# Comparative evaluation of the influence of surface modifications and two different resin cements on the bond strength of monolithic zirconia - an In vitro study

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## ABSTRACT

**Introduction:** Monolithic zirconia led to increase its indications for minimally invasive aesthetic restorations, therefore, it is important to test its adhesive potentiality.

**Objectives:** Investigation of shear bond strength (SBS) of monolithic zirconia after different surface treatments and examination of their effect on surface topography and comparing two resin cements.

**Materials and methods**: 40 zirconia discs, (Ø10mm×2mm height) were divided into 4 groups (n=10): Sandblasted discs : discs were sandblasted by 110µm Al2O3 particles for 10 seconds, at 60 psi, from 10mm distance, and $CO_2$ Laser irradiated discs : discs were irradiated with energy of 21.2 mJ was delivered in non contact mode with a wavelength of 10.6 µm at power setting of 4 Watt, duration 5.3 ms, interval 1 ms, distance 0.8mm, normal scan mode for 20 secs. Two different resin cements were used to lute the specimens thus four groups were made.

**Results:** Morphological examination of (C) showed the highest load values were obtained in Group A – Sandblast with RMGIC (3.15 ± 0.15) followed by Group C – Laser with RMGIC (2.42 ± 0.17), next comes Group B – Sandblast with SARC (1.9 ± 0.20), whereas least load values were observed in Group D – Laser with SARC (1.9 ± 0.15).

**Conclusions :** In the current study after the two different surface modifications were performed and analysed its effects on the bond strength between two resin cements and monolithic zirconia the mean and standard deviation values of maximum load and shear bond strength showed that highest values were obtained for Group A – Sandblast with RMGIC followed by Group C – Laser with RMGIC, next comes Group B – Sandblast with SARC, whereas least load values were observed in Group D – Laser with SARC. After analysing all the samples it is observed that mostly bond failure occurs at adhesive in comparison to cohesive and mixed bond failure.

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## INTRODUCTION

Nowadays, Monolithic zirconia is emerging as a promising option for fixed prosthodontics because of their high flexural strength, natural tooth colour, less wear on the antagonists, and minimum tooth preparation. For the patients with compromised occlusion or parafunction, monolithic zirconia crowns may be fabricated with as little as 0.5 mm of occlusal reduction. It is possible to produce CAD-CAM milled monolithic zirconia restorations with the new digital impression technology.[1]In spite of the advantages of monolithic zirconia restorations, bonding between adhesive resins and oxide ceramics still presents a challenge compared to that between adhesive resins and glassy matrix ceramics. Various surface treatments have been suggested for resin bonding to zirconia.[2]Cementation of ceramic restorations with the resin-based materials can improve their marginal adaptation and bonding efficacy to the tooth, most of the resin-based cements are used for this purpose.[3]To create a strong bond between a resin and ceramic, mechanical and chemical retention are needed. Roughening and cleaning the ceramic surface have been reported to achieve bond strength. MDP - containing resin cements have acidic monomers which rapidly hydrolyze silane coupling agents, producing the siloxane bonds necessary for chemical bonding.[4] However, zirconia has high crystalline content without silica phase which is resistant to the hydrofluoric acid etching and silane treatment. Due to these reasons, alternative techniques such as Laser irradiation, silica coating, sandblasting, and selective infiltration have been introduced for the treatment of the zirconia surfaces to be bonded to the resin-based cements.[5]Therefore, other surface treatment methods such as air abrasion with $Al_2O_3$ or silica coating with silica modified $Al_2O_3$ particles are frequently employed for Zirconia restorations. In addition to currently used ceramic conditioning methods, the laser is used in clinical dentistry applications such as the removal of carious dentin cavity preparation and surface treatment of indirect restorations. Macroscopic and microscopic irregularities remaining on the surface after laser applications may play an important role in bonding.[6]

On this basis, aim of the present study is to evaluate the shear bond strength (SBS) of an MDP-based Self adhesive resin cement and MDP-based Resin modified glass ionomer cement to a Monolithic Zirconia. Ourresearch hypotheses wasthat the cement type and the chemical/crystallographic structure of zirconia would not behave as significant factors or able to significantly influence the shear bond strength to the zirconia substrate.

