Shear bond strength of indirect composite material to monolithic zirconia

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PURPOSE. This study aimed to evaluate the effect of surface treatments on bond strength of indirect composite material (Tescera Indirect Composite System) to monolithic zirconia (inCoris TZI). **MATERIALS AND METHODS.** Partially stabilized monolithic zirconia blocks were cut into with 2.0 mm thickness. Sintered zirconia specimens were divided into different surface treatment groups: no treatment (control), sandblasting, glaze layer & hydrofluoric acid application, and sandblasting + glaze layer & hydrofluoric acid application. The indirect composite material was applied to the surface of the monolithic zirconia specimens. Shear bond strength value of each specimen was evaluated after thermocycling. The fractured surface of each specimen was examined with a stereomicroscope and a scanning electron microscope to assess the failure types. The data were analyzed using one-way analysis of variance (ANOVA) and Tukey LSD tests (α=.05). **RESULTS.** Bond strength was significantly lower in untreated specimens than in sandblasted specimens (P<.05). No difference between the glaze layer and hydrofluoric acid application treated groups were observed. However, bond strength for these groups were significantly higher as compared with the other two groups (P<.05). **CONCLUSION.** Combined use of glaze layer & hydrofluoric acid application and silanization are reliable for strong and durable bonding between indirect composite material and monolithic zirconia. ***[J Adv Prosthodont 2016;8:267-74]***

KEYWORDS: Zirconia; Composite resin; Shear strength; Scanning electron microscopy; Surface properties

INTRODUCTION

Use of indirect dental restorations, the yttria-stabilized tetragonal polycrystalline zirconia (Y-TZP) ceramics, used for decades in biomedicine has been increasing because of esthetics, mechanical properties, and chemical inertia. The introduction of novel Computer-aided Design/Computer-aided Manufacturing (CAD/CAM) technologies has simplified the laboratory procedures required when using Y-TZP for dental prostheses.[1] After milling, Y-TZP frameworks have to be veneered with feldspathic or glass ceramics by means of layering, the press or the digital veneering technique.[2] However, clinical studies have reported failures such as chipping (cohesive failure of porcelain) and delamination (adhesive failure between the ceramic and framework.[3-6] Preis *et al.*[2] reported that different framework designs, veneer application techniques, and firing regimes influence the number and dimension of failures in zirconia-based all-ceramic crowns.

In recent years, the monolithic zirconia (fully anatomic zirconia or translucent zirconia) has been developed for tooth- and implant- supported all ceramic restorations in order to overcome these complications.[7-10] It requires less preparation[9] because the needed material thickness for restoration is less than other all-ceramic materials that allow full anatomical restoration.[11] However, this material is opaque[12] therefore indicated only in less-visible dental areas.[13,14]

Translucency is an important factor for controlling the esthetic outcome of restoration. Monolithic zirconia restorations can be digitally cut back on the anterior area. This

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allows the restorations to have some degree of translucency. In nonfunctional areas, the facial and/or incisal area, monolithic zirconia can be veneered with limited amount of ceramic.[10]

In the previous *in vitro* study, modified and monolithic zirconia full arch reconstructions presented longer life times but lower predictability of cycles to failure.[15] Modification of the crown design, covering zirconia crowns with porcelain (~ 1.0 mm) only on the buccal side and slightly extending to the occlusal surface, can minimize the occurrence of failures, increasing the survivability of restoration when it is under fatigue.[15] Venezia *et al.*[10] reported minor chippings on the incisal margins in 3 out of 11 monolithic zirconia prostheses, which were digitally cut back in the anterior area.

Porcelain has properties such as aesthetic appearance, strength, wear resistance, and less bacterial plaque involvement. It has also disadvantages such as brittleness, time-consuming the manipulation of porcelain, and requiring technical mastery.[16] The success of the restorations is affected by various factors such as interfacial bonding, match of the framework and veneer materials, strength of the veneering ceramic, and veneering technique (which include repeated sintering in the oven).[2,15,17]

Recently, highly loaded indirect composite materials have been used as an alternative for veneering in the zirconia-based restorations.[18-23] They are prepared in the dental office or the laboratory with techniques used on combinations of heat, pressure, vacuum and light polymerization.[24] Physical properties of indirect composite materials are improved due to enhanced degree of curing through these procedures.[25]

Tescera ATL system (Bisco Inc.) is a second-generation laboratory composite system. The system consists of a micro hybrid composite and an Indirect Aqua Thermal Light Polymerization unit for processing indirect composite restorations in an oxygen-free environment. Heat, light, and air pressure use for polymerization.[26] The restorations have the superior aesthetic and mechanical properties due to the post-curing procedure took place in a pressurized environment.

