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EFFECT OF SURFACE PRE-TREATMENTS ON BONDING PERFORMANCE OF ZIRCONIA CERAMIC WITH RESIN CEMENTS

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ABSTRACT

Purpose: To establish an effective bonding for zirconia, the aim of this study was to evaluate the influence of air-borne particle abrasion, Piranha acid etching and hot acid etching pre-treatments on bond strength of zirconia with self-adhesive resin cements (Panavia SA, TheraCem) and conventional adhesive resin cement (Panavia F2.0). Also, the effect of Silano-Pen treatment on the bond strength of zirconia to resin cements was evaluated.

Materials and Methods: Eighteen zirconia blocks were cut, sintered and divided into three groups (n=6): air-borne particle abrasion, Piranha acid etching ($3H_2SO_4$:$1H_2O_2$), and hot acid etching ($1HNO_3$:1HF). From each group, the bonding surfaces of three zirconia blocks were treated with Silano-Pen. Each zirconia block was bonded to its corresponding composite block utilizing either Panavia SA, TheraCem or Panavia F2.0. Each ceramic/ resin / composite assembly was sectioned perpendicular to the bonding interface to obtain microbars of 1 mm^2 thickness. A total of 180 microbars were subjected to 10000 thermal cycles between 5°C and 55°C with dowel time of 30 seconds. Each microbar was subjected to tensile force until de-bonding. The data was statistically analyzed.

Results: The hot acid showed the highest μTBS (21.96±5.86 MPa) followed by air-borne particle abrasion (16.40±6.23 MPa) and the lowest was Piranha (15.04±7.12 MPa). With Panavia SA, there was significant difference (p=.035) between μTBS with Silano-Pen in air-borne particle abrasion and Piranha groups, also there was significant difference (p=.004) between Piranha and hot acid groups. With TheraCem, there was significant difference (p=.008) between μTBS with Silano-Pen in air-borne particle abrasion and Piranha groups, also there was significant difference (p=.003) between air-borne particle abrasion and hot acid groups. The interaction between cement and Silano-Pen was insignificant (p=.067).

Conclusions: Pre-treatment method and type of adhesive resin cement influences the effectiveness of bonding of zirconia. The hot acid etching recorded the highest bond strength, whereas the lowest was recorded with Piranha etching. Silano-Pen treatment after hot acid etching improved the bonding of zirconia to adhesive resin cement. The self-adhesive MDP-containing resin cement (Panavia SA) enhanced the effectiveness of the bond strength with zirconia.

KEY WORDS: Zirconia, bonding, self-adhesive, hot acid, Piranha, sandblasting.

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INTRODUCTION

One of the limitations for the clinical durability of zirconia is its limited ability to bond with resin cement because zirconia is a glass-free polycrystalline microstructure, non-etchable and chemically inert with low surface energy.[1-3] Various surface treatment methods were employed, to enhance bonding with zirconia, such as air-borne particle abrasion, hot chemical etching, and pyrochemical silica coating. Air-borne particle abrasion increases the surface wettability, surface roughness and provides micromechanical undercuts.[4] However, air-borne particle abrasion results in structural defects and induction of sharp cracks that enhance radial cracking during function.[5] Piranha etching solution is a combination of hydrogen peroxide and sulfuric acid which a strong oxidizing corrosive agent used to remove the organic impurities and hydroxylate surfaces.[1,6] Hot chemical etching improves the surface roughness through a corrosion controlled process which is based on removing the less well arranged and high energy peripheral atoms resulting in wider grain boundaries.[7,8] The using of hydrofluoric acid in different temperature and concentrations could produce modifications in zirconia surface.[9] Moradabadi et al[10] studied the effect of micromechanical and chemical surface treatments on bond strength of zirconia to resin cement and concluded that the air-borne particle abrasion treatment prior to acid etching process enhanced the shear bond strength. Pyrochemical silica coating depends on formation of a silica layer through the chemical reactions of silane at high temperature.[4,11]

Contemporary resin cements were classified into self-adhesive resin cements and conventional resin cements.[12] Self-adhesive resin cement contains acid functionalized monomers and conventional methacrylate monomers. Based on the functional acidic monomers, there are two popular groups; methacrylate monomers with carboxylic acid groups such as 4-META based cements and PMGDM based cements, or with phosphoric acid groups such as MDP based cements and BMP based cements.[13] Oyagüe et al[14] evaluated the hydrolytic stability of different resin cements when bonded to zirconia and concluded that water aging played an important role in the degradation of the bond strength and the bond durability depended mainly on the cement selection rather than the applied surface treatment.

