

Comparative Evaluation of Shear Bond Strength of Three Different Luting Cements toward Ceramic and Dentin for All Ceramic Restorations: An *in vitro* Study

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ABSTRACT

All ceramic restorations benefit from resin cement bonding to the tooth. No currently available luting agent is ideal for all situations. The comparison of the bonding ability of different luting cements to ceramic and dentin is thus deemed necessary. The purpose of this study was to evaluate the shear bond strength of different luting cements to both ceramic and dentin and then to evaluate the mode of bond failure by scanning electron microscopy.

Keywords: Luting cements, Bonding, Ceramic, Dentin, Shear bond strength.

How to cite this article: Zareen SA, Usman JAM, Haribabu R. Comparative Evaluation of Shear Bond Strength of Three Different Luting Cements toward Ceramic and Dentin for All Ceramic Restorations: An *in vitro* Study. *J Orofac Res* 2013;3(2):86-89.

Source of support: Nil

Conflict of interest: None declared

INTRODUCTION

Dental luting cements provide the link between a fixed prosthesis and the supporting prepared tooth structure. The ideal properties of a luting agent include fracture resistance, low solubility, color stability, adhesive bond to tooth structure, adequate working and setting time.¹ The first all ceramic crowns were developed by Charles Land in 1886 and was known as the porcelain jacket crown. For many decades it was the most esthetic full veneer restoration dentistry had to offer.² The clinical performance of all ceramic restorations is mainly influenced by the shape of tooth preparation and surface treatment of crown and abutment, type of luting agents and the adhesion between tooth structure and restorative material. However, ceramic restorations are very brittle which results in debonding and fracture of the restoration.³ To compensate for this, increase in bond strength with tooth structure becomes necessary.

When bonding ceramic to tooth structure, two different interfaces need to be considered: the dentin/cement interface and the ceramic/cement interface. Failure of adhesive seal in the above interfaces results in microleakage threatening clinical performance and longevity of the restorations, contributing to staining, recurrent caries, adverse pulpal response and postoperative sensitivity and finally the debonding of the restoration.⁴ There are wide variety of

cements, each with advantages and disadvantages. No currently available luting agent is ideal for all situations. Nevertheless, it is necessary to choose the right material from the various cements available for the long-term service of the restoration. The comparison of the bonding ability of different luting cements to ceramic and dentin is thus deemed necessary.

The aim and objective of this study was:

1. To evaluate and compare the shear bond strength of different luting cements to ceramic and dentin on the universal testing machine (Lloyds).
2. To evaluate the mode of bond failure by scanning electron microscopy (SEM).

MATERIALS AND METHODS

IPS Empress 2 heat pressed ceramic disk specimens of diameter 3 mm and width of about 2 mm were fabricated. Noncarious, intact, human mandibular premolars extracted for orthodontic purpose were collected and stored in saline until use. These teeth were then sectioned at cemento-enamel junction (CEJ) with diamond disk at 90° to long axis of the tooth and 2 mm coronally to CEJ to obtain tooth disk specimen of thickness 2 mm with sufficient area of dentin. Plastic mounting plates (approximately 40 × 4 mm) with machined screw fittings were fabricated. Each pair has an upper and lower plate. Upper plate has a beveled area of 15 mm in diameter and a hole of 3 mm in diameter which will hold the ceramic disk specimen. The lower plate has a trough of about 8 mm in diameter which holds the tooth specimen. The ceramic disk specimens were embedded in the upper plastic plate and the tooth specimens embedded in lower plate with autopolymerizing acrylic resin (DPI-RR Cold cure). A mylar strip was interposed between the specimens to standardize the cement thickness. Thirty disk specimens of all ceramic and dentin were fabricated and randomly divided into three groups of 10 each named as groups I, II and III.

Group I: Luting with GIC

Surface of tooth specimen was treated with conditioning paste (Proxylt paste, Ivoclar Vivadent, Liechtenstein). Then ceramic and tooth specimen were luted with type I glass ionomer cement (GIC) cement (Fuji I, GC Corp, Japan).

