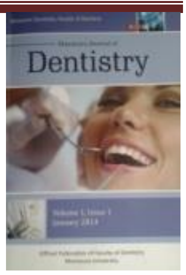

The effect of cleaning methods and primer application on bond strength to zirconia

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Abstract:

Aim: The aim of this in-vitro study was to evaluate the effect of cleaning methods and primer application on bonding to contaminated zirconia.

Materials and Methods: A total of 64 zirconia discs (8mm × 3mm) were fabricated with CAD/CAM. All discs were air-borne particle abraded using 50 m Al_2O_3 particles then divided into two main group according to application of surface cleaning after saliva contamination; A. (control test groups) (n= 16) and B. (contaminated test groups that were divided into three main test groups according to cleaning methods after contamination) (n=48) as follow; [steam cleaning (SC) (n= 16), ultrasonic cleaning (UC) (n=16), Ivoclean cleaning (IC) (n=16)]. Then, each main group was subdivided into two equal sub groups (n=8) according to primer application (Monobond N) or no primer application (NP). Composite resin discs (Nexcomp) were cemented to zirconia discs using adhesive resin cement (Multilink N). All bonded specimens were stored in water bath for 6 months. Shear bond strength (SBS) test was performed afterwards using a universal testing machine. Scanning Electron Microscope was used for failure mode examination.

Results: The statistical analyses were done using two-way ANOVA and serial one-way ANOVAs followed by Post hoc Tukey-HSD test at ($p < 0.05$). The application of primer significantly increased SBS Values of test groups compared to non-primed ones (p=0.000). The highest mean SBS (MPa) was reported for (ICP) test group (15.3±1.6 MPa) followed by (UCP) test group (11.1±2.9MPa). While the lowest mean SBS was observed for (CP) (9.5 ±3.3 MPa). There was statistically significant difference between (IC) and other test groups (C, SC, UC) (p=0.001, p=0.032, p=0.017) respectively. There was no statistically significant difference between cleaning test groups without primer application.

Conclusion: Within the limitations of the current study, primer application has a dual action. It proved effectiveness as a chemical surface treatment method in comparison with airborne particle abrasion (mechanical surface treatment). Surface cleaning methods without primer application were not of value. Ivoclean when conjugated with universal primer, best results obtained compared to the other surface cleaning methods.

Introduction

All ceramic restorations are preferred to be used as dental restorative material and this is simply due to their excellent esthetic and strength properties. [1] Zirconia ceramics are restorative dental materials fabricated by CAD/CAM thus increasing efficiency by automating the manufacturing steps and high-quality restorations can be obtained in a short-predicted time. [2]

Zirconia is a crystalline dioxide of zirconium and it has been an alternative to alumina as biomaterial and is used in dental applications for fabricating endodontic posts, fixed dental prosthesis, implant abutments. "Ceramic steel "or "white steel" is a name referred to zirconia for its excellent mechanical, biological and optical properties. [3]

As for mechanical properties of zirconia ceramics, it is a very strong metal with mechanical properties similar to those of stainless steel. It can tolerate cyclic load stresses well. Also, it is characterized by its high flexural strength and fracture toughness. [4]

Phase transformation is a special character of pure zirconia which mean that material occurs in three crystallographic structures depended on the material's temperature "polymorphism" where the material has the same chemical composition but a different atomic arrangement. At room temperature, zirconia adopts a monoclinic structure and transforms into tetragonal phase at 1170°C, followed by a cubic phase (C) at 2370°C.This phase transformation is accompanied by (3-5) % volume expansion. [5]

Low thermal degradation is another specialized characteristic of zirconia ceramic as it resembles an aging process of zirconia, referring to the surface degradation caused by the grain pullout and a subsequently micro cracking of the structure mainly due to the presence of water. When the humidity raises even with low temperatures and leads to potential microcracking and decreasing strength of zirconia. It is also considered a time dependent process that develops from the outside and continues inside the restoration. The presence of water makes the transformation develops faster. [6]

When talking about biological properties of zirconia, it was proved that zirconia is a biocompatible material as in vitro and in vivo studies have confirmed that no local (cellular) or systemic adverse reactions to zirconia ceramic were reported.

The current available approaches for bonding to zirconia are not adequate and the long-term durability for clinical application is unknown. Traditional adhesive techniques used with silica-based ceramics do not work effectively with zirconia because of absence of silica in the zirconia microstructure makes HF acid etching is not applicable for use with zirconia.

Because of chemical inertness and nonpolar zirconia surface [7], many surface treatments [8,9] techniques are indicated to improve adhesion between zirconia and adhesive cements. Chemical modification of the surface and micromechanical interlocking through air abrasion, or a combination of both are the main ways for improvement of bonding to zirconia ceramics.

