Original Article

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Bond Strength to Zirconia Ceramic after Different Surface Treatments

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Abstract:

Purpose: This study investigated tensile bond strength to zirconia after different surface treatment and effect of chemical conditioning. **Methods:** 64 Zirconia discs were fabricated and categorized according to the applied surface treatment into four groups**:** as milled, sandblasted with AL_2O_3, treated with ammonium bi-fluoride, treated with Hydrofluoric acid in the form of with zirconia etchant cloud system. Followed by primer application to the half of the specimens or no primer application to the other half, left with only surface treatment. Composite resin discs were fabricated and bonded to Zirconia using adhesive resin cement. All specimens were artificially aged. Statistical analysis was done with post hoc test using LSD. **Results:** the surface treatment did not show significant difference between all tested groups (P value = 0.467). While, the chemical conditioning showed a significant effect on the tensile bond strength measurements (P value = 0.014). No interaction effect exist between surface treatment and chemical conditioning (P value = 0.144). **Conclusion:** Sandblasting and hydrofluoric acid resulted in roughening zirconia surfaces and confirmed by SEM. Chemical conditioning has significant effect on the bond strength of resin cement to zirconia ceramic.

Key words: Chemical conditioning, Resin cement, tensile, CAD/CAM, artificial aging, bonding.

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1. Introduction:

Zirconia has become a prominent material in restorative dentistry due to its exceptional mechanical properties and biocompatibility. CAD-CAM fabricated dental restorations helped that these materials are being utilized more frequently for prosthetic restorations [1,2,3].

Resin cements can be used for cementation of zirconia restorations in cases that require higher retention offering better marginal adaptation and improved longevity of the ceramic restoration [4,5]

Different surface treatment methods were used to modify zirconia surfaces to evaluate their impact on adhesion to resin cement [5,6,7]. The most popular technique for zirconia surface treatment is airabrasion [8,9]. Sandblasting induce zirconia surface crystalline structure to transform from tetragonal phase to monoclinic phase which will result in micro-cracks formation which in turn would reduce the zirconia stability [10,11]. This occurs when the application distance is reduced, and the particle size is increased, leading to the formation of micro-cracks. These limitations weaken the ceramic's long-term mechanical properties and make it challenging to achieve uniform surface treatment in dental clinics [9,12].

Great interests have been raised in the modification of zirconia surfaces, some studies tried techniques like acid etching.[13,14] Chemical bond formation and micromechanical interlocking are important for achieving strong bond between zirconia ceramics and resin cement.[15] Multiple couplers and silane primers containing acidic groups such as 10-methacryloxydecyldihydrogen phosphate (MDP) were developed to enable the chemical bonding to zirconia [16–19].

This in vitro study had the purpose of evaluating three surface treatment methods: Sandblasting, etching by ammonium bi-fluoride and zirconia etchant cloud system with or without the use of chemical conditioning agent.

2. Materials and Methods

2.1. Zirconia discs preparation

Discs (n = 64) were milled from IPs Emax-zircad Zirconia blanks (IvoclarVivadent, Schaan, Liechtenstein) with diameter: 8 mm and thickness: 3 mm using Roland DWX-52D CAD/CAM machine. Then, sintering of the zirconia discs was carried out as follow: The zirconia discs were placed on the firing tray of the furnace. Sintering cycle was as follow; temperature was raised in two hours to reach

1500°C, holding temperature of 1500°C for another 2 hours then the specimens were cooled to less than 100°C slowly in 1 hour. After sintering, the furnace was opened and the discs were left to cool down to the room temperature.

2.2. Composite resin discs preparation

Composite resin discs (n=64) were fabricated utilizing a mold of teflon that was designed and fabricated based on needed disc specifications (4 mm internal diameter × 3 mm thickness).

