

Bond Strength to Zirconia reinforced machinable glass ceramic: Influence of different conditioning protocols

--- Date of Submission: 20-09-2023 Date of Acceptance: 30-09-2023 ---

ABSTRACT

Background:To evaluate the influence of micromechanical roughening and different primers on the bond strength of resin cement to zirconiareinforced glass ceramic.

Materials and methods: Blocks of Vita Suprinity (Lithium Silicate Glass Ceramic Reinforced by 10~12% Zirconia) were cut into 60 slices with dimensions (length= 7mm, width= 7mm, thickness= 3mm) using CAD-CAM system. The total 60 samples were divided into three main $groups(n=20)$ according to method of surface treatment: Group Ⅰ (TSC); Tribochemical silica coating using CoJet sandblast, group \mathbb{I} (Al₂O₃); air abrasion with 50μ m aluminum oxide particles, group \mathbb{I} (M.E&P); using Monobond Etch & Prime. Each main group was subdivided into two subgroups $(n=10)$ according to the primer to be applied: $\text{subgroup}(A)$; monobond N application, subgroup (B); Z.Prime Plus. A total of 60 discs of resin composite (Nexocomp) were fabricated and cemented to the treated ceramic samples using universal adhesive resin cement. All bonded specimens were stored in the water bath for 3 months and subjected to thermal stress for 3000 cycles. Shear bond strength (SBS) tests were performed.

Result: TSC surface treatment with Monobond N prime showed the highest mean SBS $(26.1 \pm$ 5.5MPa) followed by M.E&P with Monobond N $(22 \pm 3.9 \text{MPa})$. Whereas the AL₂O₃ with Z. Prime showed the lowest mean SBS $(8.7 \pm 4.4 \text{ MPa})$. Also, Monobond N primer showed a high mean of SBS (21 \pm 6.7 MPa) compared to Z. Prime Plus $(15.3 \pm 6.3 \text{ MPa})$ with a statistically significant difference of (P<0.001) regardless of surface treatment used.

Conclusion: TSC surface treatment followed by the application of Monobond N primer showed the highest shear bond strength

Key words: Glass ceramic, primers, shear bond, surface treatment.

I. INTRODUCTION

An alternative to the traditional restorative strategies for fixed dental prostheses (FDPs) is the concept of monolithic full-contour restorations made with Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) technology today **[1]**.

This conservative option permits a significant decrease in the thickness of the ceramic material, aligning with the principle of minimally invasive dentistry. It highlights the utilization of strong materials and adhesive bonding methods to repair teeth **[2]**, Hence, it is essential to exercise caution when determining the minimum thickness of the ceramic material to guarantee satisfactory mechanical functionality.zirconia-reinforced lithium silicate glass ceramic is designed for various applications such as inlay, onlay, partial crown, veneer, anterior and posterior crown **[3]**.

This innovative glass-ceramic incorporates zirconia (approximately 10% weight), making it the first glass ceramic reinforced with zirconia. Moreover, according to the manufacturer's claim, this innovative class of glass-ceramic materials effectively integrates the beneficial attributes of zirconia $(ZrO₂)$ and glass ceramic, creating a powerful combination of properties that enhance its overall performance. The incorporation of zirconia serves to strengthen the ceramic structure by interrupting cracks. It is believed that the crystallized structure resulting from this process exhibits improved mechanical properties and meets the most stringent aesthetic standards. The restoration exhibits anatomical contouring as a single, solid unit, which results in improved translucency and provides a diverse selection of shade choices **[4]**.

In contrast to traditional lithium disilicate ceramics, which lack zirconia reinforcement, ZLS glass ceramics present a notably lower crystal phase content percentage. The crystal phase content ranges from 40% to 50%, whereas conventional lithium disilicate glass ceramics typically have a crystal phase content of around 70%. Nevertheless, in ZLS materials, the crystals are smaller, and the glassy matrix is strengthened through the addition of highly dispersed zirconium dioxide (approximately 10% in weight). This incorporation of zirconium dioxide is believed to enhance the strength of the glassy phase **[4,5]**.