Air-borne abrasion with aluminum oxide particles is commonly used as a (micromechanical surface treatment) suggested by many researches as an effective method [10] in roughening and cleaning the bonding surface of zirconia. [11,12] While, others showed that it could lower the bond strength values or lead to spontaneous deboning after artificial aging (150 days of water storage and repeated thermocycling). In addition to negative results of sandblasting on mechanical properties of Y-TZP materials obtained from long-term performance due to growth of the sandblast flaws. [13,14]

Reducing air pressure accompanied by other surface treatment methods is another suggestion to reach high values of bond strength. [15] The combination of air-abrasion and priming improved long-term resin bonding to zirconia ceramic significantly. With low-pressure air-abrasion, surface roughness was reduced without affecting long-term bond strength, provided that adequate adhesive primers were applied.

Primer is demanded for successful bonding to non-silica oxides (zirconia and alumina) [16,17] and are indicated for improving the bond to both direct and indirect substrate. Primers are adhesion promoter that provide chemical bonding between dissimilar substrates. All primers work through improvement of wettability of the bonding surface. [18] They are also substrate specific agents. Chemical bond strategies of zirconia primers depend on the presence of adhesive monomers such as MDP, 4-META, MEPS, and zirconate coupler. [19]

Universal zirconia primers designed specifically for chemical bonding to zirconia and enhancing the resin-zirconia bond strength. [20] Additionally, they can be used for bonding to metal and alumina, and for intraoral repair of restorations. All previous studies confirmed the importance of primer application specially the universal primer. They concluded that better long term results obtained from using a universal primer than did a conventional silane.[15] This was due to the content of different methacrylate monomers with a functional phosphoric acid group in universal primer which is responsible for a very stable phosphate link and resistant to hydrolysis.[21] They also mentioned that premature deboning occurred solely in unprimed specimens which means that airborne particle abrasion alone is not sufficient for zirconia surface pretreatment.[22]

Other studies mentioned the importance of combination of reduced air-borne abrasion and primer application and the explanation was that the presence of phosphate monomers associated with air-abrasion procedures can provide more stable adhesion of resin cements to zirconia in oral conditions. [23,24]

Universal primers that contain MDP monomer and a silane monomer 3-MPS in their chemical composition can chemically bond with the metal oxides at zirconia ceramic surface and provide initial high bond strength to zirconia through van der Waals forces or hydrogen bonds at the resin /zirconia interface. A bio-functional monomer, 10-methacryloyloxydecyldihydrogen phosphate (MDP) conjugated with 10-MDP resin cement resulted in more reliable adhesion to zirconia that also could be an alternative to air-abrasion protocols. [25]

Previous studies confirmed that presence of primer is more important than the type of cement itself. [26] They confirmed that using conventional resin cement alone is not sufficient for long-term adhesion to ceramic and a combination of both ceramic primer and self-adhesive resin composite cement was better to maintain long term durability of bonding.

Contamination is one of the most common problems faced during bonding restorative materials, which can lead to decrease in the bond strength values. During routine dental procedures, the bonding surfaces of ceramic restorations are often exposed to contamination.

Saliva has a low surface energy that weakens the adhesive materials. Glycoprotein of saliva decreases the efficacy of interaction between the composite resin and the tooth. There are two types of contamination affect modified zirconia surfaces 1). organic (human blood and saliva) 2). inorganic contaminants (type IV dental stone).

Different types of cleaning agents were used such as etching with phosphoric acid, cleaning in an ultrasonic bath in ethanol, water washing, re-application of primer, airborne particle abrasion, steam cleaning and newly chemical cleaning paste (Ivoclean).

The effect of different cleaning methods is controversial as previous studies minimized their role in improving the bonding of zirconia have little or no effect on long-term resin bonding to zirconia ceramic comparing to a universal primer application which improved bonding to zirconia. [27] Others confirmed that airborne-particle abrasion and (Ivoclean) paste were effective in cleaning the zirconia surface. Airborne-particle abrasion yielded the highest SBS value among the groups. [28]

Long-term water storage was used to simulate the aging of resin bonding. Artificial aging also affects the durability of cleaning methods (water-rinsing, k-etchant GEL phosphoric acid and Ivoclean). [29]

MATERIALS AND METHODS

Materials utilized in this study and their main ingredients are demonstrated in (table 1).

Table 1: Description of materials used in the study.