To study the adhesion of the resin cement to zirconia, there are various testing methods such as macro-shear, microshear, macrotensile and microtensile tests. It is critical that the bonding interface must be the most stressed zone, regardless of the applied test methodology. With micro-tensile test, the small interfacial bonding zone (1 mm^2) and small dimension shows more homogeneous stress distribution; therefore, it presents more sensitive evaluation of bond performance when specimens are aligned correctly.[15,16]

The main limitation of zirconia based restoration is its low adhesive potential and the conventional adhesive techniques do not produce a high enough bond strength.[17,18] To establish a reproducible, effective and applicable bonding protocol for zirconia restorations, the objective of the present study was to evaluate the influence of air-borne particle abrasion, Piranha acid etching and hot acid etching pre-treatments on bond strength of zirconia with self-adhesive resin cements (Panavia SA Cement Plus and TheraCem) and conventional adhesive resin cement (Panavia F2.0). Also, the effect of Silano-Pen treatment on the bond strength of zirconia to self-adhesive resin cements and conventional resin cements. Thus, the null hypotheses of this study were there no differences in the effect of surface pre-treatment methods on bond strength of zirconia with adhesive resin cements.

MATERIALS AND METHODS

Partially sintered zirconia blocks (inCoris TZI C, Sirona Dental, Germany) were cut using precision cutting machine (Isomet 4000, Buehler Ltd, Lake Bluff, IL, USA) to obtain eighteen zirconia blocks measuring 10 mm length, 10 mm width and 6 mm thickness. All zirconia blocks were sintered in zirconia sintering furnace (Sirona inFire HTC, Sirona Dental Systems, GmbH, Germany) according to manufacturer's instructions. After sintering, the dimension of each zirconia block (8 mm length, 8 mm width and 4.8 mm thickness) was measured using digital caliper (Mitutoyo, Tokyo, Japan) to verify the volumetric shrinkage after sintering. After finishing using a zirconia-specific finishing kit (Eve Ernst Vetter GmbH, Germany), all zirconia blocks were ultrasonically cleaned in distilled water for 10 minutes, then air dried.[19] A light-cure composite (Tetric N-Ceram, Ivoclar Vivadent) was used to fabricate eighteen composite blocks using Teflon mold (8 mm length, 8 mm width and 4.8 mm thickness). After curing, the bonding surface of each composite block was polished, ultrasonically cleaned and air dried. Eighteen zirconia blocks were equally divided according to the surface treatment into three main groups (n=6): air-borne particle abrasion group, Piranha group (air-borne particle abrasion and Piranha acid etching) and hot acid group (air-borne particle abrasion and hot acid etching). Each group was divided into two subgroups (n=3) either directly bonded with no further treatment or treated with Silano-Pen before bonding of zirconia with resin cements. Panavia SA Cement Plus Cement Plus self-adhesive resin cement (Kuraray Noritake Dental, Tokyo, Japan), TheraCem self-adhesive resin cement (BISCO Inc, USA) and Panavia F2.0 adhesive resin cement (Kuraray Noritake Dental, Tokyo, Japan) were the resin cements used for bonding.