Furthermore, since the ceramic matrix of ZLS materials consists mainly of glass (with zirconia content ranging from 8% to 12%), this material is considered to be sensitive to acids and can undergo hydrofluoric acid etching, unlike polycrystalline ceramics **[6]**. The classification system introduced by **Valandro et al [6]**. is

founded on the presence of ceramic surface degradation caused by hydrofluoric acid (HF). Ceramics that have a significant amount of glass in their composition, such as feldspar, leucite, and lithium disilicate, are vulnerable to the effects of hydrofluoric acid. This exposure leads to the development of a micromechanical retentive surface, hence earning the designation of "acidsensitive".

The use of hydrofluoric acid demands careful attention due to its potential risk of degrading organic matter. Therefore, alternative options have been explored for treating ceramic surfaces, such as air abrasion using aluminum oxide particles and silica-coated aluminum oxide particles **[7]**.

In the process of silica coating, the high energy generated by the impact of aluminum oxide particles leads to the fusion of silica grains onto the ceramic surface. This fusion renders the ceramic surface reactive chemically to the resin cement when coupled with a silane agent. Additionally, this coating process enhances the bond strength between the ceramic surface and resin cement **[8]**.

In addition to air abrasion, and to address the challenges associated with hydrofluoric acid etching, a self-etching ceramic primer (SECP) has been suggested as an alternative. It offers a safer and less technique-sensitive method of surface treatment while combining the advantages of hydrofluoric acid and silane treatment **[9,10]**.

The impact of self-etching ceramic primer on the bonding of conventional glass ceramics has been extensively studied. Various studies have reported similar outcomes when comparing the use of a self-etching ceramic primer to traditional surface conditioning for preparing glass ceramics. These investigations highlight comparable results in terms of bonding efficacy and overall restoration performance **[9,11]**.

In addition to micromechanical surface treatment, and to achieve a durable resin-ceramic adhesion, different primers are used to enhance chemical bonding to glass ceramics **[12,13]**. Therefore, the purpose of this invitro study was to evaluate the influence of micromechanical roughening and different primers application on the bond strength of resin cement to zirconiareinforced glass ceramic.

Hypotheses of the study

The methods utilised for conditioning ceramic surface would have no effect on shear bond strength. Also there would be no significant difference in shear bond strength when utilising different primers.

Material	Product name	Main composition		Manufacturer Lot	
number					
Zirconia- reinforced lithium silicate Glass-ceramic	Vita suprinity	P_2O_5 (3-8) % $Li2O$ (15-21) % $CeO2$ (0-4) % $K2O$ (1-4) % Al_2O_3 (1-4) % $SiO2$ (56-64) % $ZrO2$ (8-12) % Pigments $(0-6)$ %	VITA -Zahnfabrik, Bad-sackingen, (Germany)	97560	
Tribochemical Silica Coating	Cojet sandblast	30 μ m silica Alumina blast coating (Al_2O_3) : agent >97%. Amorphous silica: $\langle 3\% \rangle$	Deutschland 3M GmbH, Germany	8941916	
Self-etching glass ceramic primer	Monobond Etch & Prime	phosphoric acid methacrylate, alcoholic An aqueous solution of ammonium- polyfluoride, silane, and colourants	Ivoclar Vivadent. Schaan/Liechtenstein	Z03020	
50 μ m Al ₂ O ₃	Aluminum Oxide 50 Microns	99.7% aluminium oxide	Dentify GmbH, Scheffeslstr, Engen, Germany	A2468	

II. MATERIALS AND METHOD Materials used in this study are listed in (**Table.1**).

phosphate, HEMA 2-hydroxyethyl methacrylate, BPDM biphenyl dimethacrylate, Bis-GMA bisphenol A-glycidyl methacrylate, UDMA Urethane dimethacrylate, Bis-EMA ethoxylated bisphenol A glycol dimethacrylate, SiO₂ Silicon dioxide, Li₂O Lithium oxide, K₂O Potassium oxide, ZrO₂ Zirconium dioxide, CeO₂ ceric dioxide.

Specimens fabrication:

Sixty square samples measuring (7mm width, 7mm length, and 3mm thickness) were sectioned from zirconia-reinforced lithium silicate glass ceramic (18x14x12mm3; Vita Suprinity,) were wet milled using CAD-CAM system (Ceramill Motion 3; Amann Girrbach AG). The specimens were polished manually on both surfaces using abrasive carbide papers of different grit sizes, specifically #400, #600, and #1200-grit under rising water to obtain a standardized smooth surface. All prepared specimens were ultrasonically cleaned using an ultrasonic bath containing 96% ethanol for 5 minutes.

