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INFLUENCE OF CHEMICAL ETCHING ON BOND STRENGTH OF TRANSLUCENT ZIRCONIA TO RESIN CEMENT

Walid Al-Zordk* *and* Lamia Dawood*

ABSTRACT

Purpose: To assess the effect of chemical pre-treatments (piranha, hot acid, and heteropolyacid) on the bond strength (µTBS) to zirconia.

Materials and Methods: A total of 8 sintered zirconia blocks were randomly assigned into 4 groups; Group I: Air abrasion, Group II: Air abrasion and etching using piranha solution ($3H_2SO_4$: $1H_2O_2$), Group III: Air abrasion and etching using hot acid solution ($1HNO_3$: 1HF), and Group IV: Air abrasion and etching using heteropolyacid solution (2gm/50 mL). Zirconia blocks were bonded to their corresponding pre-constructed composite blocks using adhesive resin cement. For each group, each assembly was sectioned to obtain 20 microbars. All microbars were thermocyled (10,000), then the µTBS test was done with the aid of a Universal Testing machine. To determine the mode of failure, each specimen was examined under stereomicroscope (x40). One-way ANOVA and Post Hoc Tukey tests were used.

Results: The piranha pre-treatment displayed the lowest bond strength (16.82 ±5.97 MPa), while heteropolyacid pre-treatment had higher bond strength (26.22 ±4.05 MPa). One-way ANOVA test indicated statistical significant difference between the studied groups ($P<.001$). No significant difference was observed between control and piranha groups, also between hot acid and heteropolyacid groups. However, there were statistical significant differences between the control and heteropolyacid groups, and between piranha and heteropolyacid groups.

Conclusions: According to the current findings; The novel surface pre-treatment with heteropolyacid (2gm/50 mL) performed well to improve bonding to air-abraded zirconia-based ceramics. The hot acids etching pre-treatment enhanced the bonding to zirconia.

KEYWORDS: Surface treatment, Phosphotungistic acid, Heteropolyacids, Hot acid, Piranha

INTRODUCTION

In fixed prosthodontics, zirconia is a dental biomaterial with a number of merits, including biocompatibility, superior mechanical properties, and fair optical characteristics.[1,2] However, its high surface stability poses a variety of issues in terms of chemical or mechanical bonding performance and longevity with various cementation protocols.[3-5]

* Associate Professor, Fixed Prosthodontics Dept., Faculty of Dentistry, Mansoura University, Mansoura, Egypt

Since zirconia micro-structure lacks a glassy matrix, traditional mechanical and chemical techniques that work with glass-ceramics do not really work with zirconia.[6-9]

Micro-mechanical retention and chemical bonding are needed for a stable bond between cement and zirconia.[3,10,11] It was found that different surface treatments and new adhesive systems, are helpful in enhancing the bonding to zirconia ceramics.[12,13] Resin cement with MDP (10-methaacryloyloxyidecl-dihyidrogen-phosphate) monomer is recommended to get the best bond to zirconia ceramics.[10,14,15]

Surface pre-treatments are needed for long-term bonding between the indirect restoration and the tooth structure, moreover, the cementation technique influences the clinical success of a ceramic restoration.[16,17] To strengthen the bonding of zirconia restorations, various surface pre-treatment approaches have been suggested.[18,19] Airborne particle abrasion with aluminum oxide particles is a convenient and widely used process for increasing wettability, surface roughness, and surface area for micro-mechanical interlocking.[20,21] Airborne particle abrasion, on the other hand, causes structural defects and the formation of sharp cracks, which exacerbates radial cracking during function.[22] As a result, other effective zirconia conditioning methods, such as piranha solution and hot acid etching, can be used.[5,23-27] After airborne particles abrasion, pre-treatment of zirconia with a hot acid mixture (69 percent nitric acid and 48 percent hydrofluoric acid) improved the bond between zirconia and the resin cement, and the specimens showed broader distribution roughness, resulting in micromechanical preservation and better bonding strength.[28]