## MATERIAL & METHODS

1. *Fabrication of Zirconia discs*:

The designedand milled zirconia disc using EXO CAD software was 10 x 10 x 2 mm.

**Fig. 1 - Zirconia samples**

2. *Fabrication of customized metal jig:*

After milling of zirconia discs, it was necessary to fix them into self cure acrylic resin block to accomplish surface treatment, lute the resin cements and subsequent shear bond testing. Therefore, a customized metal die was designed to standardized placement of zirconia sample on acrylic blocks

**Fig. 2 Customized metal jig**

3. *Fixation of milled Zirconia sample on auto polymerizing acrylic resin blocks:*

The split metal mould was assembled auto polymerizing acrylic resin monomer and polymer were mixed according to manufacturer's instructions and poured into this mould.Zirconia disc was oriented on the top of the mould and secured while the acrylic resin when still in dough stage. Mould was separated and acrylic resin block with the embedded zirconia disc was retrieved.

4. *Surface treatments:*

The surface treatments were carried out after fixation of zirconia samples in acrylic resin, as no self cure acrylic contamination must be done to the surface treated samples.

The specimens were divided into two major groups.

Group I - Sandblasted $AL_2O_3$. Group II - LASER .

Group I :- 20 samples from Group I were air abraded with 110µ aluminium oxide particles at 60 psi for 10 seconds maintaining 10 mm distance of zirconia surface to blaster tip.

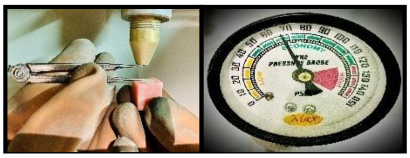


**Fig.3 – sandblasting Parameters**

Group II:- 20 samples of Group II were irradiated with CO2 laser. A $CO_2$ Laser (Rosch) energy of 21.2 mJ was delivered in non contact mode with a wavelength of 10.6 µm at power setting of 4 Watt, duration 5.3 ms, interval 1 ms, distance 0.8mm, normal scan mode for 20 secs.

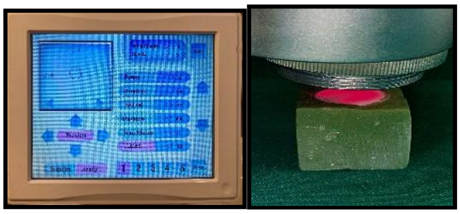


**Fig. 4– Laserparameters& irradiation**

5. *Luting of resin cements over surface treated zirconia samples:*

After surface treatments, cleaning and air drying of all the specimens were done. Each major group was further divided into two subgroups with respect to resin cements. To maintain uniform thickness of the cement two polyether sheets were used of thickness 100µ then constant load 50N was placed and light cured for 40 seconds. All specimens were cemented following the manufacturer's guidelines and in the same manner.

Group A - Zirconia sandblasted with $AL_2O_3$ and luted with Resin modified glass ionomer cement.

Group B - Zirconia sandblasted with $AL_2O_3$ and luted with Self adhesive resin cement.

Group C - Zirconia irradiated with $CO_2$ LASER and luted with Resin modified glass ionomer cement.

Group D - Zirconia irradiated with $CO_2$ LASER and luted with Self adhesive resin cement.

6. *Storage:*

The specimens were washed with an air - water spray and were then stored in distilled water at 37° C for 24 h before the shear bond strength test.

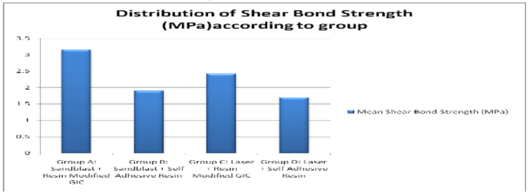
7. *Testing of Specimens:*

*(a) Quantitative assessment:* Measurement of shear bond strength using Universal Testing Machine. The shear bond strength between zirconia and resin cement for each sample was measured, calculated and tabulated, following which, statistical analysis was performed.

*(b) Qualitative assessment:* Stereomicroscope was used to observe the modes of bond failure, after the testing of shear bond strength. This test was carried out in Praj Dental lab, Pune.