Material	Composition	Product Description	Manufacturer	Lot number
Ceramill Zolid HT+	$ZrO_2 + HfO_2 + Y_2O_3$: ≥ 99.0 Y_2O_3: 6,0 - 7,0 HfO_2: ≤ 5 Al_2O_3: ≤ 0.5 Other oxides: ≤ 1	Zirconium oxide (ZrO_2) ceramic blocks of CAD-CAM	Amman Girrbach AG, Austria	1907001
Ivoclean Refill	Standard composition (in wt%) Zirconium oxide 10- 15 Water 65 - 80 Polyethylene glycol 8 - 10 Sodium hydroxide ≤ 1 Pigments, additives 4	Universal cleaning paste	Ivoclar Vivadental AG, Lichtenstein	X00617

	- 5			
Monobond® N	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylat	Universal primer, one component universal restorative primer.	Ivoclar Vivadental AG, Lichtenstein	X17917
Multilink® N	Monomer matrix consist of -Dimethacrylate, HEMA, In organic fillers - barium glass, ytterbium trifluoride, spheroid mixed oxide	Self-cure luting composite with light-curing option for the adhesive luting of indirect restorations.	Ivoclar Vivadental AG, Lichtenstein	W40132
Nexcomp	Bis-GMA, UDMA, Bis-EMA Borosilicate glass	Nano hybrid composite resin	META® BIOMED, Korea	NXC 1712112
Ethyl Alcohol (Ethanol) (absolute)	Simple alcohol with the chemical formula C_2H_6O	Dehydrated alcohol volatile and colourless liquid. Commonly, used in synthetic organic reactions in both industry and science.It also considered as a solvent of non-polar substances.	El Nasr pharmaceutical chemical company, Egypt	E0058111

Specimen Preparation:

Sixty-four discs (n=64) of yttria-stabilized zirconia (diameter: 8mm; thickness:3mm) were fabricated using CAD-CAM technology. The wax pattern of disc was fabricated then scanned using Ceramill® Map 400+. Milling of the zirconia discs were done from zirconia blank (Ceramill Zolid HT+, Amman Girrbach AG, Austria) by using ceramal® motion 2 CAD-CAM machines. Discs were sintered in high temperature furnace (ceramill therm, Amann Girbach) according to manufacturer's recommendations. The temperature was raised to 1500°C in 2 hours then kept at a final temperature (1500°C) for another 2 hours. Specimens were slowly cooled to less than 100°C in 1 hour. Surface conditioning of all discs were air abraded with 50-µm aluminum oxide particle (Basic Eco SandBlaster, Renfert, Hilzingen, Germany) for 15 s, under 2.5 bars pressure and from a distance of 10 mm. Then they were cleaned ultrasonically in (%95) alcohol for 5 min. Contamination Protocol and Experimental Design followed air-abrasion, zirconia discs were divided into eight groups according to the experimental design. Forty-eight specimens were immersed in (20) mL artificial saliva (acc. to Afnor) for (3) min. While 16 specimens were kept without contamination act as control group.

Composition of artificial saliva used in the study was as follow:

700 mg/L NaCl+ 1200 mg/L + KCl 260 mg/L +Na_2HPO_4 +1500 mg/L +$NaHCO_3$ 330 mg/L +KSCN 1300 mg/L + Urea (CH_4N_2O) of total PH= 6.7

Saliva contaminated zirconia discs were divided into three main test groups according to cleaning methods after saliva contamination (n= 48) as follow:

Group 1 (S): Steam cleaning (n=16).

After saliva contamination, zirconia discs were exposed to steam cleaning by a (steam cleaner, Tianjin, china) of (4 Bar) pressure and a water container of (200) ml

(figure7). It works through releasing streams of steam on the contaminated zirconia discs then air-drying with oil free air for 10 s.

Group 2 (U): Ultrasonic cleaning (n=16).

After saliva contamination, zirconia specimens were immersed in ethyl alcohol of (99.5%) concentration for 10 min in digital ultrasonic cleaner (MCS, Egypt).

Group 3 (I): Chemical cleaning with Ivoclean (n=16).

Specimens were cleaned with a commercial cleaning paste (Ivoclean) (Ivoclar-Vivadent, AG) according to manufacturer's instructions.

Each main test group was divided into two subgroups (n=8) according to primer application

Subgroup P: Primer application following cleaning procedures.

Subgroup NP: No primer application following cleaning procedures.

Primer application: Considering control and test subgroups with primer application. A thin coat of Monobond N was applied.

Composite resin discs preparation:

A total number of 64 composite resin discs were fabricated using plastic mold of 4 holes. Each hole has 4 mm internal diameter from the center and 3 mm thickness. The holes were filled with light cure composite resin in three increments (Nexcomp shade A2, META® BIOMED, Korea) to fabricate composite resin discs. Composite resin disc was light cured with (Blue LEX LD-105, MONITEX, Taiwan) for 20s for each increment.