Heteropolyacids have stronger acidity than hydrofluoric and sulfuric acids according to Hamett acidity classification.[29] Heteropoly acids contain tungsten, molybdenum or vanadium, they can be used as treatment solution directly on zirconium, nickel, cerium and titanium alloys.[30] The addition of tungsto-phosphoric acid to zirconia created strong acidic Lewis sites and is the strongest super acid among all known heteropolyacids.[29,31,32]

Micro-tensile tests have a superior featured strength than macro-tensile tests and this advantage makes micro-tensile test more popular and most used.[28,33-35] The aim of this study was to assess the impact of acid pre-treatments (piranha solution, hot acid solution, and heteropolyacid solution) on the microtensile bond strength of monolithic zirconia restorations. The null hypothesis was that chemical pre-treatment with strong acids would improve the bonding of zirconia restoration.

MATERIALS AND METHODS

A partly sintered zirconia disk (Katana STML, Kuraray Dental Inc., Japan) was cut into 8 blocks with dimensions of 10 mm length, 10 mm width, and 6 mm thickness using a precision cutting machine (Isomet 4000, Buehler Ltd., USA). Zirconia blocks were sintered using a zirconia sintering furnace (Tabeo-1/M/Zircon-100, MIHM-VOGT, Germany), based on the instructions of the manufacturer. The sintering temperature was 1450°C for 2 hours holding time. After sintering, the dimensions were measured using a digital caliper (ISZ-1108-300, Insize, Japan) and were approximately 8 mm length, 8 mm width, and 4.8 mm thickness. Then, the bonding surface of each zirconia block was polished with polishing kit (Diacera zirconia polisher burs, EVE, Germany).[36] All zirconia blocks were ultrasonically cleaned in an ultra-sonic device (Baku 3550, China) for 10 minutes in distilled water, and air dried. A total of 8 composite blocks were fabricated from a light-cure composite resin material (Tetric N-ceram, Ivoclar Vivadent, Liechtenstien) using a slit Teflon mold. The curing was performed using a curing unit (Gulin Woodpecker Medical Co., China). The bonding surface of each composite block was polished using silicon abrasive points (Shofu, Japan).

The dimensions of each composite block are 8 mm in length, 8 mm in width, and 4 mm in thickness. The composite blocks were cleaned in an ultrasonically in water for 10 minutes before being air dried.

A total of 8 sintered zirconia blocks were randomly divided according to surface pre-treatments into 4 equal groups (n=2); Group I: Air abrasion, Group II: Air abrasion and etching using piranha solution ($3H_2SO_4$: $1H_2O_2$), Group III: Air abrasion and etching using hot acid solution ($1HNO_3$: 1HF), and Group IV: Air abrasion and etching using heteropolyacid solution.

Surface pre-treatment: For all groups, the bonding surface of each zirconia block was air-borne article abraded with 50 µm alumina particles (Al_2O_3 Cobra, Renfert GmbH, Germany) at 0.2 MPa for 10 sec/ mm^2 at 10 mm distance perpendicular to the zirconia surface.[24,37] For Group I (control group), no further treatment was used. For Group II, the bonding surface of each zirconia block was immersed in the piranha acid solution for 4 days.[23] The piranha solution was prepared from a mixture of 96% sulfuric acid (Al Nasr Pharmaceutical Co., ADWIC, Egypt) 30% hydrogen peroxide (Piochem Co., Egypt). The piranha solution was replaced each day by a fresh one. For group III, the bonding surfaces of the zirconia blocks were immersed in the hot acid solution (heated up to 100 °C for 25 minutes). The hot acid solution is a mixture of 69% nitric acid (Honeywell International Inc., Germany) and 48% hydrofluoric acid (Honeywell International Inc., Germany).[26] Regarding Group IV, the bonding surfaces of the zirconia blocks were immersed in the heteropolyacid solution for one minute. The heteropolyacids solution (2 gm /50 mL) consists of 2 gm of phosphotungistic acid hydrate (Sigma-Aldrich, USA) dissolved completely in 50 mL of distilled water. The zirconia blocks were cleaned ultrasonically in water for 10 minutes after surface pre-treatments and air dried.