For air-borne particle abrasion group, the bonding surfaces of zirconia blocks were particle abraded with 50 μm Al_2O_3 at a distance of 10 mm and perpendicular to the surface.[20] For Piranha group, the bonding surfaces of zirconia blocks were particle abraded as in air-borne particle abrasion group. Then, the zirconia blocks were immersed in a glass beaker containing Piranha acid solution ($3H_2SO_4$: $1H_2O_2$) for 4 days.[1] The acid solution was daily replaced by fresh solution. The Piranha solution was prepared from a mixture of 96% sulfuric acid (Al Nasr Pharmaceutical Chemicals Co., Egypt) and 30% hydrogen peroxide (Piochem Co., Egypt). As regard hot acid group, the bonding surfaces were air abraded and immersed in polyethylene beaker containing the hot chemical etching solution ($1HNO_3$:1HF) which was heated up to 100 °C in water bath for 25 minutes.[21] The hot chemical etching solution was prepared as a mixture of 69% nitric acid (Honeywell International Inc., Burdick and Jackson, Seelze, Germany) and 48% hydrofluoric acid (Honeywell International Inc., Riedel-de Haën, Seelze, Germany). Finally, all zirconia blocks were rinsed with distilled water, ultrasonically cleaned in distilled water for 10 minutes and air dried. For each group, a representative zirconia block was pre-treated for SEM evaluation of the surface pre-treatments. From each group, the bonding surface of three zirconia block was subjected to Silano-Pen treatment. The bonding surfaces of nine zirconia blocks were heated with the pale blue reactive flame zone of Silano-Pen device (Bredent GmbH, Senden, Germany) according to the manufacturer's instructions. After the surface was cooled down to room temperature, the silane liquid (Silane Haftvermittler, Bredent, Senden, Germany) was applied using a disposable brush and left for 30 seconds.

Each zirconia block was bonded to its corresponding composite block utilizing either Panavia SA Cement Plus, TheraCem or Panavia F2.0 resin cements according to manufacturer's recommendations. The bonding procedures were performed under a static load of 1 kg to ensure a uniform cement layer.[22] The light curing was performed from all directions for each cement according to the recommendations of the manufacturers. The ceramic /resin /composite assemblies were stored in distilled water at 37°C for 24 hours.[23] Using a cutting machine (Isomet 4000), each assembly was sectioned perpendicular to the bonding interface area to obtain microbars of 1 mm^2 thickness. For each microbar, the cross-sectional area of the bond interface was verified using a digital caliper (Mitutoyo, Tokyo, Japan). Microbars were examined under stereomicroscope (MA 100 Nikon, Japan) at 50x magnification for selection of the intact specimens that are free from any microcracks.

A total of 180 microbars were assigned for testing of the bond strength as presented in figure 1. All specimens were subjected to 10000 thermal cycles (SD Mechatronics Thermocycler, Westerham, Germany) between 5 °C and 55 °C with a dowel time of 30 seconds.[24] Each microbar was subjected to tensile force through a universal testing machine (3345, Instron, 2519-104, 3345, Canton, MA, USA) until de-bonding. The mean value of the bond strength for each specimen was calculated in Mega Pascale (MPa) using the machine software (Bluehill Lite software, Instron, MA, USA) through dividing the load at failure (N) by the adhesive area (mm^2). Statistical analysis was done using statistical software (SPSS Statistics for Windows version 22).

RESULTS

Means and standard deviations of microtensile bond strength values are presented in Table 1. Normal and relative (marginal) distributions of data was tested by using Shapiro-Wilk's test and Levene's test that revealed normal data distribution. Regardless the bonding procedure within each pre-treatment, one-way ANOVA showed significant difference (F=6.810, p=.000) between air-borne particle abrasion, Piranha acid etching and hot acid etching surface pre-treatments. Post Hoc multiple comparisons revealed significant difference between the main surface pre-treatments with the highest bond strength in hot acid group and the lowest bond strength in Piranha group (Table 2). As regard to resin cement, there was significant difference between Panavia SA and TheraCem, and between Panavia SA and Panavia F2.0 (Table 3).

TABLE (1) Mean and standard deviation of μTBS values of the test groups.

			N	Mean	Stand deviation
Air-borne particle abrasion	Direct bonding	Panavia SA	10	21.516	7.33
		TheraCem	10	15.350	1.84
		Panavia F2.0	10	20.649	2.52
	Silano-Pen	Panavia SA	10	20.185	4.17
		TheraCem	10	10.640	4.08
		Panavia F2.0	10	10.068	4.25
	Total		60	16.401	6.23
Piranha	Direct bonding	Panavia SA	10	23.077	5.11
		TheraCem	10	11.406	3.77
		Panavia F2.0	10	5.699	0.80
	Silano-Pen	Panavia SA	10	12.982	4.31
		TheraCem	10	19.731	7.11
		Panavia F2.0	10	17.352	3.43
	Total		60	15.041	7.12
Hot acid	Direct bonding	Panavia SA	10	25.120	4.55
		TheraCem	10	17.494	8.78
		Panavia F2.0	10	22.181	6.91
	Silano-Pen	Panavia SA	10	23.001	3.04
		TheraCem	10	20.805	5.54
		Panavia F2.0	10	23.199	4.59
	Total		60	21.967	5.86

