International Journal Dental and Medical Sciences Research
Volume 6, Issue 2, Mar - Apr 2024 pp 83-87 www.ijdmsrjournal.com ISSN: 2582-6018

The Effect of 10-MDP Primer Application Prior to Saliva Contamination on ZirconiaBond Strength

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Submitted: 07-03-2024 Accepted: 17-03-2024

ABSTRACT

Objective: This research aimed to investigate the impact of applying 10-MDP ceramic primer before saliva contamination on zirconia-resin bond strength.

Materials and methods: A total of 14 yttria-stabilized tetragonal zirconia discs were CAD/CAM fabricated. The bonding surface of zirconia discs was airborne particle abraded using 50 µm Al_2O_3 particles and randomly divided into two equal groups (n=7); Group (1): no saliva contamination, Group (2):10-MDP primer application before saliva contamination followed by water rinsing.Composite discs were fabricated and then cemented to zirconia discs by adhesive resin cement.All specimens were stored in distilled water at 37 ˚C for 24 hours followed by 10,000 thermal cycles. The shear bond strength (SBS) test was used to determine the bond strength using the Instron universal testing machine. The mean shear bond strengths were compared by tabulating the results and statistically analyzing them using the student's t-test.

Results:SBS values revealed statistically significant differences (p<0.001) between the study groups.The mean shear bond strength of group(1) (16.07±2.14MPa) was higher than that of group (2) (11.26±1.55 MPa).

Conclusion: Saliva contamination negatively affected the zirconia-resin bond strength,suggesting that cleaning or preventing the contamination of the zirconia surface is essential.

Keywords: Zirconia**,** 10-MDP primer, Saliva contamination, Resin cement, Bond strength.

I. INTRODUCTION

The utilization of zirconia-based restorations has significantly increased due to their exceptional biocompatibility, high mechanical properties, easy fabrication, and satisfactory esthetics.(1)Zirconia cannot be etched to create micromechanical retention with hydrofluoric acid, unlike glass ceramic material, due to the lack of glass phase.(2)

Surface treatment of zirconia dental restorations can be achieved through airborne particle abrasion using aluminium oxide (Al_2O_3) particles, typically followed by the application of MDP, which can be used either as a primer or as a constituent of the cement. This approach is widely considered to be the gold standard for zirconia dental restoration surface modification.(3)

During the clinical try-in of indirect restorations, there is a high likelihood of exposure to contaminants such as blood, saliva, or silicone-indicating paste. The quality of the bond strength between the zirconia surface and the resin cement can be negatively impacted by contaminationbecause zirconia has a great affinity to phosphate ions, found in saliva and blood.(4, 5)

Several methods for cleaning contaminated zirconia before bonding have been reported in the literature.(6, 7)Sandblasting with alumina particles (Al_2O_3) can be used for mechanical cleaning of contaminated surfaces and to enhance surface energy, thereby restoring original bond strength. When sandblasting zirconia, it is important to follow appropriate guidelines for particle size, distance, and pressure to prevent the occurrence of large surface flaws.(2, 8)

Chemical cleaning methods for contaminated zirconia have also been reported. They are safely applied without any adverse effect on the mechanical properties of zirconia. Phosphoric acid is considered efficientin eliminating organic contaminants, but X-ray photoelectron spectroscopy proved the presence of phosphorous residue which might influence the bonding negatively. Hydrofluoric acid is readily available in the dental clinic and has been used to clean contaminated zirconia surfaces; it is believed that no residue of hydrofluoric acid remains when using this method in comparison to phosphoric acid.(2, 6)

Variouscommercially available products were advocated and became an efficient method to safely remove contaminants without influencingthe bond strength or mechanical strength of the ceramic substrate, for example, chemical cleaners for dental zirconia, such as Ivoclean (Ivoclar Vivadent, Schaan, Liechtenstein), ZirClean^{TM} (BISCO, Inc., Schaumburg, IL, USA), and KATANA^{TM} Cleaner(Kurary Noritake Dental Inc., Okayama, Japan) with different compositions and mode of actions.(2, 8)

In some clinical situations, the practitioner will contaminate the zirconia restoration following MDP primer application. In this scenario, efficient cleaning methods have not been sufficientlyinvestigated. MDP is a bifunctional monomer with a phosphate-based functional end that bonds to zirconia and a methacrylate-based functional end that bonds to resin cement. Theoretically, the application of an MDP primer to the surface of zirconia should expose the hydrophobic methacrylate ends of the bound MDP molecules. Increasing the hydrophobicity of the zirconia surface will diminishthe wetting of the zirconia by saliva, which is composed of 99% water.(9) So, the null hypothesis of this study was that the application of ceramic primer prior to saliva contamination would preserve the bond strength of resin cement to zirconia.

