Original Article

Effect of Combined Application of Hydrofluoric and Phosphoric Acids and Active Irrigation with a Microbrush on Shear Bond Strength of Lithium Disilicate Ceramics to Enamel

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Abstract

Background: This study assessed the effect of combined application of hydrofluoric (HF) acid and phosphoric acid (PA) and active irrigation (AI) with a microbrush on shear bond strength (SBS) of lithium disilicate (LDS) ceramics to enamel.

Materials and Methods: This *in vitro* study was conducted on 40 extracted teeth that received enamel preparation with a #12 cylindrical bur. Forty IPS e.max LT rods (3mm diameter, 6mm height) were fabricated and randomly assigned to four groups (n = 10) for surface treatment with 5% HF (group 1), 5% HF and AI with a microbrush for 20 seconds (group 2), 5% HF and 32% PA (group 3), and 5% HF and 32% PA plus AI with a microbrush for 20 seconds (group 4). Silane and Choice 2 cement were used for bonding rods to enamel. The SBS was measured by a universal testing machine. Data were analyzed by two-way analysis of variance (ANOVA), Bonferroni, and Chi-square tests (alpha = 0.05).

Results: Group 4 had the highest SBS, and group 1 had the lowest SBS ($P < 0.05$). Group 2 had a significantly higher SBS than group 1, and group 4 had a significantly higher SBS than group 3. AI with a microbrush significantly increased the SBS ($P < 0.05$), but the application of PA caused no significant change in SBS ($P > 0.05$). The interaction effect of PA and AI on SBS was not significant ($P > 0.05$).

Conclusion: The application of PA in addition to 5% HF acid caused no significant change in the SBS of LDS ceramic to enamel. However, AI with a microbrush significantly increased the SBS.

Keywords: Dental enamel, hydrofluoric acid, lithium disilicate, phosphoric acids, shear strength

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Introduction

Ceramic laminate veneers are conservative tooth-colored restorations used for the correction of tooth shape and/or color and restoration of teeth with broken crowns.[1,2] Laminate veneers require less tooth preparation (0.5 mm thickness) compared with prosthetic crowns.[3] Several dental ceramics, including feldspathic porcelain, leucite-reinforced ceramics, and lithium disilicate (LDS) ceramics, are used for the fabrication of dental laminates.[4] IPS e.max LDS ceramics are increasingly used in dental practice due to high strength and optimal esthetics.[5,6] The low fracture rate of LDS is its main advantage over other materials such that it can tolerate fatigue caused by 1 million cycles of 1,000N load.[7]

Ceramic laminate veneers are bonded to tooth enamel using bonding agents and resin cement. To enhance the bond strength, 37.5% phosphoric acid (PA) is used for enamel etching to create micromechanical retention. Also, hydrofluoric (HF) acid is used for porcelain etching and enhancement of bond strength to ceramic laminate veneers.[8,9] However, mineralized

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salts known as "white residues" may still remain on the ceramic surface even after thorough irrigation of the etched surface and compromise the longevity and quality of bonding.[10]

Debonding is the most important cause of failure of ceramic laminate veneers.[10] Debonding more commonly occurs in the anterior teeth, compared with posterior teeth (60% versus 35%), and also in the maxilla than in the mandible.[11,12]

This study aimed to assess the effect of combined application of HF and PA and active irrigation (AI) with a microbrush on shear bond strength (SBS) of LDS ceramic to enamel. The first null hypothesis of the study was that the application of PA in addition to HF acid would not significantly increase the SBS of LDS ceramic to enamel. The second null hypothesis was that AI with a microbrush would not significantly increase the SBS of LDS ceramic to enamel.

Materials and Methods

This *in vitro*, experimental study was conducted on 40 sound-extracted premolar and central incisor human teeth with no restoration, caries, or cracks. The study protocol was approved by the ethics committee of Shahid Beheshti University of Medical Sciences (IR.SBMU.RIDS.REC.1394.127).

