

**METHODOLOGY FOR TESTING THE MICRO-SHEAR
BOND STRENGTH OF DUAL-CURE RESIN CEMENT
TO PRETREATED ZIRCONIA SURFACE**

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Abstract

The aim of this study was to evaluate the micro-shear bond strength of dual-cure resin cement to zirconia surface pretreated with different techniques. Twenty-four sintered Y-TZP zirconia discs (Kuraray Noritake Dental Inc.) with a diameter and thickness of 11.8 mm and 5.0 mm, respectively, were used for the present study divided into four groups according to the pretreatment procedures: 1) control group without any pretreatment; 2) MDP (10-methacryloyloxydecyl dihydrogen phosphate) primer treated; 3) sandblasted with 50 μm Al_2O_3 at 2 bar, and 4) MDP treated in combination of sandblasting. The micro-shear bond strength was measured by applying an axial load on the bonded interface using a universal testing machine LMT 100 (LAM Technologies, Italy). An original methodology of elaboration of specimens for testing was developed. Statistical analysis was performed with SPSS 11.5 Inc., Chicago, IL, USA, Excel 7.0 VB. One-way analysis of independent samples (ANOVA) was used to analyze the data with the level of significance $p < 0.05$. The Bonferroni's multiple comparison test was used to reduce the instance of false positive results. The impact of surface pre-treatment procedures on bond strength was studied and the best combination was revealed.

Key words: zirconia, cement, micro-shear, MDP, monomers, sandblasting

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Introduction. Zirconia has emerged as a versatile and promising ceramic material because of its superior biological, mechanical and optical properties. Adhesive cementation of zirconia restoration is one of the most important factors for achieving clinical success; it improves retention and marginal adaptation and reduces the possibility of recurrent decay [1]. Considering the chemical inertness of zirconia, a variety of roughening methods has to be applied to promote adequate adhesion between the resin cement and zirconia. The most common method is sandblasting with aluminium oxide (Al_2O_3) particles having different shapes and sizes. The abrasion was applied at various durations and pressures [2]. Also, several coating agents were used to enhance the formation of chemical bonding with zirconia and only those agents that contain a phosphate monomer agent (MDP) were effective in establishing a reliable bond with zirconia materials [3]. One of the most popular tests for measuring bond strength is the micro-shear bond strength test – a method firstly introduced by MCDONOUGH et al. in 2002 [4]. Compared with the conventional shear bond test, the stress distribution is more concentrated at the interface in the micro-shear bond test [5] which reduces the chance of cohesive failure in the material that does not represent the “true” interfacial bond strength.

The aim of the present study was to evaluate the dual-cure cement micro-shear bond strength to zirconia after different techniques of surface pretreatment and select the most appropriate one.

Materials and methods. Surface pretreatment of the specimens. Twenty-four sintered Y-TZP zirconia discs (Kuraray Noritake Dental Inc.) with a diameter of 11.8 mm and thickness of 5.0 mm were used for the present study. They were divided into four groups (six specimens in each group) according to the surface pre-treatment procedure. The methods are listed in Table 1. All of the treatments are either applied separately or in combination with MDP primer (10-methacryloyloxydecyl dihydrogen phosphate). Currently the monomer is the only one that can make a chemical bond with the zirconia surface. In that way in addition to the micromechanical adhesion induced by the surface treatment of the material, a second chemical one is also created.

Original methodology of elaboration of specimens for testing the adhesive bond between zirconia surface and the dual-cure cement. In order to create a stalk from dual-cure resin cement (Panavia V5, Kuraray Noritake Dental Inc.) with a height of 1 mm and a diameter at the base of 1 mm, a special fabrication methodology was utilized. Firstly, a matrix shape for duplication of the zirconia specimens was made from A-silicone Betasil vario medium (Müller – Omicron & Co. KG Germany) and twelve duplicated discs are made from a modelling wax – Cavex Set Up Regular (Cavex Holland) (Fig. 1a). Then the carrier is made from a polypropylene plate for splints with a thickness of 2 mm and epoxy resin (Fig. 1b). Twelve holes are made with a diameter of 1 mm and height of 1 mm with a laboratory cutting machine Paraskop (BEGO Germany)

T a b l e 1

Sample groups' number, methods and parameters of specimen surface treatments and number of treated specimens in each group

No.	Method of surface treatment	Parameters	Number of treated specimens
1	Control group	Left as sintered, without any modification of its surface texture	6
2	Treatment only with MDP primer	Specimens were treated only with MDP primer (Panavia V5 Tooth primer, Kuraray Noritake Dental Inc.)	6
3	Sandblasting with 50 μm Al_2O_3 , 2 bar	Sandblasting with 50 μm aluminium trioxide by means of a dental sandblaster (Renfert, Germany) at a pressure of 2 bar for 5 s, from a distance of 10 mm in a circular movement at an angle of 90°	6
4	Sandblasting with 50 μm Al_2O_3 , 2 bar and MDP primer	Sandblasting with 50 μm aluminium trioxide by means of a dental sandblaster (Renfert, Germany) at a pressure of 2 bar for 5 s, from a distance of 10 mm in a circular movement at an angle of 90° in combination with MDP primer	6

