# A COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF THREE PRIMER/RESIN CEMENT SYSTEMS TO MONOLITHIC ZIRCONIA UNDER DIFFERENT STORAGE CONDITIONS: AN IN VITRO STUDY

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**Abstract**

**Purpose:** The aim of study is to compare the shear bond strength of various primer/resin cement systems to monolithic Zirconium under different storage conditions.

**Materials and Methods:** 30 disk specimens of monolithic zirconia (10 mm x 3mm) were prepared with CAD/CAM and polished with silicon carbide and bonding surface were sandblasted with aluminum oxide. Samples were divided into three groups according to the type of cement used: Duo Link Composite Luting Cement, Rely X Ultimate Adhesive Resin Cement, Panavia SA Cement Plus, and further divided into two group each based on the storage conditions into Short Term Storage and Long Term Storage. The specimen were embedded in acrylic mould and SBS test were performed. The data were analysed by non-parametric Kruskal Wallis and Mann-Whitney U. significance was set at $P<0.05$.

**Result:** The highest and lowest SBS values were obtained by kurary system (16.78) and Bisco (8.22) respectively in short term storage. Similarly in long term storage group Kurary (13.7) has highest and bisco (5.2) has lowest SBS value.

**Conclusion:** Long term storage conditions decreases the Shear bond strength of monolithic zirconia to all resin cement systems. The highest bond strength was obtained when both the primer and resin cement contain MDP in their composition.

**Introduction:** In recent years, the increase in esthetic demands of patients has resulted in a more widespread use of all-ceramic restorations in a larger number of clinical situations. A recent surge of interest in metal free restorations has prompted the use of Zirconium with high mechanical properties, good biocompatibility and adequate aesthetics [1]. Clinical failure of Zirconium restorations is mostly related to chipping and fracture of the ceramic veneer [2].

There are various systems available for the preparation of ceramic restorations using CAD/CAM technology. With advancement in CAD/CAM technology, a full contour Zirconium restoration called monolithic Zirconium has been introduced to eliminate veneer cracking. Monolithic Zirconium has a polycrystalline structure and lacks silica in its composition. Monolithic Zirconium has been indicated for patient with limited inter-occlusal space because of its ability to resist occlusal loads and patients with an unfavorable occlusion, with parafunctional habits or fracture history. The loss of retention because of shorter abutments for patients with limited inter-occlusal space may also be common cause of fixed restoration failures[2]. Earlier, conventional cements have been commercially used for luting Zirconium restorations. Now, adhesive cementation has been shown to increase the fracture resistance and fatigue resistance and improve longevity of ceramics restorations. In addition, resin cements offer the advantage of sealing minor internal surface flaws created by acid etching or air borne particle abrasion, which significantly strengthens ceramic materials.

The minimal in built mechanical retention in CAD/CAM-milled Zirconium restorations shows the need for a stronger cement with a higher retention rate[3]. Air abrasion and Zirconium oxide dedicated primers has also been shown to promote the interaction between Zirconium and luting substrate, thus improving retention. Air abrasion with alumina oxide particles aim to roughen the internal surface of restoration helps to optimize adhesion area and promote better mechanical interlocking with resin cement[4]. Artificial aging is indicated to simulate the conditions of the oral environment in laboratory studies is well established. Long-term water storage, thermocycling, or a combination of both are the most common methods[5]. There are several studies that have investigated the bond strength of different resin cement system in both simulated (thermocycling) and 3 unsimulated intraoral conditions. This study examined the bond strength under simulated conditions. However, thermocycling is substituted with long term storage in water.

Hence the purpose of this study is to evaluate the shear bond strength of three different types of primer/resin cement systems to monolithic Zirconia with short term storage and long term storage simulating thermo cycling conditions.

**Materials and Methods:**

A. **Fabrication of Samples by CAD/CAM System:** Monolithic zirconia samples were tested in this study. Samples were prepared from pre sintered blocks using CAD/CAM system and Samples were then sintered in a special high temperature sintering furnace. Specimen dimesions were measured with vernier calliper. In total, 30 disk shaped test specimens was ultrasonically cleaned in distilled water for 10 minutes. The 30 sintered ceramic samples, diameter 10 mm and thickness 3 mm, were divided into three groups according to the type of cement used and further divided into two group each based on the storage conditions.
B. **Surface Treatments:** The bonding surface of monolithic zirconia specimens were polished consecutively with 320, 600, 800, 1200 grit silicon carbide papers under water cooling. Then, the airborne particle abrasion were applied with a machine on bonding surface with alumina oxide. Disc specimens was ultrasonically cleaned in distilled water for 3 minutes.
C. **Application of Resin Cement:** Monolithic zirconia disc were divided into three main group based on type of resin cement used. Then a plexiglass mould is placed on

monolithic zirconia specimen with dimension of 5mm in diameter and 3 mm in height. First, primer agent of each system was applied on to the bonding surface. Then, resin cement was applied on the bonding surface until the mold is completely filled with the material. Resin cement was then light polymerised for 20 sec with curing light.