The sample size was calculated to be 10 in each group according to a study by Dündar *et al*.[13]

Tooth preparation

The teeth were cleaned with non-fluoridated pumice powder (Kimia, Tehran, Iran) and low-speed handpiece according to the method described by Kemper and Kilian[14] to remove dental plaque and calculus. They were then immersed in 0.2% thymol solution and kept refrigerated at 4°C for 2 days. Subsequently, they were rinsed and stored in saline for 6 months. Polyvinyl chloride (PVC) molds with 1 cm width and 2 cm depth (to match the piston of the universal testing machine) were then used to mount the teeth in auto-polymerizing acrylic resin (Acropars, Marlic, Tehran, Iran). The teeth were mounted in PVC cylinders containing acrylic resin perpendicular to the horizon to 2 mm below their cementoenamel junction. Next, the teeth received a preparation within the enamel by a #12 cylindrical bur under constant water irrigation until a smooth surface was achieved. They were then visually inspected to ensure that the preparation was within the enamel. A surveyor was used to ensure their correct orientation. The prepared surface was smoothed by a wheel-shaped bur (Diamond Burs, ISO 042/Eur 818 FG.045, Jota, Switzerland).

Fabrication of ceramic specimens

Forty cylindrical rods with 3 mm diameter and 6 mm height were fabricated from A1 shade LT IPS e.max Press LDS ceramic (Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions.[15] Briefly, wax-up was performed, and sprues were placed with a more acute angle than normal in a 200 g cylinder and invested. The cylinder was heated at 900°C for 1 hour and then underwent wax burn-out. Ingot and plunger were used at 700°C, and then, the temperature increased to 925°C and maintained for 20 minutes. Finally, pressing was performed under vacuum at 300 Psi pressure for 3–7 minutes. The fabricated specimens were then randomly assigned to four groups (n = 10) as follows.

Group 1 (control): The rods were subjected to 5% HF acid (IPS Ceramic Etching Gel, Ivoclar, Schaan, Liechtenstein) application for 20 seconds. They were then rinsed with water for 20 seconds such that no acid residue remained on the porcelain surface. The rods were then dried for 20 seconds. Two layers of silane (porcelain primer; Bis-Silane; Bisco, USA) were applied for 30 seconds as instructed by the manufacturer and dried with gentle air spray for 10 seconds. Porcelain bonding resin (Bisco, USA) was then used without curing, and Choice 2 cement was applied to the cross-sectional area of the rods. After removing the excess cement, curing was performed separately from the buccal and lingual surfaces for 40 seconds.

Group 2: The rods were subjected to 5% HF acid for 20 seconds, AI was performed with a microbrush for 20 seconds, and the specimens were dried for 20 seconds. The rest of the procedure was the same as that in group 1.

Group 3: The rods were subjected to 5% HF acid for 20 seconds, rinsed with water for 20 seconds, and dried for 20 seconds. Next, 32% PA (Uni-Etch; Bisco, USA) was applied for 40 seconds, rinsed to eliminate all acid residues, and dried completely. The rest of the procedure was the same as that in group 1.

Group 4: The rods were subjected to 5% HF acid for 20 seconds, rinsed with water for 20 seconds, and dried for 20 seconds. Next, 32% PA was applied for 40 seconds, and AI was performed with a microbrush for 20 seconds, followed by complete drying. The rest of the procedure was the same as that in group 1.

Cementation of ceramic specimens to enamel

The teeth were etched with 32% PA for 15 seconds, sufficiently irrigated, and dried such that the tooth surface remained slightly moist.[13] For bonding, one drop of bottle A and one drop of bottle B of All-Bond TE (Bisco, USA) were mixed with an applicator on a mixing pad and applied to the tooth surface in two coats. They were air-thinned for 10 to 15 seconds to obtain a homogenous surface and cured for 10 seconds.

Choice 2 resin cement (Bisco, USA) in the translucent shade was then used for bonding rods to enamel. It was applied to the rods with an applicator, and then, initial curing was performed for 3 seconds. Excess cement was removed, and final curing was performed from the buccal and lingual surfaces for 40 seconds using a curing unit with 600 mW/cm^2 light intensity.[16] The light curing unit was calibrated by a radiometer. For complete polymerization, the specimens were immersed in distilled water and incubated at 37°C for 24 hours.[17]

Thermocycling

To better simulate the clinical oral environment, all specimens were immersed in distilled water at 37°C for 7 days and underwent 2000 thermal cycles between 5 and 55°C with an exposure time of 20 seconds and a transfer time of 5–10 seconds according to ISO-11405 2003.[1,18]

SBS test

The SBS was measured in a universal testing machine (Zwick Roell, Germany) at a crosshead speed of 1 mm/minute with a 2.5 KN load cell.[19] A chisel was used to apply load parallel to the ceramic–resin cement bonding interface [Figure 1]. The load at debonding was recorded. The SBS was calculated in megapascals (MPa) by dividing the debonding force by the cross-sectional area of specimens (7.065 mm^2).

