

EFFECT OF SURFACE TREATMENT AND MICRO SHEAR PROTOCOL ON BOND STRENGTH TO YTTRIUM-STABILIZED TETRAGONAL ZIRCONIA

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ABSTRACT

Objectives: The purpose of this research was to determine how various surface treatments (sandblasting and air atmospheric plasma pressure) and the micro shear protocol (Polyethylene tube and starch-based mold) affected the bonding strength to yttrium stabilized tetragonal zirconia (IPS e.max ZirCad).

Materials and Methods: 32 zirconia samples were prepared from IPS e.max ZirCad blocks (Ivoclar Vivadent, Liechtenstein) and according to surface treatment and type of mold used to fabricate resin micro cylinders, the ZirCad samples were categorized into four groups (n=8), S.S. group; Sandblasting surface treatment (50 μ m alumina oxide) with Starch mold approach, S.P. group; Sandblasting with Polyethylene tube approach (TYGON S-030-H L Medical Tubing, United states), P.S. group; air atmospheric Plasma pressure (Piezobrush, Relyon plasma GMBH, Germany) with Starch mold approach and P.P. group; air atmospheric Plasma pressure with Polyethylene tube approach. Each IPS e.max ZirCad specimen bonded to five micro-cylinders of resin cement. A universal testing machine (Instron 3345, BOSTON, USA) was used to assess micro-shear bond strength (μ SBS).

Statistical Analysis: The collected results (MPa) from micro shear bond strength tests were subjected to a two-way analysis of variance. (ANOVA)

Results: Revealed statistically significant higher μ SBS values while using the starch-based template protocol. Also, the air atmospheric Plasma pressure surface treatment showed statistically significant higher μ SBS values than sandblasting technique

Conclusion: Air atmospheric Plasma pressure surface treatment showed better micro shear bond strength and Starch based template technique showed more accurate results for the micro shear bonding test.

KEYWORDS: bond strength, surface treatment, alternative tubing, MDP resin cement.

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INTRODUCTION

Recent years have demonstrated an increase in the use of zirconia-based materials due to their biocompatibility, increased flexural strength and aesthetic capability^[1]. When compared to the traditional way, the CAD/CAM technology streamlines the design and fabrication process by cutting down on time and effort as well as the number of steps involved^[2].

The process of bonding to zirconia is not a straightforward one. When it comes to bonded restorations, the region that is most susceptible to failure is the interface between the high crystalline zirconia fitting surface and the resin composite. Many approaches have been tried to enhance this interface chemically and mechanically^[3]. Accordingly, phosphate ester monomer based primer as 10-methacryloyloxydecyl dihydrogen phosphates (10-MDP) based self-adhesive resin cements are recommended for zirconia-based restorations^[4]. 10-MDP has various advantages, including the formation of cross links with the methacrylate groups of the adhesive resin and siloxane connections with the hydroxyl groups of ceramic surface and enhances surface wettability^[4].

On the other hand, other protocols have attempted to raise the surface roughness of zirconia by abrading it with airborne Al₂O₃ particles, then applying self-adhesive resin cement based on 10-MDP^[3,5] or using atmospheric plasma surface treatment which has been suggested as a novel and promising technique. It can be classified according to its parameters^[6]. There are two types of plasma: thermal and cold (non-thermal plasma) with gas temperature near to room temperature at the atmospheric pressure level^[7].

The cold non-thermal protocol is a plasma treatment used for chemical surface alteration, performed at a molecular level, and the bulk performance of the material is not affected^[8,9].

Additionally, plasma can increase surface energy and wettability while removing all surface contaminations^[10]. The cold plasma approach is characterized by the formation of partially ionized gas in a state of non-equilibrium, which results in the production of huge quantities of chemically active species (O₃, NO, OH, H₂O₂ radicals) under low temperatures. These radicals can alter the functional groups at the surface from non-reactive to reactive and thus improve bond strength without affecting the bulk performance of the material^[11,12].