Cementation: To avoid any potential surface contamination, the bonding procedures were begun immediately. On the bonding surface of the zirconia block, the resin cement (Panavia SA Cement plus automix, Kurary Noritake Dental Inc., Japan) was dispensed. The zirconia block was then seated over its corresponding composite block for 5 minutes while under a static load of 1 kg. According to the manufacturer's recommendations, the cement was light cured for 10 seconds from each surface (Gulin Woodpecker Medical Co., China). The assemblies were stored in distilled water for 24 hours after cementation.

Specimens' preparation and thermal aging: Each zirconia / resin/ composite assembly was fixed to the precision cutting machine and to obtain 1 mm micro-bars, the assembly was sectioned perpendicular to the bonding interface with a diamond blade (Buehler, USA). A digital caliper (Mitutoya, Japan) was used to verify the cross-sectional region of the bonding interface for each micro-bar. Additionally, each micro-bar was inspected at x50 magnification under a stereomicroscope (MA 100 Nikon, Japan) to pick only the intact specimens. Then, with a dwell time of 30 seconds, specimens were subjected to 10,000 thermal cycles (SD Mechatronics Thermocycler, Westerham, Germany) in a temperature range of 5 to 55°C.[38] Until bond strength testing, all micro-bars were stored in distilled water for 24 hours.

Microtensile bond strength test: prior to testing, the bonding interface area of each micro-bar was checked again using the digital caliper. The micro-bar was fixed using a cyanoacrylate glue (Amir A2000, China) in an attachment which allowed precise application of tensile force to the micro-bar. Then, the attachment was mounted into the universal testing machine (Instron Universal testing machine, 3345, USA). The tensile force was applied at crosshead speed of 0.5 mm/min until de-bonding. The machine software (Bluehill Lite, Instron, USA) was used to determine the bond strength.

Failure mode: For each specimen, the fragments were carefully separated and examined under x40 magnification with a stereomicroscope to assess the mode of failure as adhesive failure at the resin cement-zirconia interface, cohesive failure inside ceramic or composite, or mixed failure.[39]

Statistical analysis: IBM SPSS package version 20.0 (Armonk, NY. USA) was used to analyze the results. The Kolmogrov-Smironov Test was used to ensure that the data was normal. For comparisons, the one-way ANOVA test and the Post Hoc Tukey test were used.

RESULTS

Table 1 presents the means and standard deviations of the microtensile bond strength values for each group. The piranha pre-treatment group displayed the lowest bond strength (16.82 ±5.97 MPa), indicating low bonding between zirconia surface and resin cement when compared with other groups, while heteropolyacid pre-treatment group had higher bond strength (26.22 ±4.05 MPa). The differences in the microtensile bond strength values among groups were tested using the one-way ANOVA test, which indicated a statistical significant difference (F= 7.42214, *P*.001). There was no significant difference between the control and piranha groups (*P*=.999), and no significant difference between the hot acid and heteropolyacid groups (*P*=.113) as revealed by the post-hoc Tukey test. However, there was a statistical significant difference (*P*=002) between the control and heteropolyacid groups, as well as a statistical significant difference (*P*=.001) between the piranha and heteropolyacid groups. The mode of failure of all tested groups are presented in Figures 1 and 2.

TABLE (1) Microtensile bond strength (MPa) of the study groups.

Group	Mean ±Standard deviation
Air-borne particle abrasion	17.13 ±6.01[a]
Piranha solution	16.82 ±5.97[b]
Hot acid	22.22 ±4.51
Heteropolyacid	26.22 ±4.05[a,b]

Similar superscripted letters denote statistical significant difference between groups with Post Hoc Tukey test.