With Panavia SA, there was significant difference (p=.035) between bond strength with Silano-Pen in air-borne particle abrasion and Piranha, also there was significant difference (p=.004) between Piranha and hot acid. However, there was no significant difference (p=.408) between bond strength in air-borne particle abrasion and hot acid with Silano-Pen application (table 4). Regarding TheraCem, there was significant difference (p=.008) between bond strength with Silano-Pen in air-borne particle abrasion and Piranha, also there was significant difference (p=.003) between air-borne particle abrasion and hot acid. However, there was no significant difference (p=.752) between bond strength in Piranha and hot acid with Silano-Pen application. With Panavia F2.0, there was significant difference with direct bonding and with Silano-Pen (p=.000 and .034 respectively) between bond strength in air-borne particle abrasion and Piranha.

For interaction between tested variables, the surface pre-treatment and cement significantly affect the bond strength values. Also, the interaction between the surface pre-treatment and cement, and surface pre-treatment and Silano-Pen was significant (Table 5). SEM evaluation of each surface pre-treatments are presented in Figures 1-4.

TABLE (2) Post Hoc multiple comparisons using LSD of the three main surface pre-treatments.

Main surface treatments		Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
(I)	(J)				Lower Bound	Upper Bound
Air-borne particle abrasion	Piranha	2.7542*	.97954	.006	.8181	4.6903
	Hot acid	-4.3410*	.97954	.000	-6.2771	-2.4048
Piranha	Air-borne particle abrasion	-2.7542*	.97954	.006	-4.6903	-.8181
	Hot acid	-7.0952*	.97954	.000	-9.0313	-5.1590
Hot acid	Air-borne particle abrasion	4.3410*	.97954	.000	2.4048	6.2771
	Piranha	7.0952*	.97954	.000	5.1590	9.0313

The mean difference is significant at the 0.05 level

TABLE (3) Post Hoc multiple comparisons using LSD of Panavia SA Cement Plus, TheraCem and Panavia F2.0 adhesive resin cements.

Resin Cement		Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
(I)	(J)				Lower Bound	Upper Bound
Panavia SA	TheraCem	5.5791*	.97954	.000	3.6430	7.5152
	Panavia F2.0	4.1732*	.97954	.000	2.2371	6.1093
TheraCem	Panavia SA	-5.5791*	.97954	.000	-7.5152	-3.6430
	Panavia F2.0	-1.4059	.97954	.153	-3.6430	.5302
Panavia F2.0	Panavia SA	-4.1732*	.97954	.000	-6.10.93	-2.2371
	TheraCem	1.4059	.97954	.153	-.5302	3.3420

The mean difference is significant at the 0.05 level

TABLE (4) Comparison of μTBS values with Panavia SA Cement Plus, TheraCem and Panavia F2.0 among groups.

		Panavia SA		TheraCem		Panavia F2.0	
		Mean difference	Sig	Mean difference	Sig	Mean difference	Sig
Direct Bonding							
Air-borne particle abrasion	Piranha	-1.56095	.646	3.94380	.247	14.95023*	.000
	Hot acid	-3.60332	.290	-2.14442	.528	-1.53219	.652
Piranha	Hot acid	-2.04237	.548	-6.08822	.075	-16.48242*	.000
Silano-Pen							
Air-borne particle abrasion	Piranha	7.20294*	.035	-9.09162*	.008	-7.28364	.034
	Hot acid	-2.81651	.408	-10.16520*	.003	-13.13099*	.000
Piranha	Hot acid	-10.01946*	.004	-1.07358	.752	-5.84736	.087

*The mean difference is significant at the 0.05 level.

TABLE (5) The interaction between the study variables.