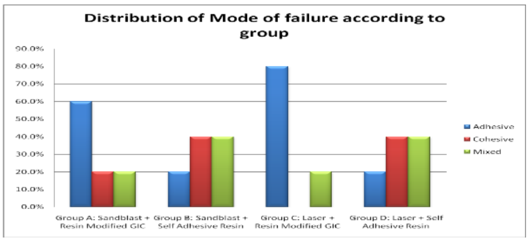
## RESULTS

After testing of the specimens, the surface of the zirconia specimen was studied under stereomicroscope. Stereo microscopes use reflected light from the object being studied, compared to the transmitted light that is used by compound light microscopes. Magnification ranges from 7.5 to 75x.The values of mean and standard deviation values of Shear bond strength (SBS) applied on different groups. They ranged from 1.9 ± 0.15 to 3.15 ± 0.15. The highest load values were obtained in Group A – Sandblast with RMGIC (3.15 ± 0.15) followed by Group C – Laser with RMGIC (2.42 ± 0.17), next comes Group B – Sandblast with SARC (1.9 ± 0.20), whereas least load values were observed in Group D – Laser with SARC (1.9 ± 0.15).



**Graph - 1: Distribution of Shear bond strength (mpa)**

The mode of failures occurred among all group. Among all the samples (40) maximum adhesive bond failures are observed (18/40) followed by mixed bond failures (12/40) and least occurred are cohesive bond failures (10/40). In Group A maximum adhesive followed by similar cohesive and mixed bond failures are observed. In Group B similar cohesive and mixed bond failures are observed followed by adhesive bond failures. In Group C Adhesive failures occur followed by mixed bond failures and no cohesive bond failure is seen. In Group D similar cohesive and mixed bond failures are observed followed by adhesive bond failures.