Ozakar-Ilday *et al.*[26] reported that restorations have a good wear-resistance performance and surface polishing due to the micro-particles in the filler and post curing increased color stability. Fruits *et al.*[27] stated that indirect resin composite restorations exhibited significantly less mean percent micro leakage at the gingival walls than the direct resin composite restorative groups. Cetin *et al.*[25] stated that indirect composite resin inlays and direct resin restorations displayed acceptable clinical performance after five-year use. However, there is limited data in the literature regarding characteristics and clinical success of these materials.[26]

Mechanical entirety and bonding of the veneering material to zirconia frameworks remain key factors for the successful performance of framework/veneer bilayered restorations.[18] Previous studies have examined various surface treatments such as sandblasting[19-21,23] and glaze layer & hydrofluoric acid (HF) application[20] for enhancing indirect composite material-zirconia bond. Sandblasting is an effective method that could improve the bond strength between framework and veneer materials. This conclusion is consistent.[19,21,23] Previous studies reported that sandblasting provide higher bond strength than grinding.[23,28] However, sandblasting caused surface damage and phase transformation (tetragonal to monoclinic) affects the mechanical properties and reliability of zirconia.[23,29]

Although HF application is commonly used for other ceramic systems in conjunction with silanization, this treatment has not been effective against the high acid resistance of zirconia due to the absence of a glassy matrix.[30,31] The glaze layer & HF application mentioned by Cura *et al.*[32] is a simple and inexpensive technique. The surface of the zirconia is coated with a thin layer of silica-based porcelain.[33] Glazed surface can etch by HF and the silanization can be applied.[34] A previous study reported higher bond strength values for indirect composite material-zirconia ceramics with the glaze layer & HF application.[20]

This study compared the effects of different surface treatments (control, sandblasting, glaze layer & HF application, and sandblasting + glaze layer & HF application) on the shear bond strength (SBS) of the monolithic zirconia and indirect composite material after thermal aging. The null hypothesis tested that different surface treatments have no effect on monolithic zirconia-indirect composite material bond strength.

MATERIALS AND METHODS

The compositions of materials used in this study are presented in Table 1. Pre-sintered monolithic zirconia blocks (inCoris TZI; Sirona, Bensheim, Germany) were cut into 2-mm thick slices using a slow-speed diamond saw sectioning machine (IsoMet 1000 Precision Saw; Buehler Ltd., Lake Bluff, IL, USA) under water-cooling. Then, the specimens were sintered in zirconia sintering furnace (inFire HTC speed; Sirona Dental Systems GmbH, Bensheim, Germany) according to the manufacturer's instructions. Fortyeight specimens were polished with 400, 600, and 1000-grit silicon carbide abrasive papers (3M ESPE, St. Paul, MN, USA) for 30 seconds using a 300 rev/min grinding machine (Minitech 233; Presi Ltd., Grenoble, France) under running water to standardize the surfaces and cleaned for 10 minutes using an ultrasonic bath (EasyClean Ultrasonic Cleaner; RenfertGmbh & Co., Hilzingen, Germany). Later, the specimens were randomly divided into 4 groups according to the surface treatment methods (n = 12).

No treatment group (Control; C): No surface treatment

Sandblasting group (S): Specimens were divested by airborne-particle abrasion with 50 μm aluminum oxide (Korox; Bego, Bremen, Germany) at a pressure of 2 bar (Basic Classic Two Tanks Sandblaster, Hilzingen, Germany), distance of 10 mm, and perpendicular to the surface for 10 seconds.

Glaze layer & HF application group (G): A thin layer of

Table 1. Composition of materials used in the study

Material	Type	Composition	Manufacturer	Batch No.
Porc-Etch	Hydrofluoric acid	Hydrofluoric acid (9%)	Reliance Ortho Prod. Inc., Itasca, IL, USA	132584
Z-Prime Plus	Primer	Ethanol (< 90%) MDP (< 10%) BPDM (< 10%)	Bisco Inc., Schaumburg, IL, USA	1500005281
Porcelain Primer	Silane coupling agent	Acetone (> 45%) Ethanol (> 45%) Silane (> 1%)	Bisco Inc., Schaumburg, IL, USA	1500002122
Porcelain Bonding Resin	HEMA-Free, hydrophobic bonding resin	Bis-GMA (30-50%) UDMA (30-50%) TEGDMA (10-30%)	Bisco Inc., Schaumburg, IL, USA	1500000277
Tescera Indirect Composite System	Reinforced microfill composite (Body)	Ethoxylated Bis-GMA (< 15%) UDMA (< 15%) Glass filler (< 80%) Amorphous silica (< 25%)	Bisco Dental Product Asia Ltd., Seoul, Korea	1400001251