Stereomicroscopic assessment

After debonding, the tooth surface was inspected under a stereomicroscope (SZX9, Olympus, Japan) at ×60 magnification using Microsoft Windows NT (5.1 Service Pack 3 Pentium Processor). The mode of failure was classified as follows [Figure 2].

Adhesive: All resin cement was debonded from the rod surface.

Cohesive: A thin layer of resin cement remained on the ceramic surface, and the rest remained on the tooth surface.[20,21]

Mixed: A combination of adhesive and cohesive failures, such that part of the cement remained attached to the ceramic surface.

Scanning electron microscopic (SEM) assessment

Five random rods underwent SEM (30.00 KV; Vega, Tescan) assessment, one intact specimen with no surface treatment, and one from each group, to analyze the quality of the salt produced on their surface at ×1000, ×2500, ×5000, and ×10,000 magnifications.

Elemental analysis of the rods

The aforementioned five rods also underwent elemental analysis by energy-dispersive X-ray spectroscopy (EDS).

Statistical analysis

Data were analyzed by Statistical Package for the Social Sciences (SPSS) version 21 (SPSS Inc., IL, USA). Levene's test was used to analyze the homogeneity of variances, and the Shapiro–Wilk test was applied to assess the normality of data distribution. As the data had a normal distribution and the assumption of homogeneity of variances was met, the effects of PA application and AI with a microbrush on SBS were analyzed by the two-way analysis of variance (ANOVA). The Bonferroni test was subsequently applied for pairwise comparisons. The Chi-square test was used to compare the groups regarding the mode of failure. The level of significance was set at 0.05.

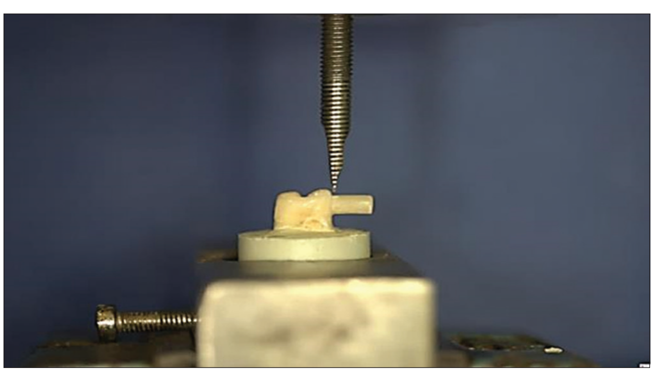

Figure 1: Load application in a universal testing machine

Results

SBS

Table 1 shows the measures of central dispersion for the SBS of the groups. The difference among the groups was significant in SBS, and the control group had the lowest SBS, and group 4 had the highest SBS ($P < 0.05$).

A two-way ANOVA showed that the effect of PA on SBS was not significant ($P = 0.234$). However, AI with a microbrush significantly increased the SBS ($P = 0.000$). The interaction effect of application of PA and AI with a microbrush on SBS was not significant ($P = 0.286$).

Pairwise comparisons of the groups regarding SBS showed no significant difference between groups 1 and 3 ($P = 0.928$); in other words, the application of PA, whether with or without AI with a microbrush, had no significant effect

Table 1: Measures of central dispersion for SBS (MPa) of the groups (n=10)

Group	Mean	Std. deviation	Minimum	Maximum
Control (5% HF)	898.18	818.7	97.7	36.34
5% HF + microbrush	601.26	947.4	34.18	99.31
5% HF + 32% PA	172.19	796.6	38.11	36.28
5% HF + 32% PA + microbrush	507.31	173.7	33.20	23.40