Bonding procedures:

After the discs received the assigned cleaning regimen, composite resin discs were cemented to previously treated zirconia discs using adhesive resin cement (Multilink®N, Ivoclar vivadent, Liechtenstein) according to the manufacturer instructions.

Specimens storage: Two hours after cementation the specimens were stored in water at 37^0C for 6 months.

Shear bond strength test: The bond strength between zirconia and composite discs was determined by a shear bond test.

Mode of failure: The interfaces of the debonded samples were examined to determine the failure pattern.

Scanning electron microscopy: (SEM) Representative specimens of each test group were examined under a scanning electron microscope (SEM) (Quanta 250-FEG, FEI, Netherlands) with accelerating voltage 30 K.V.

RESULTS

Shear bond strength_results

1)- Descriptive statistics

Table (**2**) showing mean and standard deviation (SD) of shear bond strength values (MPa) for all tested groups. Considering cleaning methods + primer application, (Ivoclean cleaning + primer application test group) showed the highest SBS value (15.3±1.6) followed by ultrasonic cleaning + primer application test group (11.1±2.9). On the other hand, control + primer application group showed the lowest SBS mean value (9.5 ±3.3) as shown in table (2) figure (26). Considering cleaning methods + no primer application steam cleaning + no primer application test group showed the highest SBS value (1.6±0.5) followed by Control + no primer group (1.2±0.4). There was no statistically difference between test groups as shown in table (2). The overall SBS values of (different surface cleaning methods+ no primer application) were low in comparison with (different surface cleaning methods+ primer application) as seen in figure (1,2,3,4).

Table (2) showing: Mean and Standard deviation (SD) of shear bond strength values (MPa) for all tested groups.

Surface cleaning methods / Primer application	**P**	**NP**
	Mean ±SD	Mean±SD
C	9.5 ±3.3	1.2±0.4
SC	10.8 ± 2.8	1.6±0.5
UC	11.1±2.9	1±0.5
IC	15.3±1.6	1± 0.6