MDP: 10-methacryloyloxydecyl dihydrogen phosphate, BPDM: Biphenyldimethacriylate, Bis-GMA: Bisphenol A diglycidylethermethacrylate, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate

a feldspathic glazing ceramic powder (65% silica, 15% alumina, 10% sodyum oxide, 5% potassium oxide, and 5% titanium oxide) mixed with stain liquid (Vita Akzent Plus; Vita Zahnfabrik, Bad Säckingen, Germany) was carefully applied as a thin coat using a ceramic brush and allowed to air-dry. The specimens were heated up to 750°C and fired for 20 minutes using a computer-programmed electrical induction furnace (Programat P300; IvoclarVivadent AG, Liechtenstein, Austria) then cooled at room temperature. Then, the specimens were conditioned with HF (9% Porc-Etch; Reliance Ortho Prod. Inc., Itasca, IL, USA) for 90 seconds and rinsed with water for 30 seconds.

Sandblasting + Glaze layer & HF application group (SG): The surfaces of specimens were sandblasted as mentioned above, cleaned ultrasonically for 10 minutes and dried. After, glaze layer & HF application was performed on the surfaces as noted above.

The surface of monolithic zirconia specimens for control (C) and sandblasting (S) groups was treated with 1 coat of Z-Prime Plus (Bisco Inc., Schaumburg, IL, USA), uniformly wetting the surface and oil-free air was gently blown for 3-5 seconds. However, in glaze layer & HF application (G) and sandblasting + glaze layer & HF application (SG) groups, a thin layer of Porcelain Primer (Bisco Inc., Schaumburg, IL, USA) was applied to monolithic zirconia surfaces according to the manufacturer' recommendation. Then, Porcelain Bonding Resin (Bisco Inc., Schaumburg, IL, USA) was applied for 10 seconds, with oil-free air gently blown across the surface to evaporate the solvent, and photo-cured for 10 seconds (Valo Cordless; Ultradent Products Inc., South Jordan, UT, USA).

A silicon tubes with a hole in the center (3.2 mm in internal diameter and 3 mm in height) was placed on the specimen surface and incrementally filled with reinforced microfill composite (Tescera Indirect Composite System, Body; Bisco Inc., Schaumburg, IL, USA). The light source (Valo Cordless; Ultradent Products Inc., South Jordan, UT, USA) was positioned to be in contact with the fluid path and the indirect composite material was photo-cured at 20 seconds. After, specimens were polymerized under pressure and light during approximately 2 minutes using Tescera ATL Light Cup (Bisco Inc., Schaumburg, IL, USA). Final polymerization was completed during 10-13 minutes using Tescera ATL Pressure/Light/Heat Cure Cup (Bisco Inc., Schaumburg, IL, USA).

All specimens were subjected to 1000 cycles of thermal cycling in deionized water from 5°C to 55°C, with 30 seconds dwelling and 15 seconds transfer times (SD Mechatronic Thermocycler; SD Mechatronic GmbH, Westerham, Germany).

SBS was determined using a universal testing machine (Shimadzu AG-X, Shimadzu Corporation, Kyoto, Japan). A knife-edge shearing rod at a crosshead speed of 0.5 mm/min was used on the bond interface. Maximum shear load at the point of failure was recorded. SBS (σ) was calculated using the load at failure (F) and the adhesive area (A): $\sigma = F/A$ (N/mm^2).

New specimens were prepared to evaluate changes in surface topography after surface treatment. The specimens were sputter-coated with gold/palladium (Polaron, Emitech Ltd., Kent, UK) and examined with a SEM (JSM-6060LV, Jeol, Tokyo, Japan) (×1000). Moreover, the fractured surfaces of the monolithic zirconia specimens were examined with a stereomicroscope (Leica model, Leica QWinV.3 software, Leica Microsystem Imaging Solutions, Cambridge, UK) (×10) and scanning electron microscope (×25, ×350 and ×1000) after the SBS test. Failure types were classified as adhesive (at the monolithic zirconia-indirect composite material interface), cohesive (within the monolithic zirconia or indirect composite material), or mixed. In cases of mixed failure, the surface of the monolithic zirconia material was partially covered by the remaining indirect composite and/or adhesive material.