(Fig. 1b). The wax prototypes are centred in the polypropylene plate in such a way that the plate allows the immediate applying of the resin cement in all holes. The produced individual matrix shape for applying the cement guarantees perpendicular positioning of the stalks on zirconia discs. In this way during the micro-shear bond strength test the stress from the cutting force is distributed in the interface between the studied materials. The gathered data is correctly interpreted and specified particularly the strength of the bond, eliminating the subjective factor – their own strength. Subsequently the carrier is filled with epoxy resin (Fig. 1c). After a 24-hour period of full toughening, the carrier is freed from the wax specimens under steam. Zirconia discs are placed in the released gaps of the wax specimens. All twelve holes are filled with dual-cure resin cement (Panavia V5, Kuraray Noritake Dental Inc.) and are subsequently light cured with Curing Light XL 3000 (3M Dental Production) (Fig. 1d).

Testing procedure. The micro-shear bond strength test was performed by a universal testing machine LMT 100 (LAM Technologies, Italy). An axial load by a knife-shaped element specially designed for the study was applied on all zirconia specimens with resin stalks. The element induces pressure only on the bonded interface between the dual-cure resin stalk and zirconia disc. Loading was performed at a crosshead speed of 2 mm/min until failure occurred with an

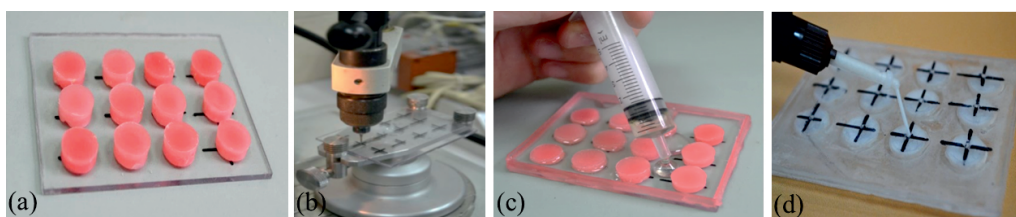


Fig. 1. Consecutive steps of elaboration of a carrier and applying the dual-cure cement

optimal load of the machine of 400 N. Failure load (N) was extracted from the computer-generated data files and was used as the main reference point for results evaluation.

Results. The micro-shear bond strength is evaluated by the following formula: $F = P/S$, where F is the compressive strength in MPa/mm², P is the value of the force in the stress zone, which is the highest at the moment of cutting and is presented by a software program both graphically and digitally, S is the area of the resin stalk. All data is processed statistically. Statistical analysis was performed with SPSS 11.5 Inc., Chicago, IL, USA, Excel 7.0 VB for applications and PraphPad Prism 3.0 (PraphPad, Soft, USA). One-way analysis of independent samples (ANOVA) was used to analyze the data with the level of significance $p < 0.05$. The data is arranged according to the statement $\text{MEAN} \pm \text{SDM}$, where MEAN is the mean value, SDM is the standard deviation.

The gathered data (Fig. 2) indicates that treatment with MDP-primer (group 2) or sandblasting (group 3) only are eleven and nine times, respectively, more ef-

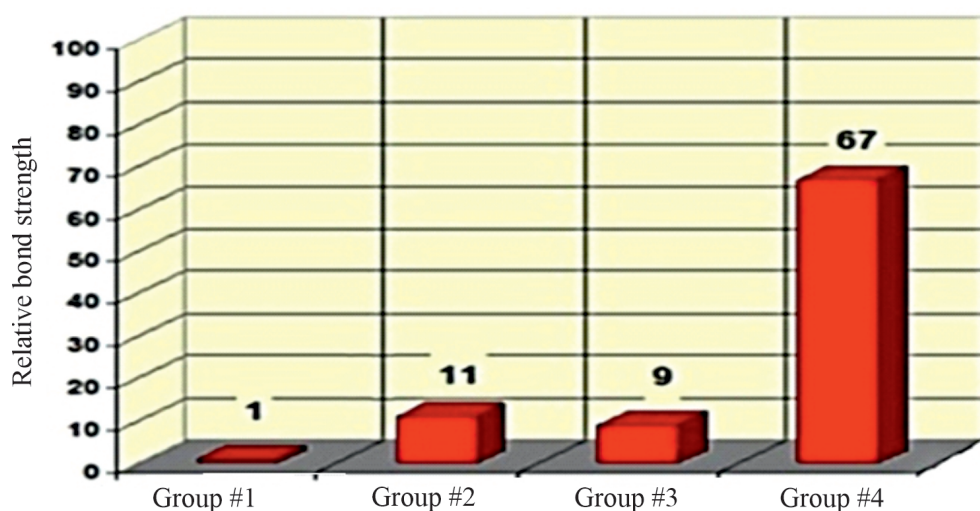


Fig. 2. Relative shear bond strength of the different groups in comparison to the control group

fective than no treatment of the surface at all (group 1). However, the combined regime of MDP-primer and sandblasting (group 4) is 67 times more effective as compared to control group.