**Graph - 2 : Distribution of mode of failure**

## DISCUSSION

Prosthodontic treatments have traditionally sought to restore lost function (chewing, speech, swallowing), while providing aesthetics that fulfil contemporary criteria for attractiveness at the present time the term ‘aesthetic restoration’ refers to ceramic restorations and in particular to porcelain restorations without any metal. Towards the end of the last century, a climate of non-acceptance of metal alloys in the mouth emerged among some dentists and in the dental product industry and given the increasing demand for aesthetic treatments.[7] The use of metal–ceramic restorations has been declining in favour of ceramic prostheses, mainly for aesthetic and biocompatibility reasons.[8] On the other hand, ceramics are fragile and brittle in nature. Several techniques have been developed to resolve those problems thus introduced Zirconia. Tremendous progress has been made in terms of mechanical performance, with a ten-fold increase in flexural strength and fracture toughness. Common important characteristics of all-ceramic systems, such as the proportion of glassy phase and amount of porosity, both influence optical and mechanical properties which involve an increase in the zirconia translucency and colour.[9] Chipping has been reported to be a major complication of zirconia based restorations to overcome this problem; the monolithic zirconia has been developed.[10] Compared to other ceramic materials, the use of monolithic Zirconia often dramatically reduces mechanical complications and the need for the preparation of much of the tooth structure and thereby leads to a prosthetic restoration that retains as much of the structure as possible. Monolithic Zirconia used for single crowns has a high survival rate.[11] Dental Zirconia has traditionally been manufactured mainly from tetragonal Zirconia crystals with a minor proportion of Yttria stabilizer (3Y-TZP); this type is extremely strong but has low translucency.[12] Y-TZP Zirconia has a unique characteristic that can transform from tetragonal to monolithic phase under mechanical stresses, resulting in extra toughness and strength and hindering crack propagation by 3% – 5% volume expansion during phase transformation.[13] Polished monolithic Zirconia restorations show decreased wear of their own surface and that of antagonists.[14] In a clinical study, monolithic translucent Zirconia crowns produced more occlusal wear than natural enamel but generated less wear in enamel than other ceramic restorative materials. Polished monolithic translucent Zirconia showed a lower wear rate on the opposing enamel.[15] With the development of computer-aided design computer-aided manufacturing (CAD-CAM) technology, the design and production of zirconia frameworks could be achieved using a digital process. Therefore, restorations using a zirconia framework became more practical.[16] Despite its robust mechanical properties, zirconia crowns clinically suffer from interfacial failures.[17] Zirconia still faces the problem of achieving the durable adhesion to resin cements essential to minimally invasive dentistry due to its lack of an amorphous silica glass phase, in fact, it is immune to the acidic etching solutions and the silane coupling agents conventionally used to increase the surface energy of ceramic substrates and improve adhesion to resin cements.[18] Both traditional acid-based cements like zinc phosphate, conventional and modified glass ionomer and composite resin cements have been extensively used with zirconia since its introduction. Due to its excellent mechanical performance, zirconia can be luted to dental tissues using conventional cements. Despite some practical advantages (no need of dental tissue conditioning, moisture tolerance and simple handling), however, these materials are not able to form a lasting bond to dental hard tissues mainly because of their hydrophilic properties, which can determine retention loss especially after aging.[19] Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a reliable bond, which has to integrate all parts of the system into one coherent structure.[20] Resin cements show higher optical features, lower solubility, and better mechanical properties (higher compressive and tensile strength, toughness, and resilience)[21], which enhance retention, marginal sealing,[22] and fracture strength of restorations[23], due to these reasons resin cements represent nowadays the most reliable choice for cementation of zirconia - fixed prosthesis, so that manufacturers extend clinical indications of 5Y-PSZ even to partial adhesive restorations like veneers and inlays/onlays. Containing acidic functional monomers, modern resin cements do not require pre-treatments and are therefore easy to manage.[24] According to the chemical nature of their acidic group, current self-adhesive resin cements can be categorized as luting agents with multi-function methacrylates (e.g. phosphoric acid esters, carbonic acid, or amino acid derivatives) and luting agents with 10 - methacryloyloxy-decyl dihydrogen phosphate (MDP).[25] MDP containing luting system seems to be the most convenient to adhesively bond zirconia restorations.[26] Self-adhesive resin cements are advocated for direct bonding on substrates, as the retention is provided by specific adhesive functional monomers (i.e. 10-MDP) and/or silanes contained within the formulation of such materials.[27] They represent the most innovative simplified class of resin cements, which rapidly reached a great popularity due to their user-friendly clinical uses. Indeed, substrate pre-treatments through acid etching and/or application of adhesive primers would not be necessary anymore.