2.3. Zirconia Surface treatment

The discs were categorized based on the type of surface treatment technique and chemical conditioning **into 8 groups:**

- **Group 1: as milled specimens with no primer application. (N=8)**
- **Group 2: as milled specimens with primer application. (N=8)**
- **Group 3: sandblasted specimens with no primer application. (N=8)**
- **Group 4: sandblasted specimens with primer application. (N=8)**
- **Group 5: etched by ABF specimens with no primer application. (N=8)**
- **Group 6: etched by ABF specimens with primer application. (N=8)**
- **Group 7: etched by etchant cloud system specimens with no primer application. (N=8)**
  - **Group 8: etched by etchant cloud system specimens with primer application. (N=8)**

2.3.1 Sandblasting of Zirconia specimens

The bonding surfaces of the zirconia discs were sandblasted using 110 μm Al2O3 particles (Sahara, Dentify GmbH, Scheffelstraße, Engen, Germany) under a pressure of 2 bars for 10 seconds with 10 mm distance perpendicular to discs bonding surfaces[9] using Renfert Basic sandblaster (Renfert, Germany). Primer was then applied to 8 specimens.

2.3.2 Etching by Ammonium hydrogen difluoride (NH_4HF_2)(Qualikems fine chempvt, Ltd.Nandesari, Vadodara, Gujarat.)

NH_4HF_2 material is supplied as powder with the ability to etch zirconia surfaces confirmed by previous studies[20,21]. It can be used as powder only or used to form aqueous slurries to etch zirconia surfaces [20,21]. Aqueous slurries give the ability of controlled application compared to powder.

Zirconia discs were etched by NH_4HF_2 as follow:

- Small amount of theNH_4HF_2powder was used to form viscous slurries by mixing with distilled water then the formed slurries were spread using spatula with amount enough to cover the whole bonding surface of zirconia discs.
- Heating of the discs was carried on in a furnace. The discs were etched by NH4HF2 for 10 minutes at 170ºC.
- After etching, the discs were rinsed in water. Primer was then applied to 8 specimens.

2.3.3 Etching by zirconia etching system group (Zirconia etchant cloud system, Mdeifive, Korea.)

Zirconia discs were etched according to manufacturer instructions as follow:

- Hydrofluoric acid gel was spread on the bonding surfaces of zirconia discs.
- The discs were placed in the safe shell which was filled with water to the marked line. The pack then was submerged in the water in the bottom of the shell which was assembled immediately and locked to snap the marking. Neutralization gel was then applied to the top of the shell to close the injection port. The reaction is carried out for 10 minutes.
- After decomposing the shell, Zirconia discs were collected and rinsed in water. Primer was then applied to 8 specimens.

2.4. **Bonding of specimens:**

Zirconia discs bonding to the composite resin discs was performed utilizing adhesive resin cement (Multilink®N,) as follow:

Each zirconia disc took its place in a mold to be fixed in the base of a custom designed device that was made to be able to deliver a 5 KG loadto the zirconia/composite discs assembly during the process of cementation.

Multilink®N resin cement then was applied on the bonding surface of the zirconia disc after being auto-mixed by the disposable auto-mix tip.

One of the composite discs was placed onto the zirconia disc after cement application to form composite/zirconia assembly. The device was used to apply 5 kg constant load on the assembly. Light curing of the cement was done then the load was removed from the device after 5 minutes and the same steps were applied to all the specimens.

2.5.**Artificial aging**

After cementation, the bonded specimens were left for an hour in room temperature and then stored in water at 37ºC for 5 months in water jars. Following water storage, specimens underwent thermal-cycling for 5000 cycles, which is equivalent to 6 months of clinical use, using thermal-cycling device (Julabo®FT200, Germany) then dried before testing.

2.6. Tensile bond Strength (TBS) measurement

A universal testing machine (Model 3345, Instron Industrial Products, Norwood, USA) was utilized to measure tensile bond strength (TBS). The test was conducted at a crosshead speed of 2 mm/min, using a chain loop alignment system to ensure axial loading free of moments. The force required for de-bonding was recorded in Newtons (N), then divided by the surface area and expressed in mega pascals (MPa): $\tau = P/ \pi r^2$ where ; τ =TBS (MPa, P =load at failure(N) π =3.14 and r =radius of resin disc(mm).

3.Results:

Bond strength results

The mean bond strength and standard deviation of each test group were calculated and presented in MPa in Table1.