An analysis for estimating the relative change of the shear bond strength in comparison to a control group was done. As mentioned above the Bonferroni's Multiple Comparison Test was used to reduce the instance of false positive results. From one-way analysis of variance it is apparent that there is no statistical significance between group treated only with MDP-primer and those treated with 50 μm aluminium trioxide ($p > 0.05$). In all other cases there is a statistical significance between the different treatment groups (Table 2).

T a b l e 2
Comparison between different surface pre-treatments

Bonferroni's Multiple Comparison Test	MEAN	SDM	p value
Group#2 vs Group#3	4.944	1.364	$p > 0.05$
Group#2 vs Group#4	-69.88	19.28	$p < 0.001$
Group#2 vs Group#1	15.10	4.165	$p < 0.01$
Group#3 vs Group#4	-74.82	20.64	$p < 0.001$
Group#3 vs Group#1	10.15	2.801	$p < 0.05$
Group#4 vs Group#1	84.97	23.44	$p < 0.001$

Discussion. The results of this study show that there is a significant difference in bond strength between the tested groups. The highest value was observed when the experimental primer was used in combination with sandblasting. This can be explained by the presence of organo functional silanes in the primer composition and their chemical reaction with the sandblasted zirconia surface.

Sandblasting, dental grinding and chemical etching are commonly used surface treatments [6]. Sandblasting involves impacting the required surface with hard particles at high velocities thereby eroding the material and leaving a roughened surface with expected higher wettability. As a result microcracks are generated during this process [7], which according to ZHANG et al. [8] are responsible for the reductions in near surface elastic modulus of sandblasted zirconia measured by nanoindentation. At the same time, the increase in strength after sandblasting of Y-TZP is often attributed to the t-m phase transformation associated with the generation of residual compressive stresses [9]. The final strength of the sandblasted material must be the result of a trade-off between damage and compressive residual stress generated by the impact of particles. This is why proper process parameters of the sandblasting treatment must be selected to achieve the desired result (higher wettability) with minimal adverse effects. The influence of sandblasting conditions such as particle size and pressure on the roughness has been studied by several authors [10,11], but very often by examining the effect of only one sandblasting parameter.

Some authors [12] found no significant changes in roughness of dental zirconia (LAVA 3Y-TZP) after sandblasting with alumina particles of sizes 25, 50 and 110 μm at 4.8 bar pressure. In addition to this OZCAN et al. [13] reported no change in roughness in dental zirconia (3Y-TZP VITA InCeram) sandblasted with 30 and 110 μm of alumina particles coated with silica at 2.8 bar pressure. This is in contradiction with the results of our study, where air abrasion with 50 μm aluminium trioxide particles had an eminent effect on adhesion strength of zirconia surface to the resin cement.

On the other hand, previous reports on Y-TZP after sandblasting have found an increase in the biaxial flexural strength of the sandblasted surface. SATO et al. [9] reported that strength increased by sandblasting with 70 and 110 μm particles at 4 bar. Similarly, KOSMAC et al. [14] also concluded that the strength increased after sandblasting with 110 μm particles at 4 bar, though the sandblasted material had lower Weibull modulus as compared to control group. SOUZA et al. [15] also confirmed increase in strength after sandblasting with alumina (50 and 100 μm) and silica (30 and 110 μm) particles at 2.5 and 3.5 bar pressure compared to control specimens. In CAD/CAM zirconia frameworks sandblasted at 3.5 bar, WANG et al. [16] noticed that the strength increased with 50 μm particle size and decreased with 120 μm particle size. The dissimilar results on the strength that exist in the literature are often associated with different type and size of particle, working pressure, nozzle size, angle of projection, and other parameters such as the microstructure of the material studied.

Our study also confirmed that sandblasting with 50 μm alumina particles increase the bond strength. However, the most significant change was observed in the combined regimes of surface treatments including air abrasion with 50 μm aluminium trioxide and MDP-primer (67 times higher values in comparison to the control group).

Another approach to improve the bond strength to zirconia is to develop a chemical interaction between the zirconia surface and the applied resin monomers. 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer has been used by several investigators for this purpose, with effective bonding between the MDP acidic groups (phosphoric acid) and the oxide layer of the zirconia [17]. High initial bond strength values were reported when an MDP-containing resin cement (Panavia F 2.0; Kuraray) was used [18]. When an MDP-containing primer (Clearfil Ceramic Primer; Kuraray) was used in conjunction with an MDP-containing resin, high bond strength values to zirconia were maintained after thermocycling [19]. These results are in total confirmation with the results of our study. The highest values were achieved in the combined regime of surface treatment including both sandblasting with 50 μm -sized particles of aluminium trioxide and the use of MDP-primer. The used cement in our investigation was also MDP-containing dual-cure resin cement as the primer itself. This led to absolutely homogeneous bond with great micro-shear strength.

Conclusions. Within the limitations of this in vitro study the following conclusions were evident:

1. Surface pretreatment of zirconia before cementation with single regime of air abrasion with 50 μm particles and treatment only with MDP-primer led to increased bond strength between the material surface and the resin cement.
2. The highest results in bond strength (more than 50 times) were achieved in the combined regimes of surface treatment with air abrasion and MDP-primer.

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