[28] Resin bonding between a tooth and a restoration is advocated for improving the retention, marginal adaptation, fracture resistance and bond strength of restorations.[29]Obtaining adhesion between a luting agent and a ceramic surface requires surface pre-treatment.[30] They are commonly caused by the weak interfacial strength between the cement and ceramic crowns that cannot tolerate the mechanical stresses in the oral environment.[31] There are different methods to improve the interfacial adhesion, and the purpose is to increase the surface area and decrease the stress levels at the interfaces.[32] Several attempts have been made to develop micromechanical and chemical retention in order to ensure reliable and predictable bonding strength to dental oxide ceramic restorations in the clinical situation. A variety of mechanical techniques have been suggested for surface modification of oxide ceramics including grinding/polishing, air-borne particle abrasion with aluminum oxide particles, tribochemicalsilicondioxide (silica) coating, and Laser conditioning.[33] The techniques are mainly aimed to roughen the cementation surface of the ceramic, which leads to increased surface area and micromechanical interlocking, thereby improving the bond strength. Furthermore, the use of resin cements or primers containing 10- methacryloxydecyl dihydrogen phosphate (MDP) has been shown to enhance the strength of the adhesion by providing chemical retention to zirconia, although the durability of the chemical bond may be compromised in the clinical conditions.[34] Airborne particle abrasion with $Al_2O_3$ abrasive particles has been identified as an effective means of achieving a stable, durable bond for alumina- and zirconia based ceramics.[35]Kim et. al. performed a study and suggested that the recommended $Al_2O_3$ particle sizes under given pressure, time, distance, and impact angle (0.2 MPa, 10 s/cm2 , 10 mm, and 90◦ , respectively) were 110 μm sand for Yttria stabilized Zirconia.[36] Different sizes of abrasive $Al_2O_3$ particles, between 50 and 110 μm, are generally used.[37] However, differences in the size of particles and the application time may induce discrepancies in the achieved results: excessively high pressure during blasting may initiate phase transition, and expedite the formation of micro-cracks, thus reducing the mechanical properties of zirconia.[38]Jevinkar P et. al. used 110-μm alumina particle for sandblasting and found an increase in the cement bond strength to zirconia.[39] Su et. al conducted a study and proposed that sandblasting with alumina particles at 0.2 MPa, 21 seconds and the powder size of 110 μm is recommended for dental applications to improve the bonding between zirconia core and indirect composite resin.[40] He et. al. (2 bar blasting pressure and 1 cm working distance)[41] and Abi-Rached et al. (2.8 bar blasting pressure and 1 cm working distance) their resultant surface roughness proved to be much higher.[42] Kwon et. al conducted an experiment to study the effect of four different sandblasting conditions (with 50 and 110 μm alumina at pressures of 0.2 and 0.4 MPa) on the bonding of adhesive resin cement and concluded that mild sandblasting of zirconia with small-sized abrasives and reduced blasting pressure is preferred to an aggressive procedure because increased surface roughness and residual stress do not directly affect the resin bonding durability.[43] The general consensus among researchers and opinion leaders is that traditional high strength zirconia (3-4 mol% yttria concentration) can be safely and effectively sandblasted with 50-110μ aluminous oxide (Al2O3) using a blast pressure of 1.5-4 bar (approximately 20- 60 psi) from a distance of 1-2 cm and for a duration of 10-20 seconds.[44] Ravi Kiran Chintapalli in 2013 did a study on effect of different sandblasting conditions on the mechanical properties of yttrium stabilized tetragonal Zirconia (Y-TZP). The specimens were sandblasted considering two different particle sizes (110,250μm). He concluded that Sandblasting with particles sizes equal or less than 110 μm and pressures less than 4bar increase the bi-axial strength of the Zirconia ceramics.[45]Previous studies employed different lasers such as neodymium-doped yttrium aluminium garnet (Nd:YAG) , neodymium-doped yttrium aluminium perovskite (Nd:YAP) , erbium-doped yttrium aluminum garnet (Er:YAG) and carbon dioxide ($CO_2$) for surface modification of zirconia ceramic, and reported varying degrees of success. The $CO_2$ laser is generally employed for intraoral soft tissue surgery because of its great absorption in water. The wavelength of the $CO_2$ laser (10.600 microm) is also well absorbed by ceramic materials, making it a suitable instrument for ceramic surface treatment.[46]Cagriural in 2010 compared the effects of various surface treatments and Laser irradiation on the shear bond strength of resin cement to Zirconia based ceramic. 40 Zirconia core specimens were made and divided as Group C is no treatment applied (control); Group SB is considered as bonding surfaces of ceramic disks were airborne particle-abraded with 110um alumina oxide particles; Group HF refers bonding surfaces of ceramic disks were etched with 9.6% hydrofluoric acid; and Group L states bonding surfaces of ceramic disks were irradiated by a CO2 Laser. A total of 40 composite resin disks were cemented with an adhesive resin cement to the specimen surfaces. He concluded that CO2 Laser etching is an effective method for conditioning Zirconia surfaces, enhancing micromechanical retention and improves the bond strength of resin cement on Zirconia ceramic.[47]