Source of variation	Type II Sum of Squares	Df	Mean Square	F value	p value
Main surface treatment (A)	1535.418	2	767.709	26.671	.000
Silano-Pen treatment (B)	1.819	1	1.819	.063	.802
Cement (C)	1010.371	2	505.186	17.550	.000
(A) * (B)	256.909	2	128.454	4.463	.013
(A) * (C)	788.371	4	197.093	6.847	.000
(B) * (C)	158.229	2	79.115	2.748	.067
(A)* (B)* (C)	697.073	4	174.268	6.054	.000
Error	4145.015	144	28.785		
Total	89412.664	180			

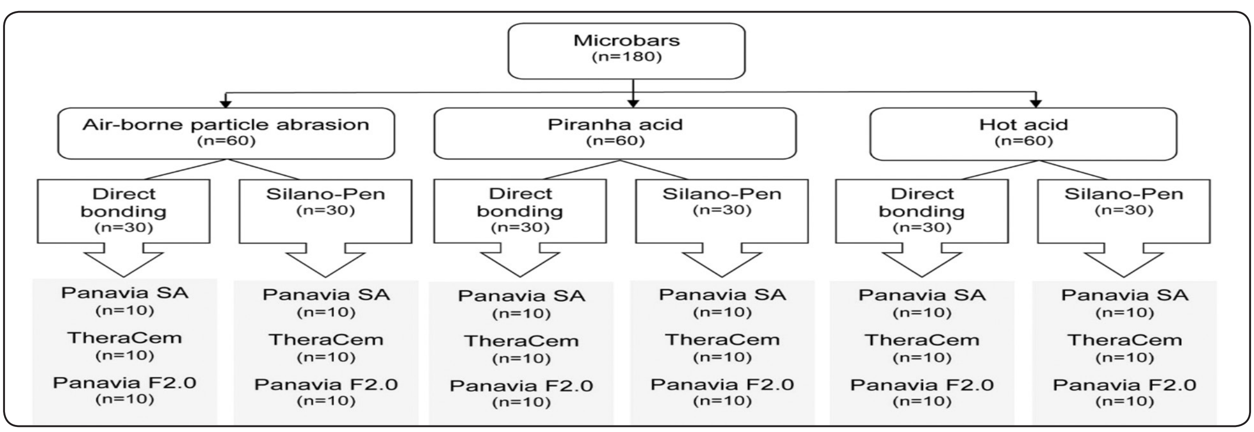

Fig. (1) Follow chart detailing the study setup.

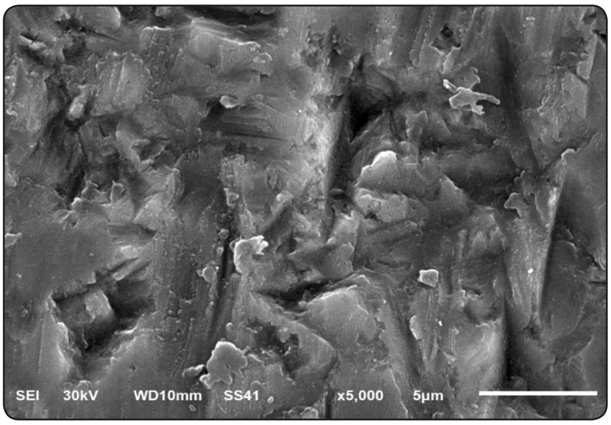

Fig. (2) SEM micrograph (5000x) showing the air abraded surface of zirconia.

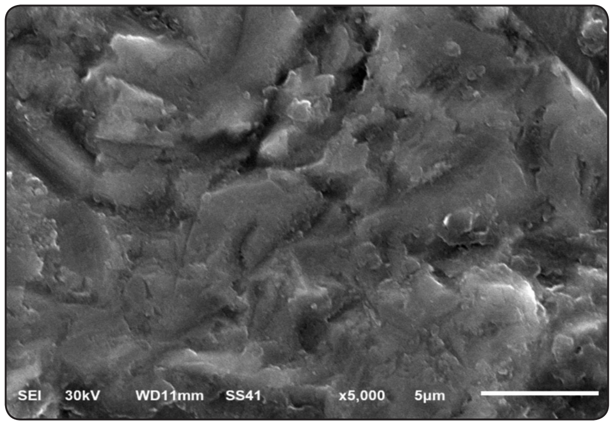

Fig. (3) SEM micrograph (5000x) showing the zirconia surface after etching with Piranha acid solution.