In order to assess and interpret the adhesive system behavior, the bond strength tests are practical and efficacious^[5]. Among several decays, the use of shear and tensile measuring tests was frequent. These tests resulted in a massive number of cohesive fractures that don't express the bond strength precisely. Therefore new method was introduced to precisely examine and measure the adhesive system's bond strength^[13].

In dental adhesion, the selection of a measuring method for the bonding strength of the adhesive system is one that can be debated. Each test has its own features, such as a unique protocol of sample preparation, dissemination of stress, area of adhesion and techniques of loading. These elements are highly identifiable and characteristic of each measuring test^[14,15].

Stress dissemination is an efficacious element in measuring tests. It's better to have smaller bonding areas to decrease the possibility of any interface defect and give better dissemination of stress. Minimal or no failures and premature failure can occur when stress is highly concentrated at the interface with a larger bonding area^[5].

In the micro-shear bonding strength measuring test, a shear force is delivered through a tiny stainless-steel wire^[14]. This method provides several benefits as it can be easily and shortly prepared, fewer teeth and smaller bonding areas to be fixed and can be standardized based on the tube's diameter^[13].

The stress dissemination is more localized at the interface in the micro shear measurement test than in the shear bond strength test ^[14], minimizing the possibility of cohesive failure within the substrate. The micro shear test, on the other hand, has many drawbacks as a marginal gap and possibility of air bubble inclusion that can be overcome by the exclusion of defective specimens to avoid premature failure ^[3,11]. In addition, attaching the conventional polyethylene tube to the material is difficult, particularly after the use of bonding system, resulting in difficulty in composite resin packing ^[15]. It also influences the adhesive interface during the cutting and removal of the tubes ^[16]. In order to develop more reliable results and minimize difficulties during the specimen's fabrication, another type of tube will be evaluated ^[17].

Therefore, the goal of this research is to assess micro-shear bond strength techniques, typical (Polyethylene tube) and alternative (starch-based mold) approaches, and type of surface treatments (sandblasting and air atmospheric plasma pressure) on the bonding strength to yttrium-stabilized tetragonal zirconia (IPS e.max ZirCad).

The null hypothesis is that neither the plasma treatment nor the starch mold would have any effect on the values of the micro-shear bonding strength.

MATERIALS AND METHODS

Samples fabrication: IPS e.max ZirCad blocks (Ivoclar Vivadent, Liechtenstein) were sliced into 32 equal slices, each measuring 12 mm by 14 mm by 2 mm using an Isomet saw (IsoMetwere00 Precision Saw Buehler, United States) under continuous water coolant. Uniform thickness is confirmed by a digital caliper. Zirconia specimens were sintered in an InFire HTC speed furnace (Sirona Dental System, Germany). Each sample was placed inside an acrylic block for easy handling and accurate fixing during testing. Specimens were separated into four groups (n=8) based on the surface treatment of ZirCad

specimens and the type of mold used to create resin micro-cylinders as follows: (S.S.; Sandblasting with Starch mold group), (S.P.; Sandblasting with Polyethylene tube group), (P.S.; air atmospheric Plasma pressure with Starch mold group), and (P.P.; air atmospheric Plasma pressure with Polyethylene tube group).

Half of the ZirCad samples were surface treated with a dental sandblasting device (Renfert, Germany) using 50 μm Al₂O₃ (Hi Aluminas, Shofu Inc, Japan) at two bar pressure for 10 s and 90-degree angulation. A standard distance of one cm, aided by a prefabricated metal frame, was used to hold the acrylic block containing the zirconia samples during the sandblasting procedure to standardize and centralize the distance between sandblasting nozzle and the samples' surface.

The other half of the samples were surface treated for 80 seconds at a maximum power consumption of 30 W and a temperature of 50°C using an Air atmospheric pressure plasma device (Piezobrush, Relyon plasma, Germany) with about 5 mm distance from the treated surface to the nozzle tip. Following the completion of the surface treatment and in advance of the bonding procedures, all samples underwent a 5-minute ultrasonic cleaning in distilled water.