Surface treatment:

Specimens were divided into three groups $(n=20)$ according to the method of surface treatment (Table.2).

Following the surface treatment, only the AL_2O_3 group was ultrasonically cleaned using an ultrasonic bath containing 96% ethanol for 5 minutes. According to the primer that would be applied, each main group was subdivided into two equal subgroups (n=10) (Fig.1): Subgroup A; Monbond N (Ivoclar Vivadent); was applied to the treated surfaces utilising a microbrush to apply a

thin layer. Permit the materials to remain for 60 seconds. Subsequently, any remaining materials were eliminated through the application of a potent stream of air. Subgroup B; Z. Prime Plus (Bisco, USA); 1-2 coats of Z. Prime Plus was applied by microbrush to the treated surfaces and allowed for 30 seconds followed by then air drying.

Fig.1 Diagram showing study design

To investigate the surface characterization of zirconia reinforced glass ceramic, one specimen was examined from each subgroup. Each specimen was air-dried, mounted on a copper stub, and coated with a thin layer of gold (Sputter Coating Evaporator, SPI-Sputter Coater, USA) before being examined with a Scanning Electron Microscope (SEM) (JEOL.JSM.6510LV, Japan) at various magnifications (x50, x500, x1000, x2000).

Composite disc fabrication:

A total of 60 discs of composite resin were fabricated using a multihole Teflon mold with dimensions of (4mm internal diameter and 3mm

thickness). The composite resin (META BIOMED, South Korea) was applied within a Teflon hole incrementally to fabricate the desired composite resin disc. Light cure polymerization was done by applying light of 1000W/cm2 intensity (liteQ LD-107, MONITEX, Taiwan) for 20 seconds from a distance of 3mm according to manufacturer instructions for each layer. After that, the discs were removed from the holes and inspected for any defects. The untreated surfaces of the composite discs were marked with waterproof markers. Bonding surfaced of the discs were air-abraded with 50μ m AL₂O₃ (SHERA ALUMINUM OXIDE, WerKstoff. Technology, Germany).

Cementation of composite discs to ceramic Specimens:

The discs of composite resin were cemented to the pretreated bonding surfaces of ZLS specimens using a conventional dual cure resin cement (DUO-LINK UNIVERSAL, BISCO, INC.SCHAUMBURG, U.S.A) according to the manufacturer instruction. Ceramic specimens and discs of composite resin were secured together

utilizing a special designed device with a lever system. (Fig.2). Universal resin cement was applied to the treated surfaces of ZLC specimens to which the composite resin discs were applied under a constant load of 5 Kg for, the excess resin cement was removed using a micro brush. After that, a light-cure (liteQ LD-107, MONITEX, Taiwan) was performed from all around for about 20 seconds for each side. The load was left after 5 minutes.

Fig.2 Special designed device for cementation

Artificial aging:

One hour after bonding, all cemented samples were submerged in a bath of water at a temperature of (37º) for three consecutive months. Then, thermocycled for (3000 cycles) using thermocycling machine between 5° and 55° in water. The time of transfer between the baths of various temperatures was 15 seconds, and the specimens were held for 30 seconds at each temperature. One day before shear testing was conducted, the specimens were placed in a water bath after thermocycling to bring them to room temperature.

Shear bond strength test (SBS):

The measurement of shear bond strength was conducted using a universal testing machine (Instron Industrial product Model 3345, Norwood, MA, USA) The load cell utilised for this

experiment had a capacity of 5 KN, and the crosshead speed was set at 0.5 mm/min. The Blue Hill Instron software was employed for data analysis. Each specimen was mounted on the testing machine, a custom-made jig consisting of two metal pieces tightened to each other by metal screws with internal holes that have a diameter similar to that of the specimen attached to the lower fixed head of the universal testing machine. A mono-beveled chisel 8mm in width and 0.5 mm in thickness is attached to the movable upper head of the machine to be flushed with a jig surface. After fixation of the specimen to the jig hole, a compression mode of force was applied at a crosshead speed of 0.5 mm/min at the ceramic /composite interface up to specimen failure. (Fig. 3) the load at which the specimen failed was recorded in (Newton) and bond strength was calculated in (MPa).