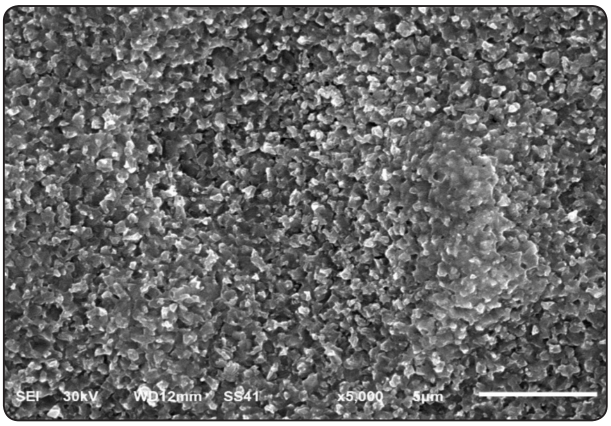

Fig. (4) SEM micrograph (5000x) showing the zirconia surface after etching with hot acid solution.

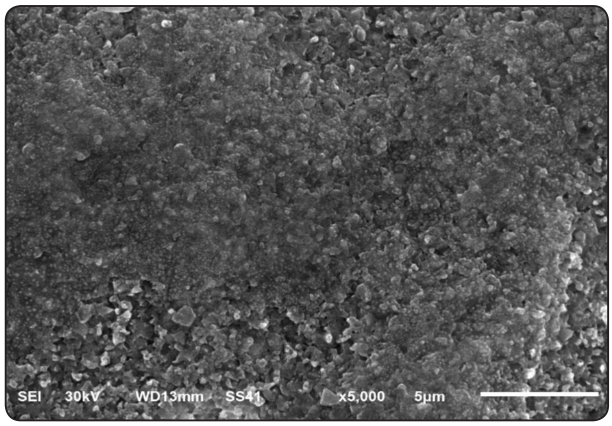

Fig. (5) SEM micrograph (5000x) showing the zirconia surface after etching with hot acid solution and Silano-Pen treatment.

DISCUSSION

There are several methods for achieving a high bond strength with zirconia, but this bond should be able to withstand the surrounding oral environment over years.[25] Recently, adhesive strategies that combine mechanical and chemical pre-treatment have been developed to improve the bond durability between resin cement and zirconia.[26] Air-borne particle abrasion with aluminum oxide particles with a particle size of 50 μm is a common and simple procedure to produce micro-mechanical roughness.[3,27] Also, the usage of 50 μm aluminum oxide particles has a less harmful effect of the surface topography of zirconia when compared with 120 μm aluminum oxide particles.[28] In the present study, Piranha acid etching solution was prepared according to Lohbauer et al[1] who observed that 96% sulfuric acid and 30% hydrogen peroxide with ratio of 3:1 for four days exhibited effective chemical preconditioning with effective hydroxylation of abrade zirconia surface. Also, hot acid etching solution was prepared according to Liu et al[21] who found that 69% nitric acid and 48% hydrofluoric acid with ratio of 1:1 at 100°C for 25 minutes resulted in improving the dissolution rate of zirconia grains with increased roughness. In the present study, air-borne particle abrasion was applied before hot acid etching to get the advantages of both methods.

The hot acid etching pre-treatment showed the highest micro-tensile bond strength values (21.96±5.86 MPa) followed by air-borne particle abrasion (16.40±6.23 MPa). The Piranha acid etching resulted in the lowest micro-tensile bond strength value (15.04±7.12 MPa). The rank of micro-tensile bond strength from lowest was Piranha "air-borne particle abrasion" hot acid. Therefore, the first part of null hypothesis was rejected. The results of the present study were coincided with that of Casucci et al[22] who found that zirconia treated with hot acid solution recorded higher bond strength than those treated with air-borne particle abrasion. Another study showed higher bond strength with hot acid etching than air-borne particle abrasion.[29] The hot acid etching improves the surface roughness and remove the superficial ceramic layer resulting in a homogenous granular and porous texture of zirconia.[8] Studies showed that the bond strength and durability after Piranha acid etching was lower than that after air-borne particle abrasion.[6,30] The inferior bond strength and durability is related to the unstable bond between the resin cement and the hydroxyl groups produced by Piranha solution.[6] Additionally, surface conditioning with Piranha solution clean and hydroxylates the surface without formation of undercuts which are particularly important in micromechanical interlocking with the resin cement.[30]