D. **Storage:** Specimens were then stored in the different storage conditions. To simulate the oral condition specimens were stored for Short term storage that is in distilled water for 24 hours at 370 C and Long term storage that is in distilled water 63 days at 370C.

E. **Sample Testing:** After short term and long term storage procedure is completed, all specimens were then embedded in chemically cured acrylic resin block. The shear bond strength was determined with the help of universal testing machine after storage period. Knife-edge blade with the crosshead speed of 1mm/minute was placed parallel to the direction of force applied at the interface between the monolithic zirconia and resin cement.

**Result and Observation: Table 1:** Comparison of Mean Shear Bond Strength among primer/resin cement in Short Term Storage.

The non-parametric Kruskal Wallis test was applied which shows that the mean rank of share bond strength of three different cements was statistically significant. ($P<0.05$).

The significantly higher mean rank 13.0 with median value 16.78 was for Kurary short term group, followed by mean rank 8.0 with median 12.92 for 3M short term group and significantly lowest mean rank 3.0 with median 8.22 was for Bisco short term group.

**Table 2:** Comparison of Mean Shear Bond Strength among primer/resin cement in Long term storage. The non-parametric Kruskal Wallis test was applied which shows that the mean rank of share bond strength of three different cements was statistically significant. ($P<0.05$).

The significantly higher mean rank 13.0 with median value 13.7 was for Kurary long term group, followed by mean rank 7.6 with median 6.8 for 3M long term group and significantly lowest mean rank 3.4 with median 5.2 was for Bisco long term group.

**Table 1: Comparison of Mean Shear Bond Strength among primer/resin cement in Short Term Storage**

| Variable | Group Short Term | N | Mean Rank | Median | Kruskal-Wallis H | |
|---|---|---|---|---|---|---|
| Shear Bond Strength | Bisco Short Term | 5 | 3.00 | 8.22 | Value | 12.500 |
| | 3M Short Term | 5 | 8.00 | 12.92 | df | 2 |
| | Kurary Short Term | 5 | 13.00 | 16.78 | P Value | 0.002 |
| | Total | 15 | Result | | Significant | |

**Table 2: Comparison of Mean Shear Bond Strength among primer/resin cement in Long term storage**

| Variable | Group Long Term | N | Mean Rank | Median | Kruskal-Wallis H | |
|---|---|---|---|---|---|---|
| Shear Bond Strength | Bisco Long Term | 5 | 3.40 | 5.2 | Value | 11.580 |
| | 3M Long Term | 5 | 7.60 | 6.8 | df | 2 |
| | Kurary Long Term | 5 | 13.00 | 13.7 | P Value | 0.003 |
| | Total | 15 | Result | | Significant | |