Group 1: as milled specimens with no primer application. (N=8) - **Group 2:** as milled specimens with primer application. (N=8) - **Group 3:** sandblasted specimens with no primer application. (N=8) - **Group 4:** sandblasted specimens with primer application. (N=8) - **Group 5:** etched by ABF specimens with no primer application. (N=8). - **Group 6:** etched by ABF specimens with primer application. (N=8). - **Group 7:** etched by etchant cloud system specimens with no primer application. (N=8). - **Group 8:** etched by etchant cloud system specimens with primer application. (N=8)

I-Statistical analysis

Post hoc LSD test was performed to detect significance difference between group means.

Group 1 (control) group exhibited the lowest bond strength (3.4 ± 0.7 MPa) among all groups.

Group 2, group 4, group 6, and group 7 showed significantly higher bond strength compared to group 1. (P=0.023)(P=0.005)(P=0.020)(P=0.026) respectively. Group 4 showed significant difference from group 3 (P=0.016)

No significant differences were found between group 1 and Group 3, Group 5or group 8.

Neither Group 5 nor Group 7 demonstrated significant difference with Group 3 or with each other. (P>0.05)

No significant differences were observed among Group 2, group 4, group 6 or group 8 in the pair wise comparisons (P>0.05)

Group 4, group 6, and group 7 showed significantly higher bond strength compared to group 1 but did not differ significantly from group 2.

II-Scanning electron microscope examination

SEM images showed mixed mode of failure in Figure_1of as milled specimens showing Smooth surface with minimal irregularities and adhesive mode of failure in Figure_2 of a specimen treated with etchant cloud system showing micro-retentions with clearly defined irregularities, suggesting effective acid interaction.

III-Failure pattern analysis

Failure patterns in Table 2 of the de-bonded specimens showed 27 adhesive failure patterns and 33 mixed failure patterns with no cohesive failure patterns.

Table 1: Mean±SD of each testing groups.

Groups	**Group 1**	**Group 2**	**Group 3**	**Group 4**	**Group 5**	**Group 6**	**Group 7**	**Group 8**
Mean ± SD	**3.4 ± .7**	**6.4 ± 1.4**	**4.2 ± 2.6**	**7 ± 2.8**	**5.8 ±3.4**	**6.4 ± 1.9**	**6.3 ± 1.6**	**6 ±2.9**

Table 2: shows failure patterns among groups.

		Group							
		Group-1	Group-2	Group-3	Group-4	Group-5	Group-6	Group-7	Group-8
Failure Pattern	Adhesive	3	2	3	3	2	5	5	4
	Mixed	3	6	5	5	5	3	3	3

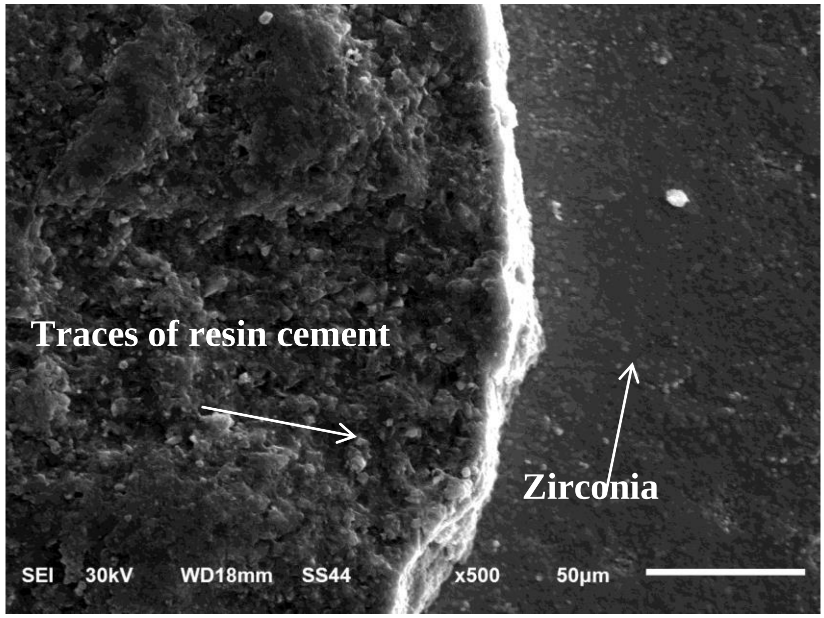

Figure_1: showing mixed mode of failure at x500 on as milled specimen.