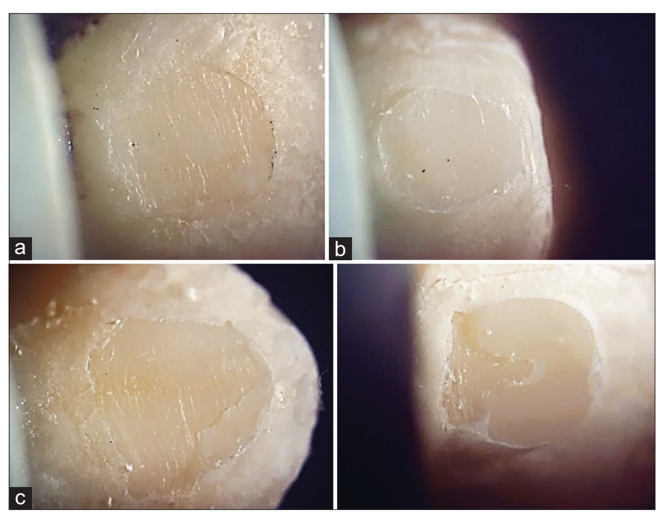

Figure 2: Modes of failure. (a) Adhesive; (b) cohesive; and (c) mixed

on SBS. The difference between groups 2 and 4 was not significant either ($P = 0.114$); in other words, in AI with a microbrush, the application of PA caused no significant change in SBS. However, groups 1 and 2 had a significant difference ($P = 0.015$) such that AI with a microbrush significantly increased the SBS. Also, groups 3 and 4 had a significant difference ($P = 0.000$) such that group 4 had a significantly higher SBS than group 3.

Failure mode

Table 2 presents the frequency of different modes of failure. As shown, cohesive failure was dominant in groups 2 (50%) and 4 (60%) (AI with a microbrush). The Chi-square test showed a significant difference in the frequency of different modes of failure among the groups ($P = 0.020$). A significant correlation existed between the type of surface treatment (group) and the frequency of different modes of failure (Pearson's Chi-square = 14.623); however, this association was not highly strong (Phi = 0.605 and Cramer's V = 0.428).

SEM assessment

Figure 3 presents the SEM micrographs of the cross-sectional areas of the rods in different groups.

EDS

In group 1 [Figure 4a], silica and oxygen had the highest percentage. Also, sodium and fluorine were noted, which can decrease the surface energy. In group 2 [Figure 4b], silica and oxygen had the highest percentage, and a reduction in the amount of fluorine and sodium was noted. In group 3 [Figure 4c], fluorine and silica had the highest percentage, and a considerable rise in sodium and potassium was noted. In group 4 [Figure 4d], silica and oxygen had the highest percentage. Zinc and aluminum were also seen with a low percentage. The percentage of miscellaneous elements, except for silica and oxygen, had significantly decreased.