**Discussion:** Since decades, fixed dental prostheses (FDPs) are the treatment option to restore function and aesthetics, after loss of a single tooth when implants cannot be placed due to anatomic restrictions[28]. With the introduction of advanced computer-aided manufacturing/computer- aided design (CAD/CAM) technologies various high-strength ceramic materials have evolved and are increasingly becoming popular as anterior and posterior FDP situations.[29,30]. Conventional cementation or bonding techniques used with $ZrO_2$ components do not provide sufficient bond strength for many of these applications. Bonding to traditional silica-based ceramics, generally using both mechanical and adhesive retentions, has been studied, and bond strengths are predictable. A strong resin bond is based on chemical adhesion between the cement and ceramic (by way of silane chemistry), and on micromechanical interlocking created by surface roughening. The most commonly used alternative for roughening the surface of $ZrO_2$ to improve mechanical bonding is surface grinding. Retention of the restoration to tooth structure and sealing of the marginal gap between the tooth and restoration are dependent on the ability of luting agent to bond to the surface of the ceramic.[23] Zirconium surface is covered with a passive film of Zirconium oxide. As chemical characteristics of zirconia ceramic surface are similar to those of metal surface, hydroxyl (O–H) groups may be present on the zirconia ceramic surface. It is also likely that a luting cement containing any polymers or monomers with polar functional groups is capable of bonding chemically with polar hydroxyl groups on the zirconia ceramic surface.(examples of cement) Therefore, all the luting cements tested in this study were expected to have the potential to bond chemically to zirconia ceramic due to their respective functional polymers or monomers [e.g. 2- hydroxyethyl methacrylate (HEMA), carboxylic acid monomer, and an organophosphate ester monomer such as 10-MDP.[22] All tested group had surface pretreatment with MDP containing primer and additional MDP in resin cement used, thought to enhance the bond strength between zirconia and resin cement. Studies prove that the ester group of MDP directly bonds to metal oxides. When phosphate monomer is applied to Zirconium, the hydrogen group of phosphate monomer and the oxygen group of zirconia slowly react to produce water molecules and to form a stable Zr-O-P covalent bond [1]. In the early stage of reaction there must be sufficient wettability to use the hydrophilic property, but excessive hydrophilicity can cause swelling that can adversely affect dimensional stability and mechanical strength, thus increasing hydrophobicity need after the initial reaction. Short and long term water storage methods has been used to simulate the intra oral conditions. Storage methods are employed as common technique to test materials in invitro studies to show their suitability in invivo situations. The result of this invitro study are in favour of the previous study. Kim et al evaluated that zirconia bonding was improved when MDP containg primer was applied to the surface.[22] De oyague reported that MDP containg resin system was best for bonding of zirconia. A study by Piwowarczyk et al demonstrated that cement containg MDP showed more stable adhesion to zirconia.[13] Similarly luthy et al claimed that all cement lost their shear bond after aging except cement containing MDP.[14] MDP is a hydrophobic monomer as it has a 10-carbon chain and two terminal ends – a polymerizable methacrylate that adheres to the resin cement and a hydrophilic phosphate that adheres to the zirconia surface chemically .Functional phosphate monomer works as a mediator between inorganic and organic substrate and contain an organofunctional group.[15] Organofunctional group first react with organic matrix of resin cement one one side and with the phosphate ester group on the other side.[6] The bond is achieved when the functional ester group and the hydroxyl reacts with surface oxide. Phosphate monomer of PANAVIA SA cement of kurary system has a terminal end hydroxyl group, which provide hydrolytic stability under water and acidic condition.[2] This shows why highest bond strength is seen with Kurary system. Methacrylate monomer in 3M cement system makes primary bond with methacrylate resin in the primer and thus improve the bond strength. The 3M cement system showed better bond strength than BISCO system, which is a conventional bis-GMA resin cement.[36] The exact percentage of MDP monomer in all primers used in the study is not known. Clearfil ceramic primer plus of kurary system contain MDP, ethano l,3- methacryloxypropyltrimethoxysilane. Blatz et al claimed that MDP containing primer increases the durability and reliability of resin bond.[11] Two monomers (organophosphate and carboxylic) of Z prime Plus makes it compatible to be used with various resin cement. A more consistent bond is seen with Single bond universal adhesive primer at different moisture level.[1] The minimum bond strength requirement is assumed to be approximately 10-13 MPa. Therefore, in accordance with the study, Kurary system has acceptable Shear Bond Strength value that can be used for clinical application as shown in table 1 and table 5. Short and long term water storage methods has been used to simulate the intra oral conditions. [26] Storage methods are employed as common technique to test materials in in-vitro studies to show their suitability in in-vivo situations. This study demonstrated (table 9, 10,11) that the shear bond strength values decreased significantly after being subjected to long term storage. There are limitations to this in vitro study. This study was performed in laboratory environment without contaiminated specimens and oral moisture conditions which can affect the clinical application. The bond strength of resin cement to zirconia was sensitive to chemical and mechanical influences in the oral cavity. Another limitation was that only single brand of monolithic zirconia was tested in this study. Long term storage is used instead of thermocycling and the storage medium used was distilled water and thus no saliva was used. SBS test has a disadvantage of non uniform stress distribution. The lack of this factor may be a limitation in our study, but can be investigated in the future by in vivo studies.

**Conclusion:** Within the limitations of the present study, it may be concluded that in both long term and short term storage conditions kurary system had the highest Shear Bond Strength value and Long term storage conditions decreases the Shear bond strength of monolithic zirconia to all resin cement systems. The highest bond strength was obtained when both the primer and resin cement contain MDP in their composition.

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