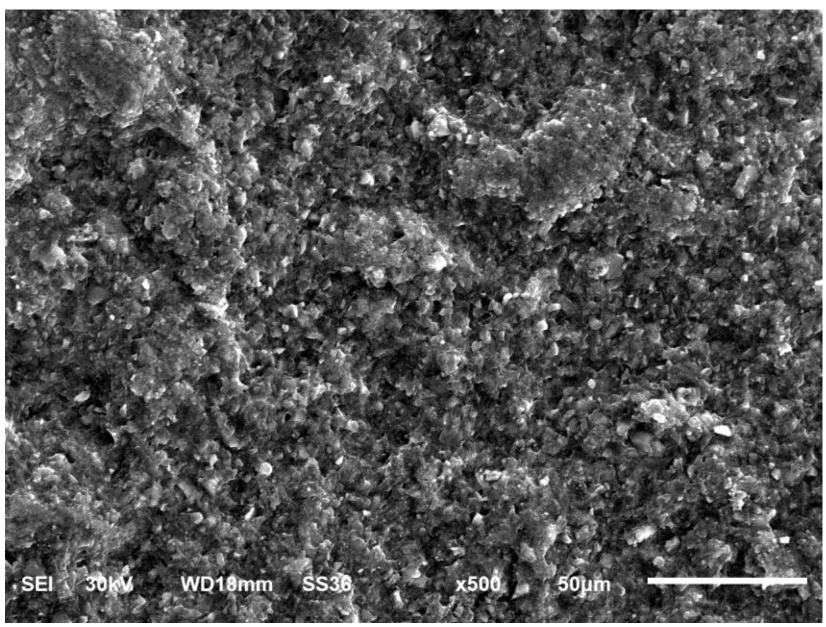

Figure_2: showing adhesive mode of failure at x500 on as etched by etchant cloud system specimen.

4. Discussion:

Restorations serving in the oral cavity are subjected to thermal and mechanical stresses which can compromise the strength of the bond between zirconia restoration and resin cement. [22] Artificial aging methods are able to simulate the intra oral conditions. Thermal cycling and long term water storage are reliable techniques to determine the obtained bond durability. These techniques were recommended by many studies. [23–25]

Gale and Darvell (1999) [26] proposed that 10,000 thermal cycles could mimic one year of oral cavity usage. In this study, specimens underwent two artificial aging processes prior to bond strength testing: five months of water storage in a 37°C water bath, followed by 5,000 thermal cycles. Each thermal cycle included immersion for 1 minute in a 5°C cold bath and 1 minute in a 55°C hot bath, with a 30-second dwell time between transitions.

Zirconia sandblasting was proved to produce surface roughness and resulted in increased surface area for bonding and increased surface energy which facilitates the flow of resin cement into micro-retentions. It could also produce hydroxyl groups on the surfaces of zirconia, which would make it more reactive with phosphate monomers.[15] Kim et al (2021) stated that zirconia surfaces that were sandblasted with 110 μm sand obtained a surface topography with greater change making the surface optimal for a favorable bonding with less surface damage.[9] Sandblasting of zirconia surface with Al_2O_3 was conducted in this study and was confirmed to produce surface roughness by SEM with no significant difference from the control group. Group 3 (4.2 MPa) did not exhibit a significant difference from the control group 1 (3.4 MPa) in TBS results, despite the surface roughness achieved.

Hydrofluoric acid ability to etch zirconia surface was investigated by many previous studies. [27–30] In this study, 9% HF gel was used in hot etching step of zirconia specimens in the form of Etchant cloud system. In agreement with **Kim et al. (2022)**[28] Hydrofluoric acid etching showed improved bond strength results between zirconia and composite resin discs. It produced zirconia surface micro-irregularities that were investigated and confirmed by SEM. This surface topography improved TBS results between zirconia and composite resin discs with significant difference between group 7 (6.3 MPa) and group 1 (3.4 MPa) (P = 0.026).