Fig.3 Application of the load for shear test

Failure mode analysis:

By examining the bonding surface of debonded specimens with a reflection microscope (S300; Inoue Attachment Corp) at x8 magnification, the mode of failure was determined and categorized into adhesive failures (occurring at the interface between the two materials), cohesive failures (within composite resin disc or resin cement), and mixed failures (involving both adhesive and cohesive aspects).

Statistical analysis:

Statistical analyses were done by Social Package for Statistical Science (SPSS) software version 25.0. Statistical analyses were done with two-way ANOVA and serial one-way ANOVAs at each level of the study followed by Post Hoc Tukey (HSD) test.

III. RESULTS

Shear bond strength test:

A two-way ANOVA analysis was employed to detect the effect of surface treatment, primer application and their interaction. Serial oneway ANOVAs test at each level of the study was conducted. Post Hoc Tukey test was conducted to compare the means of each pair of test groups at $(p<0.05)$. Box polts was used to confirm the previous results (Fig.4) .

International Journal Dental and Medical Sciences Research Volume 5, Issue 5, Sep-Oct 2023 pp 297-311 www.ijdmsrjournal.com ISSN: 2582-6018

Fig.4 Box Plots showing mean shear bond strength

The test reveal that there is a significant statistical effect (p<0.001) on the changing of priming agent and surface treatment methods on

the shear bond strength with 94.9% of shear strength can be affected by their combined effect.(table.3)

b. Computed using alpha = .05

Serial one-way ANOVAs.showed that both surface treatment, $(p=0.000)$ (Table 4), type of primer ($p=0.002$), (Table 5) showing highly significant effect.

Table 5: One-way ANOVA considering different primers

Shear Bond Strength	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	465.931	1	465.931	11.013	0.002
Within Groups	2453.809	58	42.307		
total	2919.739	59			

Following One-Way ANOVAs, a Post Hoc Tukey test was used to detect pairwise comparison between different studied groups. Considering different surface treatment methods, there was a statistically significant difference between AL_2O_3 and other types of surface

treatment (TSC and M.E&P) regardless of type of primer (Table 6).

Considering different primers, There was a statistically significant difference between the two primers Monobond N and Z.Prime when surface treatment with (TSC and AL_2O_3) were utilized. (Table 7).

International Journal Dental and Medical Sciences Research Volume 5, Issue 5, Sep-Oct 2023 pp 297-311 www.ijdmsrjournal.com ISSN: 2582-6018

***Indicate the statistically significant difference**

Table 7: Statistically significant differences between tested groups according to the type of primer used.

Failure mode:

Most of specimens showed a cohesive failure accept for the group of $AL_2O_3+Z.P.$ where only 2 of totally 10 specimens showed a cohesive failure mode. The group of T.S.C+M.N and M.E&P. showed no adhesive failure. While mixed failure was mostly occurred within the group of $AL₂O₃(N=20 Spcimens).$ (Fig.5) (Fig.6).

Fig.5 illustrated the failure modes of each group where the total numbers for each group was 10 specimens.

Fig.6 Failure patterns: **A**; cohesive failure within composite resin disk with remaining resin cement. **B**; Adhesive failure between vita block and resin cement. **C**; mixed failure illustrated the remaining resin cement and composite resin disk.

Scanning electron microscope (SEM) examination:

Furthermore, SEM images depicting various surface treatments administered to the vita suprinity surface are shown in (Fig.7). the monobond etch and prime exhibit noticeable, welldefined, elongated crystals with increased retentive shoulder irregularities and large deep porosities,

which arise from the dissolution of the glassy matrix, compared to irregular, blindly short crystals with tiny fissure like porosities formed by AL_2O_3 sandblasting group. While the tribochemical silicacoated group displayed a coarse surface irregularity with extensive deepened porosities where most of them filled with resin cement.

Fig.7 SEM images showing the topographical changes in the surface of vita suprinity according to the surface treatment applied: **A**; Tribochemical silica coated. **B**; AL_2O_3 sandblasting. **C**; Monobond Etch and Prime.

IV. DISCUSSION

The null hypotheses investigated in this study proposed that there would be no significant difference in shear bond strength when utilising different primer, and that the method utilised to conditioning the machinable glass ceramic surface would have no effect on shear bond strength values. The study findings demonstrated significant

differences in shear bond strength (SBS) values attributed to the use of various surface treatment methods (p < 0.001). Similarly, there were significant variations observed when different priming agents were employed ($p = 0.002$). Consequently, both of the tested hypotheses were rejected, indicating that both the surface treatment methods and priming agents had a notable influence on the shear bond strength values.