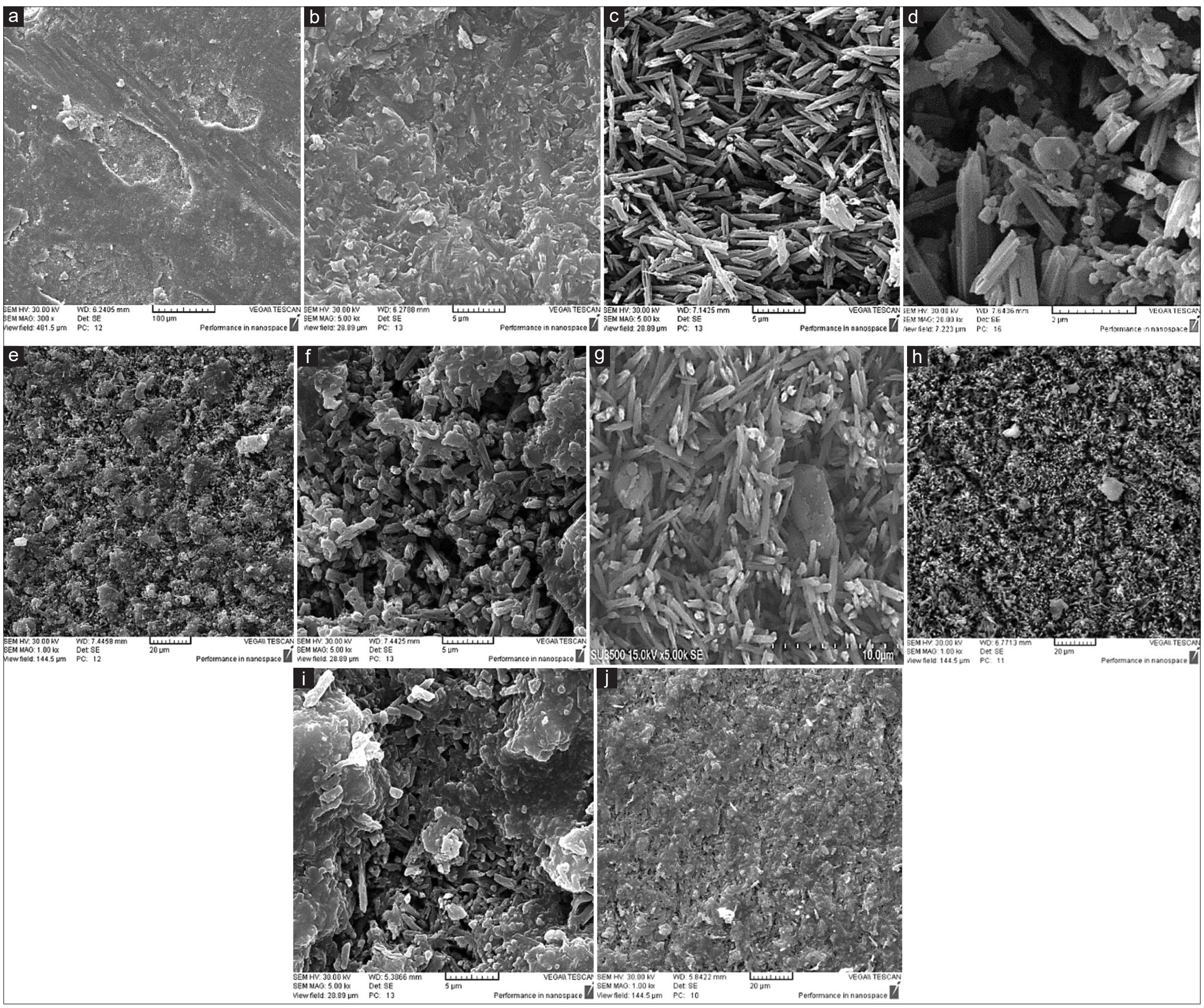

Figure 3: SEM micrographs of the cross-sectional areas of rods in different groups; (a and b) no surface treatment; (c and d) 5% HF acid for 20 seconds; (e and f) 5% HF acid + AI with a microbrush each for 20 seconds; (g and h) 5% HF acid for 20 seconds and 32% PA for 40 seconds; (i and j) 5% HF acid for 20 seconds and 32% PA for 40 seconds + AI with a microbrush for 20 seconds (×1000 and ×5000 magnifications)

Table 2: Frequency of different modes of failure in each group (n=10)

		Group				Total
		1	2	3	4	
Adhesive failure	Count	7	3	9	2	21
	%within group	70.0%	30.0%	90.0%	20.0%	52.5%
Cohesive failure	Count	1	5	1	6	13
	% within group	10.0%	50.0%	10.0%	60.0%	32.5%
Mixed failure	Count	2	2	0	2	6
	% within group	20.0%	20.0%	0.0%	20.0%	15.0%
Total	Count	10	10	10	10	40
	% within group	100.0%	100.0%	100.0%	100.0%	100.0%

