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EVALUATION OF SHEAR BOND STRENGTH OF MULTILAYERED ZIRCONIA AND LITHIUM DISILICATE CERAMIC TO RESIN CEMENT

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ABSTRACT

Objective: The purpose of this study was to evaluate the effect of two surface treatment protocols and aging on the shear bond strength (SBS) values of monolithic zirconia and lithium di-silicate glass ceramic.

Materials and methods: Eighty ceramic specimens were separated into groups to evaluate SBS using a universal testing machine. There were two main groups based on ceramic material: lithium disilicate glass ceramic (IPS e.max CAD) and monolithic zirconia (IPS e.max ZirCAD prime). Each main group was further subdivided into two surface treatment subgroups of 20 specimens each: (1) hydrofluoric acid etching with silane coupling agent (2) sandblasting. Additionally, each surface treatment subgroup was split into two classes: before and after thermocycling. All ceramic plates were bonded using a light-cured resin cement. SBS data was collected for statistical analysis.

Results: Thermocycling significantly decreased the SBS mean values concerning zirconia ceramic with sandblasting from (13.01 ± 3.89) to (3.12 ± 0.96), while with hydrofluoric acid mean values from ($9.8 \pm 3.0.$) to (2.36 ± 0.35). Zirconia ceramic revealed significantly lower SBS than lithium disilicate in all groups $P<0.05$.

Conclusion: Thermocycling decreased the SBS of both ceramics regardless of surface treatment type applied. Sandblasting surface treatment was better than hydrofluoric acid with silane when applied to zirconia ceramic. IPS ZirCAD prime ceramic still revealed weaker bond stability in comparison with IPS e.max CAD ceramic.

KEYWORDS: Ceramics, Hydrofluoric acid, Sandblasting, Shear bond strength, Thermocycling

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INTRODUCTION

In response to the increasing demand for aesthetic improvements in modern restorative dentistry, there has been an introduction of cutting-edge monolithic all-ceramic materials such as lithium disilicate glass-ceramic and zirconium dioxide, aimed at replacing the traditional metal-ceramic restorations[1]. They are widely used nowadays, for being biocompatible combined with an appealing natural-looking appearance to mimic the tooth structure's optical properties, additionally attaining exceptional mechanical properties [1, 2].

Monolithic zirconia ceramics have the most optimal mechanical properties of all dental ceramics, allowing for a combination of favorable esthetics and high strength [3]. Nonetheless, a significant issue noted with zirconia restorations is the challenge of maintaining retention and achieving a long-lasting bond with cement[4]. One important aspect influencing the clinical effectiveness of ceramic restorations is the bond's longevity[5]. Ceramic surface treatments, either mechanical (sandblasting)[6] or chemical (hydrofluoric acid (HF) etching)[7], aim to improve bond strength. However, the wet oral environment and prolonged chewing forces make creating a long-lasting bond very challenging[8, 9].

Increasing the surface free energy can help increase the adhesion between the two dissimilar materials[10,11]. Increased surface free energy improves the surface's wetting capacity for resin-cement bonding [12].

Ceramics made of lithium disilicate glass satisfy aesthetic and practical requirements. As ceramics made of silica demonstrate strong physical and chemical bonding potential with resin cements. This process is accomplished by first etching the surface with HF acid and then applying a silane coupling agent for conditioning [6].

HF acid etches lithium disilicate ceramics by reacting with the silica-containing glassy matrix, forming hexafluorosilicates. This process involves selectively removing the glassy phase, which creates a rougher ceramic surface by revealing the underlying crystalline structure. By roughening the surface, it enlarges the area that allows for the micromechanical bonding of resin cement [13].

On the other hand, zirconia lacks silicon dioxide in its microstructure, and is considered a dense polycrystalline structure, so, conditioning its surface using HF acid etching prior to resin cement application has become a real obstacle [14, 15]. Numerous research has addressed the contentious topic of how different surface treatment techniques, cement compositions, and ageing regimens affect zirconia's bond strength. Various researchers have established that zirconia ceramics can attain an adequate shear bond strength (SBS) through a sandblasting technique. This method involves air abrasion systems that emit a flow of air mixed with aluminum oxide (Al2O3) particles, which are used to abrade surfaces[16,17]. Particle diameter of 50 µm was found to give optimum results as concluded by some authors[18]. This mechanism relies on loosening the contaminated layers, creating surface irregularities and porosities, thus allowing the adhesive to interlock mechanically into these pores[16,19]. These surface irregularities and roughness provide a larger surface area for the bonding agent [16, 17].

Although sandblasting is used extensively as the mechanical method for increasing the zirconia's surface roughness, however, its effectiveness was influenced by several factors such as the abrasive particle size, the timing and the distance used for spraying the substrate[18]. Specifically, when huge grains are being used for closely-spraying the zirconia, the latter mechanical strength and qualities degrade, also microscopic fissures[21] occur as a result of the intense thermal shock that causes the zirconia phase to change from tetragonal phase to monoclinic phase. Such a transition significantly affects the zirconia restorations' long-term mechanical stability and permanent fracture resistance [18, 21]. However, some evidences showed that resin cement infiltrates these cracks, thereby reinforcing the ceramic. Thus, sandblasting has been proven to increase bond strength by roughening the surface and enhancing surface area for cement bonding[22]. Nonetheless, the surface micro-cracks caused by sandblasting will reduce the long-term dependability of zirconia[23].