Data were analyzed using SPSS for Windows version 22.0 (SPSS Inc., Chicago, IL, USA). The normal distribution of data was firstly checked and verified using the Kolmogorov Smirnov test. One-way analysis of variance (ANOVA) and Tukey LSD tests were used, where $P < .05$ was accepted as significant.

RESULTS

The one-way ANOVA test indicated that the difference among the groups was statistically significant ($P < .05$). Table 2 displays the mean SBS values and standard deviations for all groups. There were statistically significant differences among the groups ($P < .05$), except G and SG groups ($P > .05$).

The results of failure type assessments after SBS test are summarized in Table 2. Mixed failure was observed in tested groups, excepting the C and S groups in which both adhesive and mixed failures were observed. Cohesive failures of the monolithic zirconia or indirect composite material failures were not found.

Fig. 1A shows the SEM image of specimen in control group characterized by a relatively smooth surface. There were marks occur in the surface of the specimen during cutting and grinding. After sandblasting, the zirconia surface was uniformly rougher and exhibited surface textures with sharp edges and shallow pits (Fig. 1B). The specimens of the G and SG groups exhibited different images from other groups. SEM images of these specimens displayed the formation of micropores at different widths and depths, due to the dissolution of glass matrix depending on the HF application (Fig. 1C and Fig. 1D).

Fig. 2A show SEM image of the surface applied

Table 2. The groups of study, the results of shear bond strength test (MPa) and failure type

Surface treatment	Adhesive system	Composite material	Group	Mean (SD)	Failure type	
					Adhesive	Mixed
Control (No treatment)	Z-Prime Plus		C	0.92 (0.69)^{A}	3	9
Sandblasting	Z-Prime Plus		S	12.49 (2.70)^{B}	1	11
Glaze layer & HF application	Porcelain Primer + Porcelain Bonding Resin	Tescera Indirect Composite System	G	18.41 (3.99)^{C}	-	12
Sandblasting + Glaze layer & HF application	Porcelain Primer + Porcelain Bonding Resin		SG	17.35 (6.73)^{C}	-	12

Same uppercase letters were not significantly different at $P < .05$.
SD: Standard deviation.

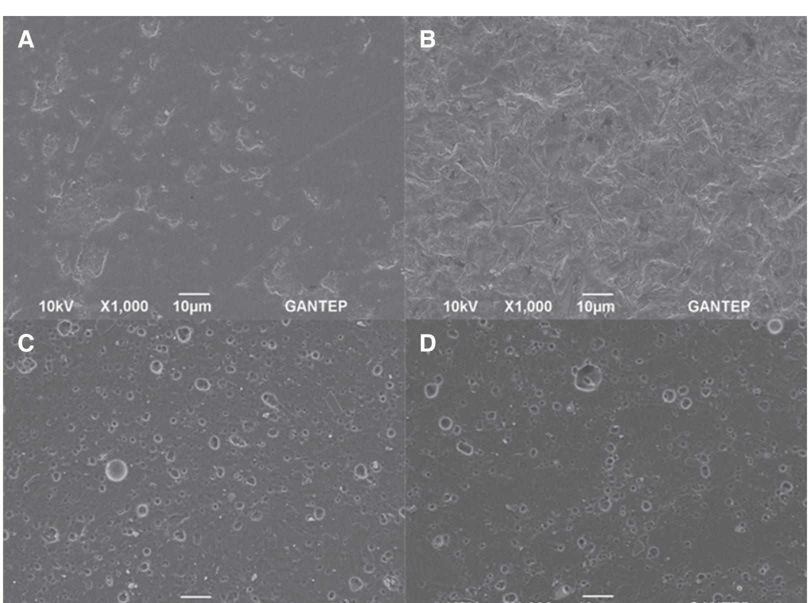

Fig. 1. SEM micrographs of monolithic zirconia specimens: (A) control, (B) sandblasting, (C) glaze layer & HF application, (D) sandblasting + glaze layer & HF application.

Z-Prime Plus after grinding. No scratches generated by the abrasive papers can be seen. In the C group, the SEM images of the fracture interface revealed remains of the priming agent and the indirect composite material on the surface of specimen (Fig. 2B and Fig. 2C).