Group II: Luting with RelyX ARC

Surface of the tooth specimen was etched with 37% phosphoric acid (Gluma etch 20 gel, Heraeus Kulzer, Germany) for 15 seconds, rinsed for 10 seconds and gently air dried. Dentin bonding agent (Adper Single bond, 3M ESPE, USA) was applied and light polymerized for 10 seconds. Surface of ceramic specimen was etched with 9% hydrofluoric acid (Ultradent Porcelain Etch) for 20 seconds followed by silane application (Monobond S, Ivoclar Vivadent, Liechtenstein) and then luted with RelyX ARC luting cement (3M ESPE, USA) and plates were clamped with machined screws and cured for 20 seconds.

Group III: Luting with Variolink II

Surface of the tooth specimen was etched with 37% phosphoric acid (Gluma etch 20 gel, Heraeus Kulzer, Germany) for 15 seconds, rinsed for 10 seconds and gently air dried. Dentin bonding agent (Excite DSC, Ivoclar Vivadent, Liechtenstein) was applied and light polymerized for 10 seconds. Surface of ceramic specimen was etched with 9% hydrofluoric acid (Ultradent Porcelain Etch) for 20 seconds followed by silane application (Monobond S, Ivoclar Vivadent, Liechtenstein) and then luted with Variolink II luting cement (Ivoclar Vivadent, Liechtenstein) and cured for 20 seconds.

Testing Procedure

All the specimens were immediately placed in an isotonic saline solution and the machined screws were removed after 1 hour. After 24 hours, the specimens of groups I, II and III were subjected for shear bond strength using Lloyds universal testing machine. Shear load at failure was recorded in Newton's and converted to stress in MPa. Then the fractured specimens were further evaluated for SEM examination. Specimens were sputter-coated with gold alloy and examined under SEM at 20 kV and the specimens were viewed and photographed at original magnification $\times 1,000$.

RESULTS

The specimens were subjected to shear bond strength testing using universal testing machine (Lloyds) (Table 1). Mean and standard deviation were estimated from the sample for each study group. Mean values were compared between different study groups by using one-way analysis of variance (ANOVA) followed by Scheffe's multiple range procedure. In the present study, $p < 0.05$ was considered as the level of significance (Table 2). SEM study was done on fractured specimens and mode of bond failure was analyzed and tabulated⁵ (Tables 3 and 4).

Table 1: Shear bond strength (MPa) of different luting cements to both ceramic and dentin

No. of specimens	Group I	Group II	Group III
1.	5.2	15.00	16.26
2.	4.6	15.56	17.28
3.	4.2	14.14	15.27
4.	5.2	14.56	16.69
5.	4.9	14.00	16.40
6.	4.5	14.28	16.42
7.	4.8	14.00	17.11
8.	4.2	14.56	17.39
9.	5.0	15.84	16.17
10.	4.6	15.13	17.25

Table 2: Mean, standard deviation and test of significance of mean values between different groups

Groups	Mean \pm SD	p-value	Significant groups at 5% level
I	4.74 \pm 0.37	<0.0001	III vs I
II	14.71 \pm 0.65	Significant	II vs I
III	16.71 \pm 0.72		

Table 3: Scanning electron microscopic evaluation

Groups	Adhesive failure	Cohesive failure	Mixed failure
I Dentin	4 (16%)	19 (76%)	5 (20%)
I Ceramic	16 (64%)	4 (16%)	7 (28%)
II Dentin	4 (16%)	17 (68%)	7 (28%)
II Ceramic	4 (16%)	18 (72%)	6 (24%)
III Dentin	3 (12%)	19 (76%)	6 (24%)
III Ceramic	3 (12%)	20 (80%)	5 (20%)

Table 4: Scanning electron microscopic evaluation interface

Group I	Cement dentin interface—predominantly cohesive failures Cement ceramic interface—predominantly adhesive failures
Group II	Cement dentin interface—predominantly cohesive failures Cement ceramic interface—predominantly cohesive failures
Group III	Cement dentin interface—predominantly cohesive failures Cement ceramic interface—predominantly cohesive failures

Mean value in group III (16.71 ± 0.72) was significantly higher than the mean values in group I (4.74 ± 0.37) and in group II (14.71 ± 0.65) ($p < 0.05$). Further, the mean value in group II was significantly higher than the mean value in group A ($p < 0.05$) (Table 2).