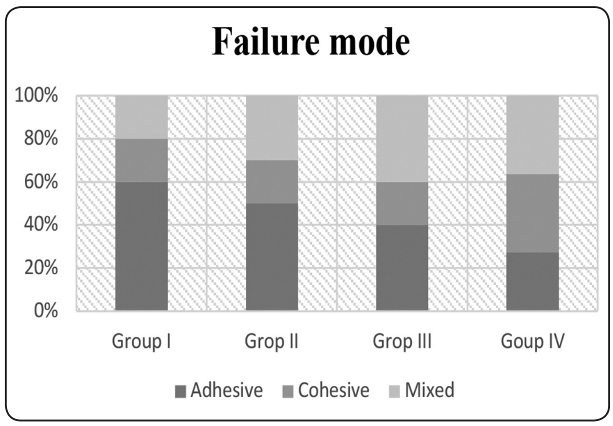

Fig. (1) Failure modes among the study groups.

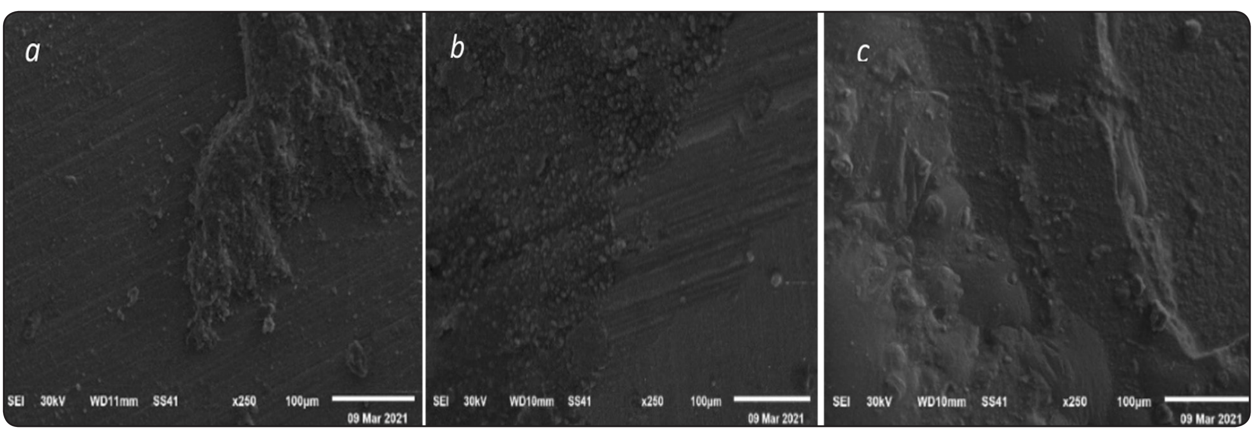

Fig. (2) SEM micrograph images; a: adhesive mode of failure, b: cohesive mode of failure, and c: mixed mode of failure.

DISCUSSION

Since strong acids (heteropolyacids) have an effect on the bond strength of zirconia ceramics, the null hypothesis of the current study that chemical pre-treatment with strong acids would strengthen the bonding of zirconia restorations was accepted.

The MDP-containing resin cement was used in the current study since it has been reported that MDP-containing resin cements have the highest bond strength with abraded zirconia due to the chemical interaction between the functional phosphoric acid monomer and the hydroxyl groups on the zirconia surface.[5,40] Clinically, cemented dental restorations are subjected to a variety of conditions, and simulated ageing may be used to simulate intra-oral envirments.[41] Thermal ageing was used in this study to mimic oral conditions that could affect the cemented restorations.

Zirconia specimens were air abraded with 50 µm aluminum oxide in this study. The air-borne particle abrasion is one of the most commonly employed pre-treatments which can be easily done and resulting in improved surface roughness, micro-mechanical undercuts and enhancing adhesion to zirconia.[5,37] It was found that air abrasion increased restoration retention nevertheless the type of the cement used.[42] Air abrasion with 50 µm had reduced deleterious effect on the zirconia surface when compared to that with 120 µm at the same pressure.[43] In term of duration and power of the procedure, the effect of air abrasion on zirconia flexural strength is controversial.[8] Using of air-borne particle abrasion at low pressure seems to reduce the damage of the abraded surface of the zirconia.[44] The use of resin cement was thought to heal small surface flows caused by air-borne particle abrasion and strengthen the ceramic.[11]