In the present study we have advocated some surface treatments that are readily available to clinicians without the need of a complex laboratory setup. The surface treatments used in the study are Sandblasting unit using Al2O3 particles and CO2 Laser. The above surface treatments are accessible to clinicians and are not technique sensitive. Therefore they can be skillfully performed in a clinic set-up. However there are some combination of surface treatments and resin cements which have been never been evaluated and compared for increasing the shear bond strength of fixed zirconia prosthesis. The zirconia discs were surface treated using different parameters and luted with resin cements. The samples were subjected to shear loading. The load at which the bond failure occurred was recorded for each sample. The data obtained for subjected to statistical analysis. The values of mean and standard deviation of Shear bond strength (SBS) applied on different groups. They ranged from 1.9 ± .15 to 3.15 ± .15. The highest load values were obtained in Group A – Sandblast with RMGIC (3.15 ± .15) followed by Group C – Laser with RMGIC (2.42 ± .17), next comes Group B – Sandblast with SAR (1.9 ± .20), whereas least load values were observed in Group D – Laser with SAR (1.9 ± .15). Using one-way analysis (ANOVA), $P< 0.051$ was determined which showed significant difference in post hoc test. The statistical analysis showed significant difference between the groups A,B,C & D. All groups were statistical to other groups.

The purpose of various pre-treatments on the zirconia is to obtain better bond strength results in the Shear Bond Strength test, which will then result in longevity of the prosthesis. Sandblasting remains the easiest and most popular technique to improve the material surface and continues to be studied in the literature. Several studies evaluated the effect of different surface treatments and their influence on the bond strength, here were compared sandblasting with $CO_2$ Laser on the zirconia surface for the shear bond. It was shown that sandblasting could improve the surface area and create desirable surface irregularities. Among the resin cements Resin modified GIC carries both the properties of resin cement as well as Glass ionomer cement shows better results when compared with self adhesive resin cement. The equipments and the materials used to treat the zirconia surface in this study are readily available and can be arranged in a clinical setup without the need of an advanced laboratory setup. Moreover these treatments can be performed by the clinician himself, without having the dependency on the laboratory technician. Therefore they can be utilized in routine clinical practice. This study opens the horizons for further studies where the surface treated Zirconia prosthesis could be evaluated for interactions with different resin luting agents after different surface treatments. The modes of bond failure were categorized as:Adhesive, Cohesive and mixed type. Adhesive bond failure between zirconia surface and resin cement, this failure occurs between the adhesive and the adhered residual resin cement remaining at any location on one surface only. Cohesive bond failure occurs when upon separation the adhesive remains on both the surfaces. This occurs when the cement is weak for the intended application. Cohesive failure of resin cement is detected if a whole layer of resin cement is attached over the zirconia surface and the other part of the resin cement is separated, showing bond failure within the resin cement itself. Cohesive and Adhesive failure (Mixed failure) exhibits some cohesion and some adhesion failure. The failure exhibits areas of smooth surface as well as areas which are rough. The strength of the bond and the proportion of surface smoothness or roughness depend on the level of degradation of the bond interface. Measuring the bond strength is one method to evaluate the effectiveness of an adhesive system and, consequently, to predict performance in vivo. Tensile and shear tests are mostly used for this purpose. Nonetheless, many studies have reported that the type of fracture occurring after a shear bond test is often cohesive (inside the resin cement) rather than adhesive (at the interface). Cohesive fracturing is rarely observed in clinical bonded restorations. In contrast, in vitro tensile bond testing causes more adhesive fractures, which is more useful for evaluating real bond strength.[48]

Limitations of this in-vitro study were as the surface was kept free from any contaminants to prevent decrease in bonding efficacy.[49] Hence, in clinical try-in stage contamination can't be avoided by blood, saliva, and water or silicone indicator. This may lead to compromised bonding protocol.[50] Another limitation is the not cost effective, requires a Laser set up.

## CONCLUSION

Within the scope and limitation of this study, following conclusion were drawn:

1. Surface treatments showed significant amount of alterations in the bonding surface of zirconia when observed under stereo microscope.
2. Stereomicroscope analysis revealed that 110µm Al2O3 particles and created more surface roughness than Laser group, the highest values of shear bond strength was observed among sandblast group in comparison to Laser group.
3. Resin modified GIC shows more shear bond strength when compared with Self adhesive resin cement.
4. Failure analysis with stereomicroscope showed that adhesive bond failure was most common, followed by mixed bond failure, whereas cohesive bond failure was least observed.

In the current study after the two different surface modifications were performed and analysed its effects on the bond strength between two resin cements and monolithic zirconia the mean and standard deviation values of maximum load and shear bond strength showed that highest values were obtained for Group A – Sandblast with RMGIC followed by Group C – Laser with RMGIC, next comes Group B – Sandblast with SARC, whereas least load values were observed in Group D – Laser with SARC. After analysing all the samples it is observed that mostly bond failure occurs at adhesive in comparison to cohesive and mixed bond failure.

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