In order to simulate the intraoral conditions, the specimens was stored in a water for 90 days followed by 3,000 cycles of thermal stresss. This extended duration of exposure allows the adhesion composites to reach their maximum water absorption capacity where during the initial two months of storage, the highest amount of water absorption occurs in the adhesive bonding materials **[14]**. The specimens' ageing processes did not result in any bonding failure before shear test. The shear bond strength test is widely recognized as the standard laboratory method for assessing the adhesive properties of resin-bonded ceramic restorations and ceramic repair systems. It has been extensively employed in numerous studies as the primary means of quantifying bond strength **[15]**. Furthermore, it provided more standardized results. During the shear bond strength test, stresses are mostly focused to the bond interface, resulting in a homogenous stress distribution at the contact. These shear forces were considered to be the primary causes of in-vivo bond failure **[16]**.

In the present study, the TSC surface treatment combined with Monobond N primer demonstrated the highest mean shear bond strength value of $(26.1 \pm 5.5 \text{ MPa})$. Notably, no instances of adhesive failure were observed in this particular treatment group. **Al-Thagafi R (2016)** and others **[17]** also reported the same results. Nevertheless, this procedure is being used to restore the ceramic with composite resin. During the process of silica coating, the high-energy impact generated by the aluminum oxide particles causes the fusion of silica particles on the ceramic surface. This fusion renders the ceramic surface chemically reactive to the resin cement through the silane agent, ultimately leading to an increased bond strength between the cement and the ceramics **[18]**. Adding to that, **Salem A, Mohsen C 2020 [19]** concluded that the atomic force microscope surface analysis showed a high result of roughness associated with tribochemical silica coating sandblasting of ZLS compared to HF or AL_2O_3 . This fact supports our high result of shear bond strength for the TSC group. Furthermore, **Özcan and Bernasconi (2015); Saleh et al (2019) [21]** founded that sandblasting coupled with the use of MDPcontaining primers, is a highly recommended approach for bonding zirconia. This method is favored due to the dual benefits it provides: sandblasting enhances the surface energy and creates a rougher texture**[22]**, while MDP significantly augments zirconia's shear bond strength (SBS) through chemical bonding with metal oxide **[23]**. So, by using silica coating sandblasting surface treatment accompanied with

primers containing 10 MDB for zirconia reinforced lithium silicate glass ceramic (vita suprinity) will achieve micromechanical bonding by increasing surface roughness, chemical process by forming a covalent siloxane link with a ceramic hydroxyl group, and finally the chemical reaction of 10 MDB containing primer with the metal oxide of zirconia present in vita suprinity. This may explain the low result of SBS of a group treated with $AL₂O₃$, where there will be just a micromechanical adhesion by roughening action of sandblasting and also chemically by the action of primer. Additionally, **Vichi A. et al (2021) [24]** concluded that the greater zirconia content within ZLS may account for the higher prevalence of mixed failures observed for this ceramic through the functional phosphate monomers that chemically bond to zirconium oxides and, consequently, to ZrO2 containing glass ceramics(10-12%). However, when defects created by TSC treatment are not properly filled by the adhesive system, sandblasting ceramic surfaces can have a negative impact on the mechanical qualities **[25,26]**.

On the other hand, the surface treatment with AL_2O_3 showed the lowest mean value of SBS compared to other groups. This result is in the same line with the finding obtained by **Nakhaei M. et al (2023) [27]** where they concluded that sandblasting induces the formation of uneven surfaces that develop wedge-shaped fissures without generating uniform micro-retentive features. Additionally, this process may lead to the creation of microscopic cracks in glass ceramics. **Altan(2019) [28]** also revealed that The effectiveness of AL_2O_3 sandblasting surface treatment is limited on Vita Suprinity blocks and zirconia-based ceramics. This outcome is likely due to the lower surface hardness of Vita Suprinity blocks compared to zirconia, making sandblasting less effective on these materials and adding to that the nature of the adhesion bond created by AL_2O_3 air abrasion. Air abrasion with alumina particles is not suitable for glass ceramic surfaces because it may cause microcracks in the ceramic surface, resulting in prosthesis mechanical failure **[29]**.