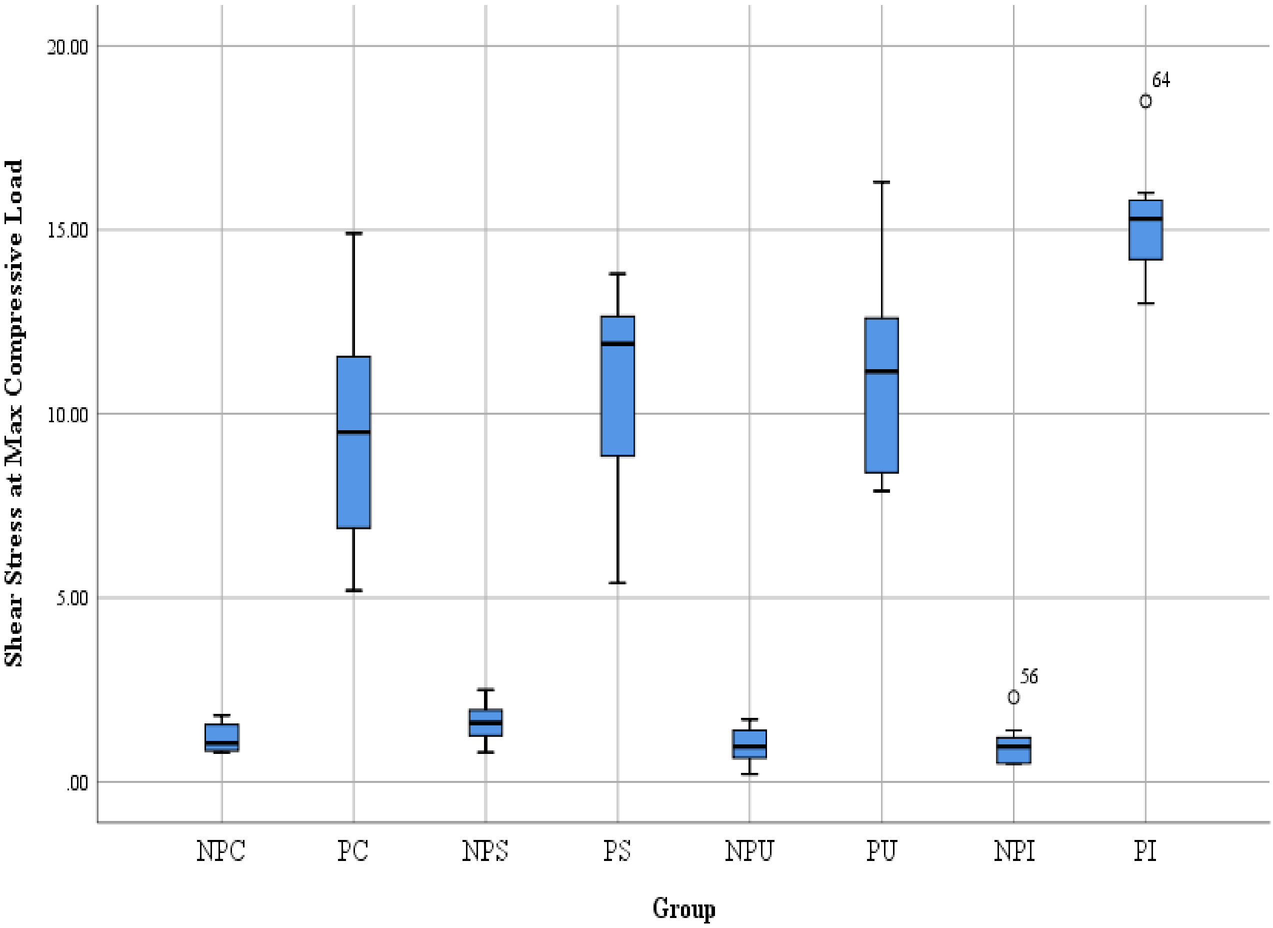

Figure 1: Box Plot for test and control groups with and without primer application.

Failure Mode

Failure pattern of all deboned specimens showed adhesive, cohesive and mixed failure patterns as shown in figure (1-4)

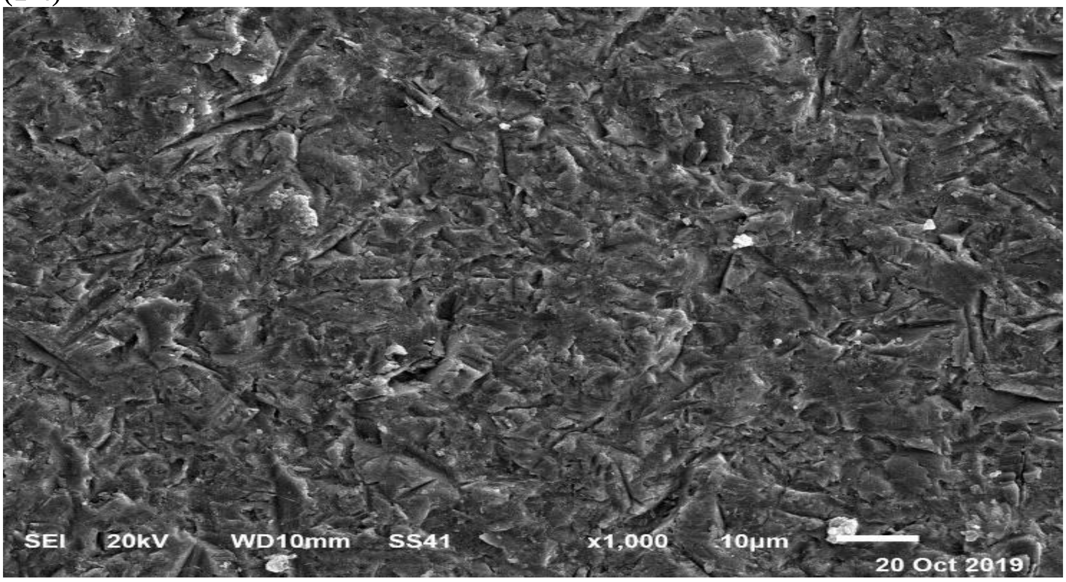

Figure 2: SEM image showing adhesive failure at composite/zirconia disc interface.