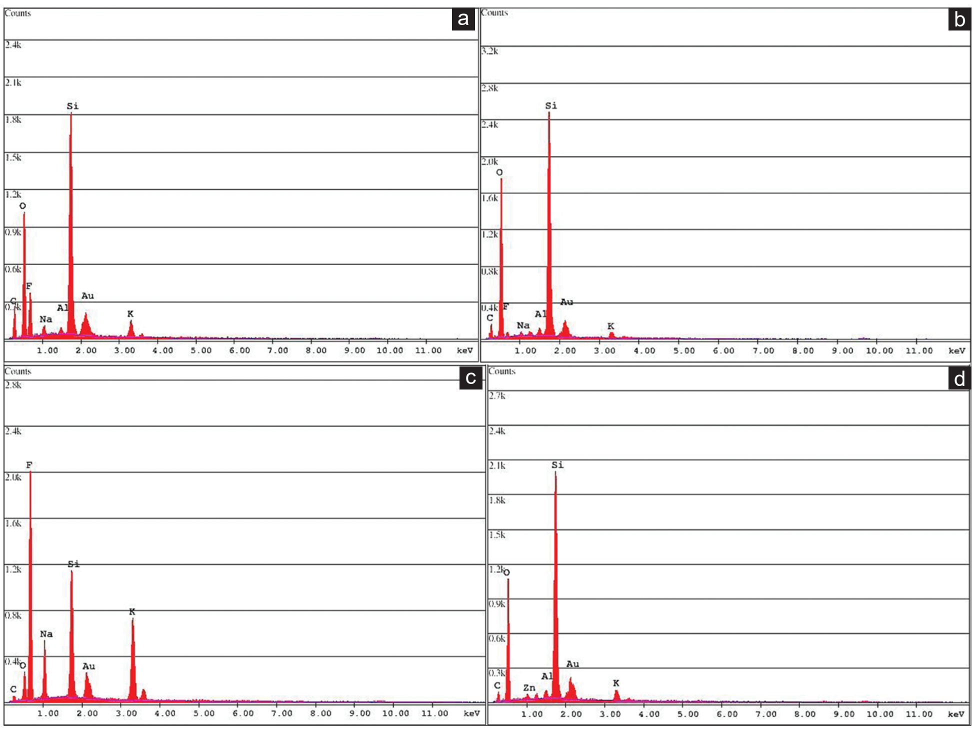

Figure 4: Elemental analysis of the surface of rods in the four groups by EDS: (a) group 1; (b) group 2; (c) group 3; and (d) group 4

Discussion

This study assessed the effect of combined application of HF and PA, and AI with a microbrush on SBS of LDS ceramics to enamel. The results showed that AI of LDS ceramic surface with a microbrush significantly increased the SBS to enamel. However, the application of PA had no significant effect on SBS. Thus, the first null hypothesis of the study was accepted, but the second one was rejected.

The literature is conflicting regarding the effect of etching on strength of dental ceramics. Several studies[22-24] showed that the etching of dental porcelain decreased its strength by 21% to 40%; however, they did not have a control group. Thus, their results were not highly reliable. SEM micrographs of the surface of rods in the present study revealed that the application of HF acid created numerous topographic irregularities on the surface of IPS e.max Press rods, and amorphous masses with a glass-like structure rich in silica and oxygen (as detected by EDS) were also seen on the surface. Similarly, Höland *et al*.[24] reported that the main crystalline phase of IPS Empress was composed of long LDS crystals, and the second phase was composed of lithium orthophosphate surrounded by a glass matrix. According to Höland *et al*.,[24] HF acid eliminates the second phase and glass matrix crystals and causes irregularities in the first-phase crystals. SEM micrographs in the present study revealed the same pattern. Also, the presence of a vitreous layer on the surface decreased the SBS.

According to Yen *et al*.,[25] the effect of etching can be explained by the chemical mechanisms that occur in the etching process. Feldspathic porcelain is composed of a glass matrix with an amorphous tetrahedral silicon network.[26] Insoluble feldspar and leucite crystals are present within the matrix, and their volume depends on the formulation used by the manufacturer.

HF acid reacts with the silica phase of feldspathic porcelain and produces hexafluorosilicate with a production rate of 0.44 µm/minute as stated by the manufacturer.[27] Consequently, a honeycomb (hexagonal) pattern is created on the porcelain surface, leading to micro-retention. A similar mechanism exists for PA. Although LDS ceramic was used in the present study, which has some differences in content and phases with feldspathic porcelain, it appears that the behavior of both porcelain types follows the same pattern when subjected to etching.

SEM micrographs in the present study revealed an etched surface pattern highly similar to that described in the literature. From group 1 to group 4, the surface became more irregular and the rate of porosities increased. Also, the surface in group 4 showed a higher frequency of holes and porosities. Moreover, it appears that PA acts as a reinforcing agent and enhances access to underlying debris; thus, microbrush could more easily remove deep debris, and therefore, not only does the penetration depth increase but also greater amounts of debris are removed. In a recent study, Filho *et al*.[28] showed similar surface roughness of IPS e.max Press after using PA and ultrasonic bath. They reported a reduction in surface roughness following immersion of specimens in an ultrasonic bath after PA application.[28] These observations confirmed the results of the SBS test in the present study both microscopically and theoretically (insignificant increase in use of PA and significant increase in use of microbrush). Elemental analysis of the surface of ceramics in the four groups in the present study also revealed a reduction in the number of different elements on the surface. A significant reduction in fluorine, sodium, and potassium was noted in groups 4 and 2, resulting in significant enhancement of SBS. Zinc was also found in group 4, which can effectively increase the bond strength.