In contrary, Moradabadi et al[10] showed that the zirconia treated with air-borne particle abrasion recorded higher bond strength than that treated with air-borne particle abrasion and HF/HNO_3 etching at room temperature for two minutes. The addition of this etching solution to the abraded zirconia surface leading to deformation of surface roughness created by air-borne particle abrasion leading to deformation of this roughness to be rounded and which results in reduction of the micromechanical retention.[10] In the current study, the acid solution was performed for 25 minutes at 100°C. That temperature with hot acid etching has an essential role in molecular motion as the higher the temperature, the protons become more easily ionized leading to more acidic effect.[8] Another study reported that the addition of Piranha solution to the abraded zirconia had higher bond strength than air-borne particle abrasion using 110 μm particle size.[1] The possible explanation is that the aggressive air abrasion using bigger particle size could result in ditching between resin cement and zirconia surface.[31]

Concerning Silano-Pen, the results showed that the interaction between main surface pre-treatment and Silano-Pen was significant, while no significant interaction between Silano-Pen and cement. The heat treatment using Silano-Pen could improve the surface hydroxylation resulting in a more reactive zirconia surface.[31] Moreover, Silano-Pen enhances the surface wettability and produces a dense scattering nanosilica grains leading to a stratified surface topography.[32,33] Additionally, the role of air-borne particle abrasion with hot acid etching in creation and improving the surface roughness could not be neglected.[20]

In the present study, a comparison was held between MDP-containing conventional resin cement (Panavia F2.0) which is commonly used in researches and two self-adhesive resin cements which are methacrylate-based (TheraCem) and MDP-containing (Panavia SA) self-adhesive resin cements. The results of the present study showed that Panavia SA Cement Plus showed high bond strength. These results could be related to the high content of acidic phosphate functional monomers in self-adhesive resin cement which could increase hydrophilicity of the cement resulting in hydrolytic degradation due to the higher water sorption.[34] Tanis et al[35] showed that the using of MDP-containing resin cement improved the bond strength with air-borne particle abrasion. Another study reported that the abraded zirconia specimens bonded with methacrylate-based cement showed lower bond strength than bonded with MDP-containing resin cement as the MDP monomers creates a chemical interaction with zirconia.[36] Also, the results of the present study revealed that the highest bond strength with hot acid etching. The hot acid etching improves the surface roughness by dissolving the less well arranged peripheral atoms of zirconia surface resulting in larger grain boundaries formation which increase mechanical interlocking with resin cement with no phase transformation.[23]

With Panavia F2.0, there was significant decrease in the bond strength (5.69±0.80 MPa) after aging with Piranha group. This bond strength value is considered a very low value for acceptable clinical bonding as the range of 10-13 MPa was suggested as the minimum clinically acceptable bond strength.[37,38] Although the increased inorganic filler (59vol%) has a significant role in improving wear resistance, mechanical properties and reducing polymerization shrinkage, it affect the proper viscosity and the suitable film thickness.[39] Moreover, this cement needs hand mixing which possibly leading to incorporation of air bubbles and resulting in reduction of the mechanical properties.[40]

New zirconia ceramics for dental restorations are continually under development, only one type of zirconia was tested in the present study. The results obtained should be verified in future studies in comparison with more surface condition methods and with more prolonged aging. The specimens were produced and examined under ideal conditions which may not reflect actual clinical conditions. Further clinical studies are needed to confirm the relationship between surface pre-treatment, Silan-Pen and cement to confirm the durability of the bonding protocol for zirconia restorations.

CONCLUSIONS

Within the limitations of the current study, it was concluded that:

1. The surface pre-treatment method and type of adhesive resin cement influences the effectiveness of bonding with zirconia-based restoration.
2. The hot acid etching pre-treatment recorded the highest bond strength, whereas the lowest bond strength was recorded with Piranha acid etching pre-treatment.
3. Silano-Pen treatment after hot acid etching improved the bonding of zirconia to adhesive resin cement more than after air-borne particle abrasion.
4. Among the tested adhesive resin cements, the self-adhesive MDP-containing resin cement (Panavia SA Cement Plus) enhanced the effectiveness of the bond strength with zirconia restoration.

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