Zirconia etching with Ammonium Bi-fluoride was proved to enhance its bonding strength to resin cements in previous studies. [20,21] **Ruyter et al. (2017)[**20] used NH4HF2 on zirconia surface as powder or an aqueous slurry with no statistically significant difference in the bond strength values. **Akazawa et al. (2019)[**21] used NH4HF2 as powder with no water addition. They heated the zirconia specimens after NH4HF2 application at 170ºC for 10 minutes. A previous study examined the impact of NH4HF2 application on zirconia surfaces for measuring bond strength of resin cement to zirconia ceramic under varying heating temperatures and durations. Three protocols were employed: 170 °C for 20 minutes, heating at 170 °C for 10 minutes, and heating at 190 °C for 10 minutes. The findings indicated that no statistically significant differences among the protocols. [31]

In the current study, aqueous slurries of ammonium bi-fluoride had been applied on the surface of zirconia specimens which was further heated for 10 minutes at 170ºC. This process produced higher tensile bond strength between the zirconia discs and the composite resin in group 5 (5.8 MPa) in comparison to group 1 (3.4 MPa) but with no statistically significant difference. However, our results showed no significant difference between the different surface treatment techniques.

Chemical bonding between zirconia ceramic and resin cement in addition to micromechanical retention is proved to achieve bond that is strong and durable. [16] Many studies stated that MDP results in wettability improvement and chemical bonding enhancement so the use of MDP either within priming agents or resin cements resulted in enhanced bond strength between resin cement and zirconia ceramics. [17,19] Monobond®N was used as a priming agent to conduct this study.

In agreement with other studies.[16,17, 19,32–35] Chemical conditioning showed significant difference in the tensile bond strength measurements. Groups 4, 6 showed significantly higher bond strength compared to group 1 but did not differ significantly from group 2, where only chemical conditioning was applied with no micromechanical surface treatment. This demonstrates the enhancing effect of primer application on bond strength, regardless of surface treatment. However, group 8 did not differ significantly from group 1 in spite of primer application.

Primer application resulted in higher bond strength values in the control, sandblasting and etching with ammonium bi-fluoride groups. Contrary to our expectations, the sub-group etched by hydrofluoric acid with no primer applied showed higher TBS measurements than that one where primer was applied. A previous study [36] also found that using MDP-containing resin cement showed lower bond strength after acid etching. They proposed that zirconia's surface hydroxyl group may have been removed by chemical etching which would have decreased the TBS of the resin cement containing MDP.

5.Limitations of the study:

The study was conducted under controlled laboratory conditions (in vitro) with a limited duration of investigation. The 5-month water storage period and 5,000 thermal cycles represented only a small portion of the anticipated lifespan of a prosthetic restoration. Moreover, the study was limited to the use of a single primer. To substantiate the findings, clinical studies are essential. Additionally, further in-vitro and clinical research is recommended to explore the effects of chemical conditioning, following hydrofluoric acid etching of zirconia surfaces, using different priming agents on the bond strength to zirconia ceramics.

6.Conclusions :

The following conclusions were found within the limitations of this in-vitro study;

1. Chemical conditioning has significant effect on the bond strength of resin cement to zirconia ceramic
2. The Etchant cloud system emerged as a promising protocol for clinical application.
3. Further investigations would be recommended to explore the compatibility between chemical conditioning and etched surfaces by Etchant cloud system and their effect on the bond strength to zirconia ceramics

4. CONFLICT OF INTEREST:

The authors declare no conflicts of interest.

5. FUNDING:

This study received no financial support from public, commercial, or non-profit organizations.

6. ETHICS:

All experimental protocols were reviewed and approved by the Ethics Committee of the Faculty of Dentistry, Mansoura University, under reference number (A17030123).

7. Availability of data and materials

The data sets used and/or analyzed during this study are available from the corresponding author upon reasonable request.

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