DISCUSSION

Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties that better simulate the appearance of natural dentition. In spite of their many advantages, ceramics are fragile under tensile strain. This weakness can be attributed to the presence and propagation of microflaws present on the surface of the material, making

the ceramic susceptible to fracture, making the cementation process vital for the clinical success of all ceramic restorations. The purpose of this study was to evaluate the shear bond strength of different luting cements to both ceramic and dentin and then to evaluate the mode of bond failure by SEM. Owing to its widespread popularity and usage, one of the recently introduced ceramic system—IPS Empress 2 is used in this study. IPS Empress 2 is a lithium disilicate, heat pressed all ceramic material. IPS Empress 2 contains 70% lithium disilicate crystals and smaller concentration of lithium orthophosphate crystals.

Noncarious, intact, human premolars extracted for orthodontic purpose were used due to their easy availability. These were then sectioned and mounted on plastic mounting plates with machined screw fittings. Interposition of mylar strips ensured uniform film thickness of 40 μm .⁶ The luting agents selected for comparative evaluation were GIC and two commercially available resin cements—RelyX ARC and Variolink II resin cements. GICs was formulated in 1976 as a dental restorative material and has been in major use for more than 30 years for increased patient acceptance. These are primarily adhesive cements containing acid soluble calcium fluoroaluminosilicate glass and aqueous solution of polyacrylic acid in a concentration of about 40 to 50%.⁷ Owing to its widespread popularity and long track record and as the present study involved comparative evaluation of different luting cements, the inclusion of GIC in this study was considered appropriate.

Synthetic resin cements based on methyl methacrylate have been available from 1952 for cementation of inlays, crowns and appliances. Since 1986 resin cements have gained popularity because of their use in bonding of esthetic ceramic and resin bonded bridges. They can be polymerized by light, chemical polymerization or both. But, dual cured cements are found to have higher hardness values when compared to chemically cured cements.⁸ The major advantages of resin luting agents include increased bond strength when used in conjunction with silane coupling agents,⁹⁻¹¹ increases the fracture resistance of the tooth and the restoration itself and minimizes the microleakage due to better wettability and bonding to tooth structure.¹¹

In group I (luted with GIC)—the dentin surfaces were surface treated with conditioning agent (Proxylt paste Ivoclar Vivadent, Liechtenstein) to remove the smear layer and surface debris. The smear layer reduces dentin permeability and limits the strength of dentin bonding agents because of the relatively low cohesive forces holding the smear layer together and to the dentin. Its removal results in higher bond strength of dentin adhesives.⁵ It also promotes ion exchange, chemically cleans the dentin, and increases surface energy. In groups II and III (luted with RelyX ARC, Variolink II)—the dentin surfaces were first acid etched with 20%

phosphoric acid (Gluma etch 20 gel, Heraeus Kulzer, Germany) to remove the mineral phase and increase the porosities of the tissues resulting in the formation of resin tags which are extension of adhesion resin in to open dentinal tubules.⁶ This is followed by the application of dentin bonding agent to fill the resin tags and form a chemical bond between resin cement and dentin.⁵