II. MATERIALS AND METHODS

Materials used in this study and their basic compositions are shown in **(Table 1)**. Fourteen discs of yttria-stabilized tetragonal zirconia were fabricated using the IPS e.max®ZirCAD (Ivoclar Vivadent AG, Schaan,Liechtenstein) by CAD-CAM technology.AutoCAD software was used to produce a zirconia disc design (8 mm ×3 mm). It was saved as a (.STL) file to be exported and read by the CAD/CAM machine software.Zirconia discs were dry-milled utilizing a 5-axis milling machine (Roland DWX-52D DGSHAPE milling machine, Osaka, Japan) and sintered in the furnace (LHTCT 01/16, Nabertherm GmbH, Lilienthal/Bremen, Germany)at 1500 ˚C for 2 hours.

After completing the milling and sintering phases, the zirconia discs' bonding surface was airborne-particle abraded using 50 µm aluminum oxide (Al_2O_3) particles(Basic Eco Sandblaster, Renfert GmbH, Hilzingen, Germany) at a pressure of 0.2 MPa for 10 seconds. The sandblaster tip was perpendicular to the disc surface and at a distance of 10 mm. Zirconia discs were then ultrasonically cleaned (Vitasonic II, Vita Zahnfabrik, Germany) in pure ethanol for 3 minutes, rinsed with distilled water, and air-dried.(10)

Zirconia discs were randomly divided into two equal groups (n=7) as follows; Group (1):control group: no saliva contamination,Z-PRIME^{TM} PLUS (Bisco Inc. Schaumburg, IL, USA) was applied on bonding surface following the airborne-particle abrasion, waiting for 30 seconds and then air-dried for 3-5 seconds according to manufacturer's instructions, Group (2): Z-PRIME^{TM} PLUS (Bisco Inc. Schaumburg, IL, USA)was applied as mentioned in the group (1) then zirconia discs were immersed in artificially prepared saliva for one minute, rinsed with water spray for 20 seconds, and air-dried.(6)

Composite discs (5 mm × 2.5 mm) were fabricated using the light-cured composite resin Neo Spectra^{TM} ST HV (Dentsply Sirona, Konstanz, Germany) thatwas condensed in a specially designed Teflon mold. Duo-Link Universal^{TM} (Bisco Inc. Schaumburg, IL, USA) resin cement was used for the cementation of composite discs to the zirconia discs under a static load of 1 kilogram (Kg) for 5 minutes using a cementation loading device so a uniform cement layer was created.(11)

The resin cement was initially light-cured for 3 seconds from all directions using an LED curing device (Gulin Woodpecker Medical Instrument Co, Ltd, Guangxi, China) to remove the excess cement with a disposable micro-brush. The final light curing was performed corresponding to the manufacturer's instructions for up to 40 seconds.

All specimens were stored in distilled water at 37 ˚C for 24 hours.Then, they were subjected to 10,000 cycles of thermocycling between a temperature of 5 ˚C and 55 ˚C with a dwell time of 30 seconds at each temperature.(12)The bond strength was determined by the shear bond test using the Instron universal testing machine (Model 3345, USA).

The bonding surfaces of debonded specimens were observed under the stereomicroscope (SZ61TR, Model SZ2-ILST, Olympus Co., Shinjuku-ku, Tokyo, Japan) to determine the mode of failure. The mode of failure was classified as one of the following types: **Type I** (cohesive) when the failure occurred within the resin cement, **Type II** (adhesive) when the failure occurred at the zirconia disc/resin cement interface, and **Type III** (mixed failure) representing a combination of adhesive and cohesive failure. A representative specimen for each failure mode wasselected and scanned by SEM (JSM.6510LV, JEOL Ltd., Akishima, Tokyo, Japan).

Table 1. Materials utilized in the study.

Material	Composition	Manufacturer	Lot number
IPS e.max® ZirCAD	88.0 % - 95.5% ZrO_2,> 4.5 % - ≤ 6.0 %Y_2O_3, ≤ 5.0 % HfO_2, ≤ 1.0 % Al_2O_3, ≤ 1.0 % other oxides	**Ivoclar Vivadent** AG, Schaan, Liechtenstein	X46187
Z-PRIME™PLUS	BPDM, HEMA, MDP, Ethanol	**Bisco Inc.** Schaumburg, IL, USA	2300010396
Duo-Link Universal™	Base: Bis-GMA, TEGDMA, UDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass filler	**Bisco Inc.** Schaumburg, IL, USA	2300000645
Neo Spectra™ ST HV	SphereTEC® fillers (d3, 50≈15 µm); non-agglomerated barium glass and ytterbium fluoride; filler load (78–80 wt%); highly dispersed, methacrylic polysiloxane nanoparticles	**Dentsply Sirona,** Konstanz, Germany	2210000350

III. RESULTS

The mean shear bond strengths were compared using the student's t-test revealing a statistically significant difference between the study groups. The mean shear bond strength of group (1) (16.07±2.14 Mpa) was higher thanthat of the group (2) (11.26± 1.55 Mpa)**(Table 2), (Figure 1)**.Failure patternsanalysis of debonded specimensshowed predominant adhesive failure in group (2) while group (1) showed some mixed failure patterns in addition to the adhesive failure**(Table 3)**.