Then, a template made of starch-based plastic was customized to be adapted to the surface of the zirconia samples. Each template had five openings 1 mm diameter and 1 mm depth, acting as molds for the resin cement. Then, a Theracem (Bisco, Chicago, U.S.A, MDP calcium-fluoride releasing self-Adhesive) dual cure resin cement was packed in the starch mold by the supplied auto-mixing and intra-radicular tips. After that it's laminated by a transparent plastic strip and pressed gently against a glass slab. Each sample was left to self-cure for five minutes before being exposed to a light emitting diode output (Emitter D. Shuster Eq, Brazil) for 20 seconds at 900 MW/cm² to activate it, as per the manufacturer's recommendations.

Polyethylene tubing (TYGON S-030-H L Medical Tubing, Saint Gobain Performance Plastics, United States) was used with the rest of the samples. A translucent mold was placed over specimens and filled with resin cement to generate 1 mm diameter and 1mm height resin cement micro cylinders. The bonding procedures were allowed as previously described.

A professional dentist carried out all previous procedures at 24°C room temperature. The specimens were stored in distilled water at 37°C for 24 hours. The tubes were then cut and discarded. Using a blade, the polyethylene tubes were sliced in half, while the starch molds were carefully dissolved under a water stream. To rule out any defective samples, the bonding surfaces of all samples were examined with a StereomMicroscope at a magnification of 10X. (Discovery V20, Zeiss, Germany). Air bubbles, marginal gaps, and other imperfections were ruled out and replaced in the samples.

After the bonding procedure, the μ SBS test was performed with the wire loop approach with a 0.2mm stainless-steel thin wire and was secured to the upper jig of the universal testing machine (EMIC DL 1000: Instron, SJ. Pinhais, Brazil) and then attached to the resin-micro cylinder. The acrylic blocks were installed in the lower jig of the machine. The shear stress was performed at a crosshead speed of 1mm/min and a cell of 1 K.N. till the bonded sample failed. The parallelism between load cell, wire, and resin interface was an important concern, keeping the load application as close as possible to the bond interface.

Statistical Analysis; at a significance level of 5%, all statistical tests were carried out at Origin-Pro 2015 (Origin Lab Co, Northampton, Massachusetts, U.S.). The bond strength results in Mega Pascal were used as the measuring unit for each sample. As a result, the two-way analysis of variance (ANOVA) was utilized after ensuring parametric data distribution (Shapiro Wilk’s test).

RESULTS

The results of the two-way ANOVA exhibited significant differences between the subgroups. μ SBS values were statistically significantly higher with the starch-based mold technique compared to the Polyethylene tube technique ($p \leq 0.05$). (Fig 1) (Table 1)

There was also a significant difference ($p < 0.05$) between different surface treatments. The air atmospheric plasma pressure group had higher μ SBS values (20.22 ± 1.76) than the Sandblasting group (15.12 ± 1.64). (Fig 1) (Table 1)

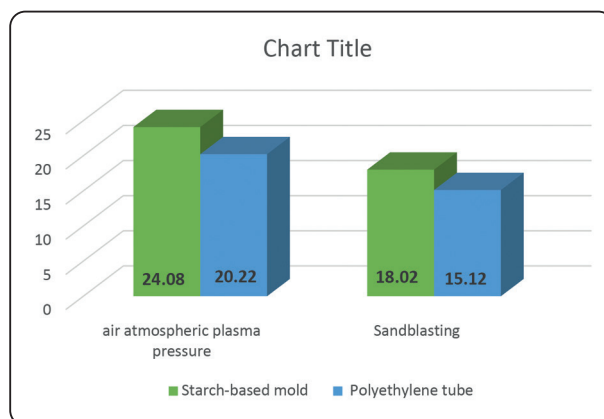


Fig. (1); A bar chart depicting the average μ SBS (MPa) between the various treated surfaces.