Figs. 3A and 3B are SEM images of the sandblasted group after SBS test. The remnants of adhered material on the surface of the specimen indicated mixed failure observed. Both of glaze layer & HF application treated groups exhibited mixed type of the failure (Fig. 3C and Fig. 3D).

DISCUSSION

The present study aimed to estimate the availability of indirect composite material veneered monolithic zirconia prostheses. The null hypothesis that different surface treatments have no effect on indirect composite material-monolithic zirconia bond strength was rejected. In the present study, the G and SG groups had significantly higher bond strengths than the other two groups. However, there was no significant difference in bond strength between the G and SG groups.

Previous studies have shown that indirect composite material produced low bonding strength values when applied to untreated zirconia ceramic surfaces.[23] The effect of priming agent on the bond strength of indirect composite material to zirconia ceramic has also been evaluated[19-21] and it has been reported that it could increase the bond strength.[18,21]

In the groups G and SG, since the glass phase was created on the surface, Porcelain Primer and Porcelain Bonding Resin were applied to the surfaces while Z-Prime Plus was applied to the monolithic zirconia specimens in the C and S groups according to the manufacturer's instructions. In this study, untreated monolithic zirconia specimens used to determine the effect of the priming agent and this group

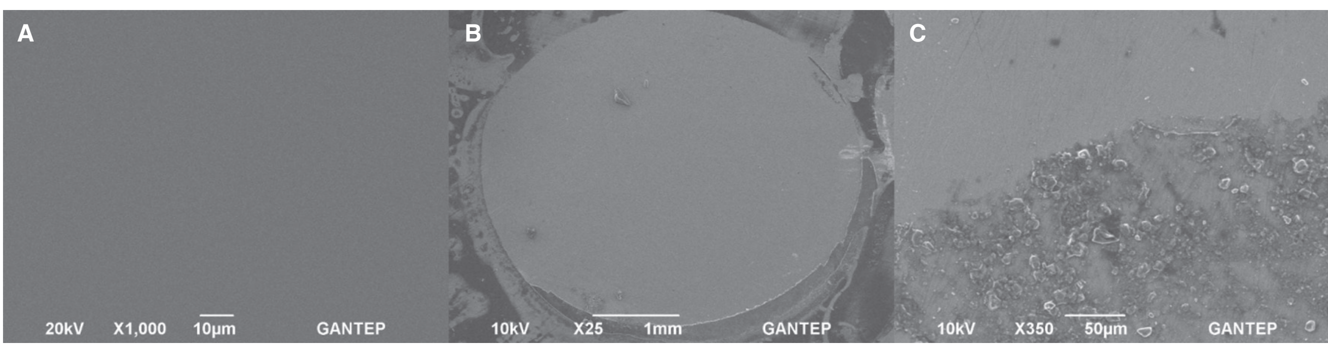

Fig. 2. (A) SEM micrograph of monolithic zirconia specimen applied Z-Prime Plus, (B) control group exhibited mixed failure, (C) in the surface of a specimen from the control group, both monolithic zirconia and remnants of Z-Prime Plus and indirect composite material were visible.