Table 2. Mean shear bond strengths of the study groups.

Group	Group (1)	Group (2)	Test of significance
Mean±SD	16.07±2.14 **a**	11.26±1.55 **b**	t =4.83 p<0.001*

Methacrylate-modified polysiloxane (organically modified ceramic) dimethacrylate resins, ethyl-4 (dimethylamino) benzoate, and bis(4-methyl-phenyl) iodonium hexafluorophosphate. Filler load: 78–80% by weight: Spherical, pre-polymerized SphereTEC fillers (d 3,50 ≈ 15 µm), non-agglomerated barium glass and ytterbium fluoride - Different letters denote significant difference.

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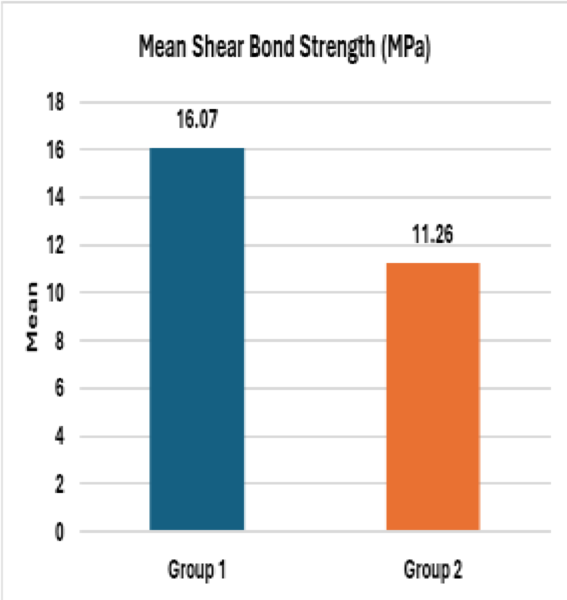

Figure 1. Bar chart showing the mean shear bond strength of the study groups.

Table3. Failure patterns of tested groups.

Group	**Group (1)**	**Group (2)**
AdhesiveFailure	5	7
Cohesive Failure	0	0
Mixed Failure	2	0

IV. DISCUSSION

Achieving a long-lastingrestoration is reliant on successfully forming a reliable bonding of the restorative material. Failure to decontaminate the bonding surface is a commonreasonfor bond failure.Consequently, ensuring all bonding surfaces are devoid ofcontaminants is significant for the longevity of the restoration. Try-in process of zirconia restorations results in contact of bonding surfaces with contaminants (saliva, blood, etc.) in the oral cavity. Various techniques and cleaning methods exist for removing the contaminants and restoring the surface prior to cementation.[8]

A study by**Angkasith et al. (2016)**[9]suggestedthat the application of a ceramic primer prior to the try-in phase would protect the surface from saliva contamination. When the ceramic primer is agitated on the surface of the zirconia restoration, phosphate groups bond to ZrO_2, exposing the hydrophobic methacrylate ends of the amphipathic molecule. The exposure of the methacrylate end of the molecule creates a hydrophobic barrier, which was assumed to inhibit surface wetting and subsequent saliva contamination.

However, the results of the present study did not coincide. Perhaps the surface contamination present at the time of cementation may have interrupted the hydrophobic barrier of the ceramic primer, allowing organic material to attach to the zirconia surface. These outcomes were in agreement with a study by**Sulaiman et al. (2022)**.[8]Therefore, rinsing with water may be insufficient fortotally eliminatingsaliva contamination from the bonding surface of the zirconia.

Despite the shear bond strength of group (2) in which the 10-MDP primer applied prior to saliva contamination was lower than that of the control group (1), it was suggested by authors in previous studies that at least 10 MPa is the clinically sufficient level of bonding strength.[13, 14] So, it may be considered an acceptable bond strength but it didn't preserve the original SBS.Therefore, the null hypothesis was rejected. The predominant adhesive failure in the group (2) coincided with thedecrease in the SBS.

V. CONCLUSION

10-MDP primer application before saliva contamination followed by water rinsingmay be insufficiently effectiveand additional measures should be applied. Cleaning of zirconia restoration bonding surfaces is mandatory after the try-in procedure tomaintain the bond strength.

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