TABLE (1) Mean and S.D. of μ SBS (MPa) between different groups

Technique	Plasma	Sandblasting
Starch-based template	24.08±1.5a	18.02±1.78b
Tygon tube	20.22±1.76c	15.12±1.64

Means with different superscript letters are statistically significant ($p \leq 0.05$)

DISCUSSION

The bonding of zirconia and resin cement is critical to the long-term viability of zirconia-based restorations [2]. It has been reported that the inner surface roughening of the restorations increases the bonding surface area, which facilitates the wetting

of the restoration with resin-based materials. Several surface conditioning procedures used to improve the bond strength of zirconia to resin cement, including sandblasting with Al₂O₃, plasma surface treatment, tribo chemical silica coating, laser irradiation, etc. Nonetheless, there has yet to be agreement on the ideal zirconia surface treatment. For this purpose, sandblasting and plasma treatment were applied to IPS e.max ZirCAD^[4].

Sandblasting improves bond strength by roughening the inner surface and increasing this area for bonding^[3]. Zhang et al.^[18] claimed that sandblasting induces micro-cracks on the surface, which decreases the zirconia strength. It was demonstrated, however, that adhesive resin flowed into micro-cracks, quietly strengthening the ceramic^[19].

Plasma treatment has gained popularity in recent years as a unique and promising surface treatment, with evidence that it may enhance zirconia resin bond strength^[7,8]. There are various types of plasma devices on the market, with atmospheric plasma being the most easily clinical technique^[10,11].

Also, the type of adhesive cement affects micro-shear bond strength. Accordingly, the 10-MDP based self-adhesive resin used in the present study (to build up micro-shear resin specimens⁷) is recommended for zirconia-based restorations since the formation of cross links with the methacrylate groups of the adhesive resin and siloxane connections with the hydroxyl groups of ceramic surface thus enhances surface wettability and adhesion^[4].

In the present study, TheraCem was used to fill both tubes with the supplied auto-mixing and intra-radicular tips and laminated by a transparent plastic strip, pressed gently against a glass slab to prevent air inhibiting layer. All tested subgroups were allowed for self-cure for 5 minutes to allow chemical conversion of the resin, followed by light activation using the light-emitting diode with 900 MW/cm² light output for 20 seconds as the manufacturer recommended. The types were left for

24 hours to allow for the complete polymerization of the dual-cured resin cement before cutting or immersion in water^[4].

The micro-shear bond strength test is the most accurate and dependable approach to assess the bond strength without including any additional variables. Connecting multiple micro-cylinders to a single substrate without running the risk of interface errors or faults also makes testing more affordable^[13].

Several studies recommend using the traditional polyethylene tubing to fabricate composite resin samples for the micro shear bond test, and they employed this methodology in their studies^[13,16&17]. The present study evaluates and compares the effect of a starch base mold as a substitute to polyethylene tubing to fabricate micro shear bond test samples.

Since using polyethylene tubing for sample fabrication in previous studies has reported problems^[16,17], Foong et al.^[20] reported that with some adhesive systems, polyethylene tubing couldn't be attached to the substrate, making the composite resin packing difficult. They explained that polyethylene mold could change the air inhibition layer and the adhesive layer thickness, which may affect the bond strength^[18]. Yet, inserting the adhesive resin into starch tubing is much easier, probably because of the inert components of starch tube that prevent any interaction with composite resin material^[20].

Additionally, many studies have reported failures of pre-test samples during the polyethylene tubes removal^[13,16&17]. Tygon tubing increases the development of premature failures of the pre-test samples, as occurred in this study. These results can be hypothesized by the development of stresses during the application of pressure caused by cutting the polyethylene tube with a blade. Applying any stresses prior to the test would adversely influence the actual bond strength at the interface. Contrarily, the starch tubes degraded from the resin specimens without any cutting after being immersed in water which is a favorable and desirable feature in

this approach^[16,17]. It becomes hydrated and then degraded without any stress applied at the interface. An additional advantage promoted by starch base mold is the immersion in water which helps for more water sorption by the resin, to simulate the *in vivo* conditions compared to the partial isolation provided by polyethylene tubing.

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