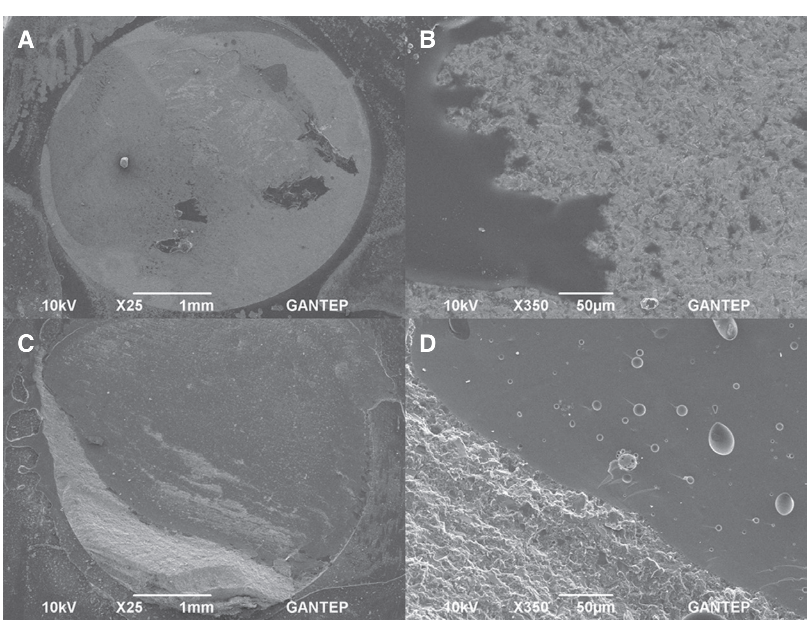

Fig. 3. SEM micrographs of monolithic zirconia specimens. (A) sandblasted group exhibited mixed failure, (B) remnants of Z-Prime Plus and indirect composite material were seen in the surface of sandblasted specimen, (C) glaze layer & HF application group exhibited mixed failure, (D) in the glaze layer & HF application group, the indirect composite material on the monolithic zirconia was observed.

had the lowest bond strength.

Sandblasting creates surface roughness by cleaning the surface of framework and increases the mechanical or chemical bond strength between metal or other substrates and composite material.[21] There were significant differences in bond strength between C and S groups in present study. The bond strength value was higher after sandblasting. According to this result, it can be said that sandblasting significantly affects bond strength and producing a stable bond without appropriate surface treatment may be difficult. This was due to a synergistic effect produced by the increased contact area on the chemical interactions between the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and carboxylic monomers of the Z-Prime Plus and the monolithic zirconia surface. The reported data of the present study on the effect of the sandblasting and priming agent application on bond strength is in agreement with previous studies.[18,20,28]

The MDP monomer has a phosphoric acid group which can be bonded to hydroxyl groups in the zirconia surface and a carboxylic acid group will bond to composite resin.[35] Z-Prime Plus is an MDP-containing primer and applied one or two coats without being specific about light polymerization according to the manufacturer's instructions. In a previous study, Seabra *et al*.[35] reported that the higher bond strength was obtained with applying 2 coats of Z-Prime Plus followed by light polymerization compared to the other groups. In present study, one coat of primer was applied and it was not light-polymerized.

Organofunctionalsilanes contain 2 different reactive functional groups that can react and couple with various inorganic and organic materials. They are used as adhesion promoters, increasing the union of dissimilar materials. The reaction of hydrolysable functional groups to the surface hydroxyl groups of inorganic substrates creates a siloxane bond. Organic functional groups react with functional groups of resins.[36,37]

The glaze layer & HF application on monolithic zirconia surface followed by silanization have resulted in a significantly improved bond with indirect composite material. This result is associated with formation of strong chemical bonds between the silane and the silica in the porcelain.[20] This data was confirmed by the findings of previous studies.[20,33] In the present study, a thin layer of low-fusing porcelain was applied to monolithic zirconia surfaces for G and SG groups and specimens were fired. Results of the present study revealed that this procedure, regardless of application of sandblasting, is able to provide a more retentive surface than sandblasted monolithic zirconia. This reasoning is further supported by the prevalence of mixed failures in G and SG groups. Moreover, it can be said that sandblasting before glaze layer & HF application does not enhance the extra micromechanical interlocking.

The data for SBS test were supported by the SEM images. SEM image of S group showed a unique corrugated appearance as shown in previous studies.[20,21,23] This corrugated appearance is considered to indicate the phenomenon of strong adhesion to resist the shear force. This seems to have been a result of the sandblasting rather than the priming agent although the results showed that the bond strength significantly increased compared with the C group. Glaze layer & HF application groups exhibit numerous micropores at different widths and depths, due to the dissolution of glass matrix depending on the HF treatment that was applied. This view is similar to the study of Fushiki *et al*.[20].

In present study, in the groups with the lower SBS values (C and S groups) a few adhesive failures were found, whereas in the groups with higher SBS values (G and SG), only mixed type failures were observed. In G and SG groups, the monolithic zirconia surface is covered with more indirect composite material than the other two groups (C and S). This result is a sign of generating strong adhesion by etched glaze layer.

In vitro studies are necessary for understanding the laboratory performances of materials. However, they are not sound criteria for the prediction of their clinical efficacy. Further studies may be required to compare complete restorations and the effects of mechanical load cycling in artificial saliva. Controlled long-term clinical studies are needed to confirm the success of treatment procedures.

CONCLUSION

Within the limitation of this *in vitro* study, the following conclusions can be drawn: The glaze layer & HF application provides high bond strength between indirect composite material (Tescera Indirect Composite System) and monolithic zirconia (inCoris TZI). Indirect composite materials may be a promising alternative as a veneering material for monolithic zirconia restorations.

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