In the current study, piranha etching solution represented the lowest bond strength value (16.82 ±5.97 MPa). Hallmann et al[16] found that the air-borne particle abrasion had a higher bond strength than piranha solution, and the low bond strength with piranha is attributed to the formation of compounds on the surface of zirconia and unstable bond between resin cement and the hydroxyl groups formed by piranha solution. Additionally, piranha etching solution clean and hydroxylates the surface of zirconia without formation of undercuts which are important for the micro-mechanical interlocking. On the other hand, Lohbaur et al[23] reported higher bond strength after etching of abraded zirconia with piranha solution, which could be attributed to using larger alumina size (110 µm).

The hot acids etching solution used in this study was formulated based on the work of Liu et al[26], who reported that etching zirconia surface with a 1:1 mixture of HF acid (48%) and HNO_3 (69%) at 100 °C for 25 minutes improved zirconia grain dissolution rate, surface roughness, and bonding surface area to cement, with no phase transition. The results of the present study showed higher, but not significant, micro-tensile bond strength after hot-acid + air-borne particle abrasion pre-treatment (22.22 ±4.51 MPa) compared with air-borne particle abrasion (17.13 ±6.01 MPa). These results were supported by several studies.[24,26-28] Liu et al[26] reported that zirconia treated with hot acids etching had better adhesion to resin cements than zirconia treated with other surface treatments. Hot acid etching increases surface roughness by dissolving the zirconia surface's less well-arranged peripheral atoms, resulting in wider grain borders and better micro-mechanical adhesion with the resin cement.[26]

This *in vitro* study was the first to assess the effect of heteropolyacids (phosphotungistic acid) with dental zirconia as surface pre-treatment method. In the current study, the chosen concentrations were based on a SEM pilot study indicated that etching of zirconia surface with concentrations more than 1gm50 / mL for 1 min 2wt% are effective up to 4 wt%, while 15 wt% of heteropolyacids provides the highest recorded acidity. The acidity decreases when the heteropolyacids loading exceeds 15 wt%.[32] The results of the current study showed a statistical significant difference between tested chemical pre-treatments and heteropolyacids chemical pre-treatment, which recorded the highest value of microtensile bond strength (26.22 ±4.05 MPa). These results were supported by the study of Gaucci et al[36] who found that specimens etched with acids had higher bond strength than air-borne particle abrasion and untreated specimens. Also, Lee et al[15] found that strong acid etching improved the bond strength, and increased concentration of acid was directly proportional to bond strength. Sakrana et al[28] found that the specimens treated with strong acid etching recorded the highest bond strength over those treated with air-borne particle abrasion or Silano Pen. The results of Lee et al[17] revealed that the strong acid etching increased the bond strength. Previous studies reported that the minimum clinically acceptable range for bond strength was 10-13 MPa.[34,9] As a result, the findings of this study demonstrated that etching using the heteropolyacids can guarantee resin bonding to zirconia ceramics.

The cohesive mode of failure indicated greater bond strength in the current study.[35] The primary mode of failure in Groups I and II was adhesive failure, which can be due to a weak bond between zirconia and cement. Additionally, the adhesive failure could be related to the reality that the microtensile bond strength test estimates a small interfacial bonding zone.[35]

The phase transition and mechanical properties of zirconia specimens after application of surface pre-treatments using strong acids are not evaluated in this *in vitro* study. A number of resin cements must also be tested. In addition, clinical trials would be needed to validate these *in vitro* findings.

CONCLUSIONS

Based on the results of this *in vitro* study:

1. The novel surface pre-treatment using heteropolyacid (2gm/50 mL) improved bonding to zirconia-based ceramics that had been air-abraded.
2. Hot acids etching pre-treatment improved the bonding of resin cement to air-abraded zirconia.

Clinical Relevance: Considering the bond strength results after aging, the novel surface pre-treatment with heteropolyacid perform well to improve bonding to zirconia. For clinical indication, further aspect needs to be studied and evidence should be based on clinical studies.

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