The ceramic specimens were first etched with 9% hydrofluoric acid (Ultradent Porcelain Etch). Studies have shown that hydrofluoric acid attacks the glass phase of ceramics, creating surface microporosity, thereby allowing the formation of mechanical interlocking with resin. According to Holand et al, the main crystal phase of IPS Empress 2 glass ceramic is formed by elongated crystals of lithium disilicate. A second phase is composed of lithium orthophosphate. A glass matrix surrounds both crystalline phases. Hydrofluoric acid removes the glass matrix and the second crystalline phase creating irregularities within the lithium disilicate crystals and thereby results in increased bonding.¹² This is followed by silanization with Monobond S (Ivoclar Vivadent, Liechtenstein). Silane coupling agents enhances the formation of chemical bond between the inorganic phase of the ceramic and the organic phase of the resin and increases the wettability of ceramic surface.⁶ Other methods of surface treatment of ceramics include sandblasting with 50 μm aluminum oxide particles, surface roughening with coarse diamond bur, etching with 40% phosphoric acid solution.^{9,13} In this *in vitro* study, the shear bond strength of conventional GICs and two commonly used resin luting cements to IPS Empress 2 all ceramic and dentin was evaluated. Shear loading was performed using universal testing machine and maximum shear load at the point of failure was recorded. Shear bond strength were calculated by dividing the force at which the bond failure occurred by the specimen bonding area.⁶ The results obtained were then statistically analyzed by one-way ANOVA. The testing was performed at a significant level of $p = 0.05$.

Maximum bond strength was obtained for group C specimens luted with Variolink II followed by group B specimens luted with RelyX ARC. Increased bond strength of Variolink II resin luting cement can be attributed to higher filler content of the cement compared with other cements.¹⁴ Another possible explanation for the increased bond strength would be the presence of urethane dimethacrylate (UEDMA) in its composition. This monomer is more flexible than bis-GMA because of urethane linkages and presents lower viscosity which facilitates the migration of free radicals, increasing the degree of crosslinking which in turn results in better adhesion and increased bond strength.¹⁴ This is in accordance with studies done by Asmussen, Peutzfeldt (1998).¹⁵ Andree Piwowarczyk et al determined the shear bond strength of various cementing

agents to IPS Empress 2 and concluded that Variolink II exhibited maximum bond strength values.¹⁶ Bond quality, however, should not be assessed on strength data alone, because the mode of failure is also important. This information may yield predictions of clinical performances.¹⁶ Failure analysis through SEM examination revealed predominantly cohesive failures at the resin-dentin and ceramic-resin interfaces for both Variolink II and RelyX ARC luting cement in accordance with studies done by Janda (2002),¹⁷ Mutlu Ozcan (2001).¹⁸

These resin cements form a hybrid layer which is a molecular level mixture of collagen and resin polymers. It is formed by the diffusion of monomers that have been placed on the conditioned dentinal surface and subsequently polymerized *in situ* (Nakabayashi 1982).¹⁹ Bond strength of both group A was found to be inferior to that of both groups B and C. Although conventional GICs has many advantages to its merit, lower bond strength was reported when compared to resin cements. On SEM examination, GICs exhibited cohesive failures at cement–dentin interface. This is due to the formation of chemical bond to tooth tissue by reaction with the calcium salts in the tooth structure.⁷ But adhesive failures were predominant at cement/ceramic interface due to lack of chemical and mechanical union between GIC and ceramic surface. This *in vitro* study allowed an immediate assessment of the bond created between the cement and the restorative material. It is admissible, to compare the measured *in vitro* results obtained in a controlled environment. However, these tests cannot adequately simulate clinical situations in every detail. The final evaluation of material performance should be determined using long-term clinical studies.

SUMMARY AND CONCLUSION

With the limitations of this study, it has been concluded that:

1. Maximum shear bond strength values were obtained for Variolink II resin luting cement followed by RelyX ARC luting cement. GIC showed least bond strength values.
2. On SEM examination, the mode of failures seen was predominantly cohesive for both Variolink II and RelyX ARC resin luting cements at resin-dentin and resin-ceramic interface suggesting improved bond strength.
3. GIC showed cohesive failures at dentin-cement interfaces. However, adhesive failures were predominant at cement-ceramic interface suggesting inadequate bond strength with the ceramic surface.

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