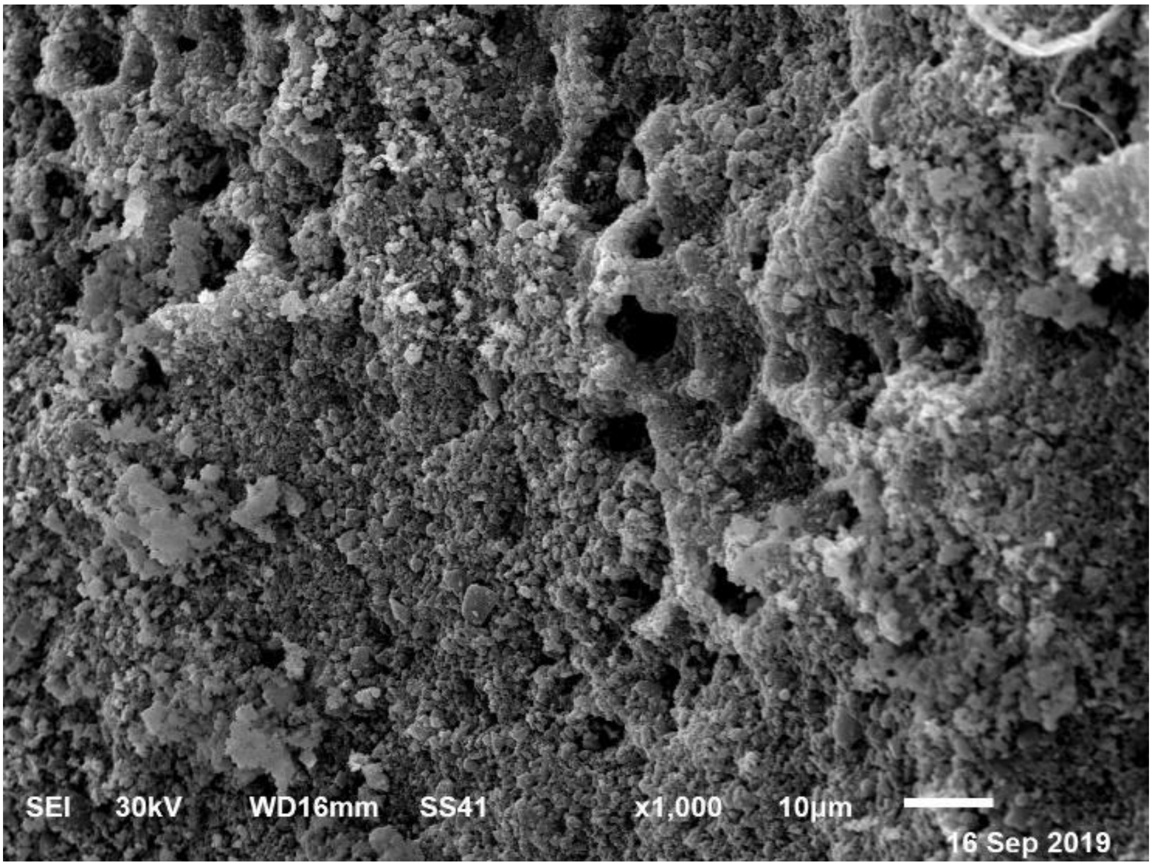

Figure 3: SEM image showing cohesive mode of failure in composite resin.