Blatz MB (2003) [30], stated that air abrasion is not recommended for cementation of silica-based all-ceramic restorations as they found that the sole airborne particle abrasion provides insufficient bond strengths and the excessive airborne particle abrasion induced chipping or a high loss of ceramic material.

For the Monobond Etch & Prime(M.E&P), showed also a high result of SBS values slightly less than TSC but without a significant difference for Mononbond $N(P=0.170)$

and for $Z.P$ rime($P=0.093$). The notable high shear bond strength (SBS) values observed in SECP (Self-Etch Ceramic Primer) can be attributed to the micromechanical interlocking that occurs within the surface irregularities created. Scanning Electron Microscope (SEM) examinations revealed evident topographic changes and increased surface roughness in ZLS (zirconia-reinforced lithium silicate) when treated with SECP. These findings are consistent with previous research indicating that HF and SECP produce different etching patterns on glass ceramics **[31,32]**. Additionally, it has been established that the link between fluoride and silica is incredibly strong **[33]**. As a result, the increased bonding achieved with SECP may be attributed to the chemical affinity between the silica in ZLS and the ammonium trifluoride in SECP.

Dalla-Nora. et al (2022) [34] concluded that regardless of the usage of cement, surface treatments using hydrofluoric acid and ceramic self-etching primer had similarities to one another, also **Prado and others [35]** demonstrated that the self-etching ceramic primer achieved stable bonding after aging when used as a surface treatment for lithium disilicate and a feldspathic ceramic. On the other hand, the same researchers in the same studies, they revealed a high rate of pretest failure in SECP group. These findings disagree with our result because the group of SECP showed a high values of SBS without pretest failure which may result from the application of a primer containing 10 MDB increases the adhesion bond with ZLS as well as enhance the long-term adhesion bond of SECP by the action of primer on both silica and metal oxide in zirconia. **Guimarães. et al (2018) [36]** also concluded that the new ceramic self-etching primer is an effective alternative for simplified ceramic surface treatment when an adhesive agent is applied after it where they found that the utilization of bonding adhesive fosters improved interaction between ceramic and resin cement, resulting in a higher occurrence of mixed failures compared to the MBEP group alone. This outcome is likely attributed to the stronger chemical bond that is established between the ceramic and resin materials.

Donmez (2020) [37] Indications from the study suggest that MEP etching shows great potential as a surface treatment technique, leading to a significant enhancement in SBS (shear bond strength) values. the researcher concluded that MEP application proved to be an efficient surface treatment method, substantially improving both Ra (surface roughness) and SBS values when compared to the control samples.

The result of TSC and AL_2O_3 groups show a significant difference according to the primer used; TSC+M.N 26.1±5.5Mpa, TSC+Z.P
16.8±3.7Mpa, AL₂O₃+M.N 14.7±5.1Mpa, $16.8 \pm 3.7 \text{Mpa}$, $AL_2O_3 + M.N$ AL2O3+Z.P 8.7±4.4Mpa. while for the result of M.E&P group, there is a difference but without a statistical significant between M.N and Z.P plus $(22 \pm 3.9 \text{Mpa}, 20.6 \pm 3.4 \text{Mpa})$ respectively. Consequentially, the use of Monobond N silanization primer results in a high value of SBS than Z.Prime Plus for Vita zirconia-reinforced lithium silicate. One potential interpretation is that varying functional phosphoric acid and methacrylate groups exhibit distinct levels of hydrolysis resistance, leading to diverse bonding strengths with glass ceramic and resin cement **[38]**. Another contributing factor might be the inclusion of silane in the composition, such as in Monobond N, which enhances the glass ceramic wettability and facilitates bonding with the resin cement. The present study results concerning Z.Prime were unexpected. Despite zirconia primer Z.Prime containing 10-MDP, its bond strength exhibited the lowest values among all groups. Previous reports suggest that the carboxylic acid group might compromise the bond between 10-MDP and the methacrylate monomers in the resin cement, resulting in a weakened bond **[39,40]**. Furthermore, the lower bond strength observed in Duo-link resin cement and Z Prime plus was attributed to potential chemical distinctions in the base monomers or solvents used in the primers, variations in the primer initiation systems, or differences in the concentration of MDP **[41]**.

V. CONCLUSION

TSC surface treatment followed by the application of Monobond N primer showed the highest shear bond strength.

Abbreviations:

FDPs Fixed dental prostheses

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