The present results revealed significant enhancement of SBS by AI with a microbrush in groups 2 and 4; these groups also showed a higher frequency of cohesive failures, while adhesive failure was dominant in groups 1 and 3. In the present study, adhesive failure was dominant (52.5%) followed by cohesive (32.5%) and mixed (15%) failures. Giraldo *et al*.[29] evaluated the effect of active (with microbrush) and passive (without microbrush) application of PA on bond strength of LDS ceramics and showed a higher bond strength when both HF acid and PA were used with a microbrush, which was in line with the present findings. However, they performed a micro-shear test and did not perform thermocycling. The mode of failure was dominantly adhesive in their study, which was in agreement with the current findings. However, some other studies reported the dominance of cohesive failures in the substrate surface or composite resin.[30,31] The geometrical shape of specimens and unequal force distribution during the application of shear force can affect the results.[32] Usually, adhesive failures are associated with lower SBS, while cohesive failures are accompanied by higher SBS values.[33-35]

The present results indicated that AI with a microbrush significantly increased the SBS. Magne and Cascione[36] and Türkkahraman *et al*.[37] also reported the same results. Cleaning the surface with a microbrush after etching results in the removal of loose particles and creates a higher rate of porosities. Accordingly, the mechanical retention increases through micro-retention, resulting in the enhancement of bond strength. Filho *et al*.[28] compared the micro-tensile bond strength of IPS e.max Press ceramic after different surface treatments, including HF acid etching with and without PA etching and ultrasonic bath. They concluded that only active application of HF and PA maintained the bond strength high after thermocycling, and their combination minimized the reduction in bond strength. Pérez *et al*.[38] assessed the effect of different methods of removal of conditioning residues on the bond strength of LDS ceramics. They evaluated the efficacy of air–water spray, immersion in an ultrasonic bath with distilled water for 2 and 4 minutes, and application of PA for 2 and 4 minutes for this purpose. They reported that application of 37% PA for 4 minutes yielded the lowest bond strength, which may be because 37% PA can not only remove 9% HF acid, but also results in higher amounts of sodium, potassium, and calcium remnants on the surface that form a layer between the glass matrix of the ceramic and the resin cement, which decreases the bond strength.[38] Another study evaluated the effect of post-etch cleaning on surface microstructure, surface topography, and micro-SBS of LDS ceramic to resin cement. The specimens were etched with 9.6% HF acid with no post-etch cleaning in group 1, etched with 9.6% HF acid for 20 seconds followed by rinsing with water and post-etch cleaning with 37% PA in group 2, and etched with 9.6% HF acid followed by active application of 37% PA and post-etch cleaning in an ultrasonic bath for 5 minutes in group 3. The micro-SBS was significantly different among the three groups and was the highest in group 3 followed by group 2 and then group 1. The surface topography and surface microstructure were also significantly different among the three groups.[39]

Silane coupling agent was used in all groups in the present study. Hayakawa *et al*.[40] evaluated the effect of surface treatment and silane application on the SBS of resin to porcelain. They compared HF acid and PA and showed higher SBS in the use of HF acid. Studies reported that the application of silane significantly increased the SBS of groups etched with different concentrations of HF acid and highly recommended it to maximize the SBS and durability of restorations.[41,42]

This study had several strengths. A macro-shear test was performed, and thermocycling was also conducted to better simulate the clinical setting and increase the generalizability of the results to the oral environment.

However, the effect of air abrasion on SBS was not evaluated in the present study; also, only one type of resin cement was used for bonding rods, which were the main limitations of this study.

Future studies are required to compare the effects of HF acid etching plus AI with a microbrush with and without air abrasion on the SBS of LDS ceramics to enamel. Also, micro-shear test and different types of resin cement should be used in future studies.

Conclusion

The application of PA in addition to 5% HF acid caused no significant change in the SBS of LDS ceramic to enamel. However, AI with a microbrush significantly increased the SBS.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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