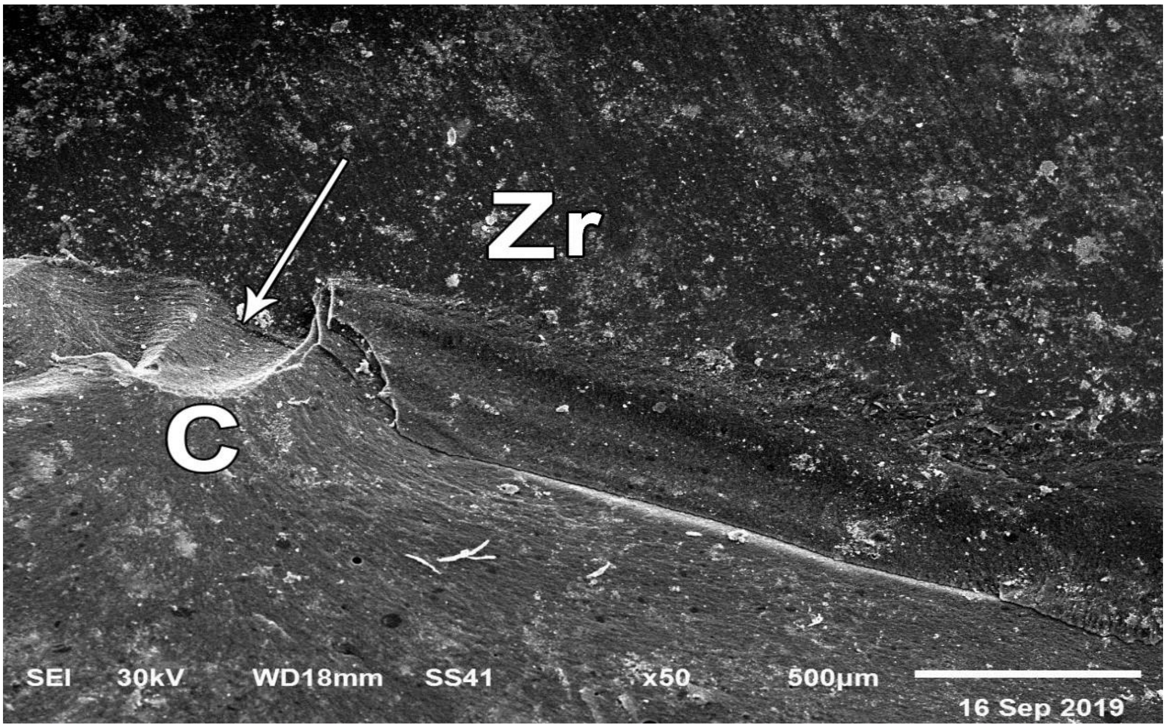

Figure 4: SEM image showing mixed mode of failure
NP: The arrows represent interfaces between composite resin discs ruminants (C) and zirconia (Z).

DISCUSSION:

The current study evaluated the effect of cleaning methods and primer application on bonding to contaminated zirconia. The first null hypothesis was accepted as the cleaning methods had no effect on improving of bond strength values. While, the second null hypothesis was rejected as the primer application had an essential role in improvement of bond strength to zirconia.

Zirconia ceramics have marvelous advantages over other types of ceramics [but] their bonding is still a problematic issue. For this reason, several attempts [are] carried out to achieve a strong and durable bond that insures the long-term clinical performance of zirconia ceramics.

Air-borne abrasion is considered as an effective mechanical method for modifying zirconia ceramic surface through increasing surface energy and surface area for bonding and wettability. However, using of air-borne particle abrasion is controlled by specific parameters. [(30)] Any differences lead to contradictory results. It may also trigger the phase transformation (T-M) on the surface of Y-TZP that creates stresses within the bonding substrates. Eventually, it could compromise the long-term stability and reliability of zirconia ceramics.

The results of current study showed that bond strength values obtained from control tested group without primer application (CNP) were (1.2±0.4 MPa) confirmed that, using of air abrasion alone as a surface treatment method is not enough to maintain a strong and durable bond strength between zirconia and resin luting agents and this is consistent with previous studies. [(14)]

There is no doubt that, air abrasion can be used as an effective cleaning method as it cleans the inner surface of restorations. It is considered as the most effective cleaning methods and there was a consensus from previous studies that air-borne abrasion increases the surface area and chemical activation of the bonding surface because it removes organic contaminants.

Based on findings of this study, control test group that received only air-borne abrasion with no primer application (CNP) recorded low bond strength value (1.2±0.4) compared to control test group with primer application (CP) (9.5 ±3.3 MPa). Importance of air-abrasion is limited to clean zirconia ceramic surface and primer application is mandatory.

Enhancing the chemical bonding by primer application which considered as (adhesion promoting agents) is essential as they can replace air-borne abrasion [(16)] without loss of zirconia bonding strength. In the current study, the effect of primer application (chemically) surpassed the air-borne abrasion method (mechanically) as bond strength values of all tested groups received primer application were (CP= 9.5 ±3.3, SCP=10.8 ± 2.8, UCP=11.1±2.9, ICP=15.3±1.6 MPa) that higher than those without primer application (CNP=1.2±0.4, SCNP=1.6±0.5, UCNP= 1±0.5, ICNP=1± 0.6 MPa) and this was agreed with previous studies. [(22,31,32).]

Surface treatment of low-pressure abrasion protocol or grinding followed with (Monobond N) universal primer application gave the similar shear bond strength values of the high-pressure abrasion protocol. Monobond N is a universal primer was used in this study that consists of silane methacrylate for glass ceramic, sulfide methacrylate for precious metal alloys, and phosphoric acid methacrylate for oxide ceramics and base metal alloys. (7,16) Zirconia ceramic is composed of a glass-free material which means that the silane methacrylate in primer composition has no effect. Methacrylate monomers with a functional phosphoric acid group in (Monobond N) are used to establish the bond to zirconia (methacrylate zirconium oxide compound) as zirconia has high affinity to phosphoric acid representing a typical trait of zirconia ceramic.

