷. Methacryloyloxyde Dihydrogen Phosphate monomers in self-adhesive cements have been proven to be effective for adhering to the non-silica-based polycrystalline materials of zirconia. Numerous studies have shown that phosphate monomers are promising chemical agents for improving zirconia bonding. This is due to the ability of phosphate monomers to form chemical bonds with the zirconium oxide layer on the surface, as well as having polymerizable resin terminal end groups (eg, methacrylate), which enable cohesive bonding to appropriate resin cements^{38,39}.

Because of the simplicity of the shear test protocol and easy specimen preparation, SBS testing is considered as one of the methods most used for bond strength measurement, and the results of the measured SBS with various conditions are reported in the literature⁴⁰. Moreover, the non-technique sensitivity makes it an extensively method for the evaluation of dental adhesives^{41,42}. Many studies showed higher strength values generally found with shear bond strength test⁴³. However, non-uniform stress distribution in the adhesive area should be taken into consideration^{44,45}. The explanation for this fact was that stresses are mostly concentrated in the tooth substrate or composite, hence causing its premature failure before failure of the adhesive interface itself⁴⁶. There are several factors contributing to the

variability in results retrieved from shear testing and the variation in a factor may lead to a dramatic change in the final results. The presence of different loading techniques, specimen dimensions, cross head speeds, bonding protocols, substrates, and storage conditions make it very difficult to compare results retrieved from different SBS studies or combine them in single meta-analyses

Within our study, shear bond strength test was performed by applying the force parallel to the bonding interface using a mono-bevelled knife-edged chisel, till debonding or failure occurred. The shear bond strength was calculated by dividing the maximum load(N) to the surface area in mm² of the disc sample. Namely shear bond strength value (in MPa) is the stress on the unit of area^{47,48}.

Failure mode analysis could provide highly valuable information for the detection of weaknesses in different testing methodologies in order to improve their reliability, providing results that represent the actual strength of adhesive junction.

The mean results of shear bond strength values of indirect zirconium silicate filled composite (Ceramage) used in this study (5.67 ± 3.36 Mpa) were high but not statistically significant higher than that of Zirconia ML discs (Katana) (3.44 ± 2.57 Mpa). Thus, the null hypothesis of this study was accepted.

The inferior bond strength of the control group of Zirconia ML (Katana) disc samples could be attributed to inertness and the polycrystalline microstructure of Zirconium oxide with lack of silica that can help to increase bond strength, when etched with Hydrofluoric acid with later salinized to produce efficient bonding^{49,50}.

However, studies conducted by Leinfelder KF⁵¹ and El saka SE⁵² their results for the indirect zirconium silicate filled composite resin when compared to other CAD/CAM composite resin blocks showed inferior bond strength due to the polymerization method of this type of restorations, which

creates multiple defects due to the incremental method of building up these restorations in the lab, which is mainly controlled by the operator.

One of the limitations of this study, being an in-vitro study, the samples were not subjected to thermo-mechanical aging thus, it didn't reflect the real conditions in the oral cavity and other affecting factors such as saliva, temperature and pH changes, and occlusion. The disc samples used did not represent the actual shape and dimensions of different restorations.

V. CONCLUSIONS

Within the limitations of this study, we can conclude that:

1. The use of zirconium silicate indirect resin restorative material did not improve the shear bond strength with tooth structure, when compared to zirconia.
2. Fabrication technique for different materials could affect the shear bond strength to dentin surface.

VI. CLINICAL RECOMMENDATIONS:

1. Surface treatment for both zirconia & hybrid ceramics is mandatory for efficient bonding in the oral cavity.
2. Clinicians should consider the oral hygiene of individuals when selecting aesthetic materials.

VII. REFERENCES

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