Previous studies explained that the affinity of zirconia to phosphoric acid was due to the ability the phosphate group to react with zirconium, forming zirconium phosphate, where each phosphate group is bound to three zirconium atoms (tridentate bridging mode) or to one zirconia atom (tridentate chelating mode) resulting in a thermally and hydrolytically stable interface. (7)

The capacity of zirconia to constitute chemical interaction with cements is limited. Resin cements with dual activation are the most indicated for zirconia cementation than other cements that are chemically or photo-activated. As for self‑adhesive cements, they promote adequate adhesive resistance to zirconia. (33)
The preferred protocol for resin bonding to zirconia is the combination of surface roughness and treatment with a phosphate-containing zirconia primer followed by cementation with hydrophobic non-phosphate-containing resin cement. (22) Application of self-adhesive resin cement without pretreatment was not sufficient for improving the strength of the bonding to an untreated zirconia ceramic surface. (34)

Multilink N is a self-adhesive resin cement used in this study, does not contain (MDP) in its composition. It presented significantly better bond strength values than those of the other systems when used with universal primer (Monobond N) which composed of three different functional monomers as previously mentioned. (35,36) when phosphate monomers in universal primer associated with air-borne particle abrasion procedures, they provided more stable adhesion of resin cements to zirconia after aging. (26,35) Zirconia-based dental ceramic bonded with Multilink resin cement had the highest microtensile bond strength. (37،38) However, previous studies confirmed that the type of cement had no effect on bond strength to zirconia (35) and this comes in line with current study.

During try-in the luting surfaces of ceramic restorations get contaminated with saliva, blood, or silicone indicators. (39) The type of contamination determines the method of cleaning. So, maintenance of zirconia intaglio surface (inner surface of zirconia) free of contaminants is a challenge. (27)

Saliva contamination considered one of the main causes of reducing resin bond strength. It adversely affects resin bonding to zirconia because it leaves an organic adhesive coating in the first few seconds of the exposure which is resistant to washing. (29) Artificial saliva was preferred to be used in this study than natural saliva to ensure reproducibility and standardization of experiments. Moreovever, artificial saliva contamination did not affect the SBS of zirconia ceramics.

Previous studies stated the role of cleaning methods in improving bond strength to zirconia so, many different cleaning methods were applied. [28] So, cleaning agents can improve the adverse effect of saliva contamination on zirconia, but this effect varies depending on the cleaning method.

Ivoclean is a chemical cleaning paste which composed of an alkaline suspension of zirconium oxide particles. So, it acts like a" sponge" as phosphate contaminants from saliva absorbed by particles of Ivoclean than ceramic surfaces and leaves behind a clean zirconium oxide. It also has high pH which might be responsible for improving the bond strength [40] when used in combination with universal primer. Because of application of MDP in alkaline conditions showed higher bond strength than that obtained in acid conditions that allowed better formation of MDP-zirconia bonds.

Application of zirconia primer to the sandblasted zirconia surface is recommended by previous studies whether the surface is contaminated with saliva or not. [41,42] According to the results of this study, Ivoclean+primer application tested group (ICP) recorded the highest bond strength value (15.3±1.6 MPa) among other cleaning methods (CP= 9.5 ±3.3, SCP=10.8 ± 2.8, UCP=11.1±2.9 MPa). Ivoclean has been proved to maintain adequate SBS values after 150 days of storage comparable to the uncontaminated zirconia. [43-45]

Using of ultrasonic cleaning as a surface cleaning method of zirconia ceramic is controversial. Some researchers found that it was more effective on polished zirconia surface. While, others reported that ultrasonic cleaning of dental restorations after sandblasting should be avoided because it decreased the adhesive strength of resin luting material.

Similarly, using steam cleaning is also controversial, as application of steam cleaning had a positive influence on bond strength between porcelain and titanium with less adhesive failure. [46] However, it can abrade the surface of type III dental stones and cause weight loss. Our study findings agreed with previous results which showed that different cleaning methods had no effect on bond strength without primer application. [27,47]

Long-term water storage and thermocycling are commonly used methods of artificial aging that affect the resin bond to ceramic. Previous studies confirmed that water storage for a period of one month was the cut off value and time interval is very important to initiate degradation of ceramic-cement interface. Water storage is simple, low-cost and less aggressive than thermocycling which lacks standard agreement. Water absorption may cause the resin cement layer to thicken and expand, thereby disrupting the established bond and increasing bond degradation. Water hydrolysis may also break chemical covalent bonds created by conditioning primers. [48]

The shear bond strength test has been considered the most common laboratory technique for evaluating adhesives of resin-bonded ceramic restorations and ceramic repair systems. This test was used for measuring bond strength in a large number of studies. [49]

The examination of the failure mode patterns showed that nearly all failure modes in unprimed specimens occurred at the ceramic/resin interface (adhesive failure type) which considered an indication of weak bond strength. While, all primed ones showed mixed mode of failure. All previous studies agreed with the results of the current study. [19,21]

CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn:

1) Universal primer is considered an ideal surface treatment method (chemically) than air-borne particle abrasion method (mechanically).

2) Ivoclean when conjugated with universal primer best results obtained compared to other cleaning methods used in this study.

3) There is no value of different cleaning methods used in the current study without using universal primer.

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