Other research indicates that chemical surface treatments such HF acid etching boosted surface energy and, as a result, surface wettability. The link between resin cement and zirconia is then strengthened by silanization that followed [24]. Other authors stated that silanization will not make a difference in the bond durability [25]. Acid etching has the benefit of consistently roughening the material's surface without imposing any force on it, which eliminates the possibility of material chipping[26], other than avoiding phase transformation, acid etching showed other advantages superior to sandblasting like the compatibility with resin cements containing MDP, the phosphate monomer of MDP chemically bonds with the hydroxyl groups on the etched zirconia surface, also the roughened surface created by HF acid treatment allowed the micro-mechanical retention of the cement, as well as corroding the zirconia surface exposing more reactive sites for chemical bonding with the resin cement, further enhancing the bond strength and durability [18].

Shear and microtensile bond strength testing are commonly utilized methods in restorative dentistry and dental materials research to compare products and techniques[27]. It is believed that shear forces are the main cause of restorative material bonding failures *in vivo*[28].

This study focused on evaluating and contrasting the SBS of IPS e.max CAD and IPS e.max ZirCAD prime, when applying two distinct surface treatments (HF acid etching and sandblasting), both before and after undergoing artificial aging. The null hypothesis posits that there is no significant differences in SBS between the two types of ceramic materials or the surface treatment methods, regardless of whether the specimens are aged or not.

MATERIALS AND METHODS

Study design

This study is an *in-vitro* study.

Materials

The materials utilized in this study are presented in table (1).

TABLE (1): List of the materials used in this study

Material	**Trade name/Manufacturer/City/ Country**
Monolithic Polychromatic gradient zirconia Batch #: Z03VHG	IPS e.max ZirCAD Prime, Ivoclar, Schaan, Liechtenstein One Partially sintered disc Incisal: 5Y-TZP Dentin: 3Y-TZP Shade: A2
Lithium disilicate glass-ceramic Batch# : Z01SBK	IPS e.max CAD HT, Ivoclar, Schaan, Liechtenstein Two partially sintered blocks Shade: A2
Ceramic etchant	Power C-etching gel. Paraná, Brazil (hydrofluoric acid 10%)
Light cured resin cement.	Calibra Ceram Adhesive Resin Cement, DENTSPLY Sirona,USA
Silane coupling Agent	Pre hydrolyzed Porcelain silane (Ethanol &Acetone) Bisco, Inc. Schaumburg, IL60193
Aluminum oxide particles	Henan Province, China

Methods:

Sample size calculation:

The determination of the sample size for this study was guided by a previous study [23]. The control group displayed a mean SBS of 9.26 with a standard deviation of 0.51. Assuming the intervention group would have an estimated mean of 10 and aiming for an effect size of 1.45, with 80% statistical power and a significance level (alpha) of 0.05, the sample size calculation suggested that 10 specimens per group were necessary. These calculations were performed using G*Power 3.1.9.7 software, tailored for an independent t-test. Consequently, each test group required a total of 10 specimens.

Sample grouping

For the SBS testing, 80 square ceramic specimens measuring 10 mm x 10 mm x 0.5 mm were prepared. These specimens were equally divided into two main groups, with each group containing 40 samples. Group A comprised IPS e.max CAD specimens, while Group B consisted of IPS e.max ZirCAD Prime specimens. Further, each group was subdivided into two batches of 20 samples, based on the surface treatment method employed: subgroup 1 underwent HF etching and silane coupling agent application, and subgroup 2 was subjected to sandblasting. In each of these subgroups, half of the specimens (10 samples) were tested before and the other half (10 samples) after undergoing artificial aging. This resulted in four distinct testing categories for each type of ceramic material. The aim was to assess the bond strength of the two different ceramics, comparing their performance with various surface treatments both before and after being subjected to accelerated aging processes.

Specimen's preparation

Specimens of IPS e.max CAD blocks and IPS e.max ZirCAD prime discs were precisely cut using a high-precision, water-cooled diamond saw (Isomet 4000, Buehler, USA), as illustrated in Figures 1 and 2. Using a blade rotating at 2500 rpm, discs of 0.6mm thickness were sliced from these blocks and discs. The resulting ceramic specimens were shaped to a size of 10mm x 10mm x 0.5mm. However, the zirconia specimens were initially made 20% larger to compensate for shrinkage during firing. The thickness of all ceramic specimens was verified using a digital caliper for accuracy. Subsequently, these specimens underwent a crystallization firing process as per the manufacturer's guidelines. This step caused the zirconia specimens to shrink to the final dimensions of 10mm x 10mm x 0.5mm, aligning with the experiment's specifications [25].

Surface treatments conducted, were as follows

1. **HF acid etching + silane coupling agent:** The samples underwent etching with 10% HF acid (Power C etching gel, Paraná, Brazil) for a duration of 60 seconds. After etching, they

Fig. (1): IPS e.max CAD block being crafted using Isomet 4000 device

Fig. (2): IPS e.max ZirCAD prime being crafted using Isomet 4000 device were rinsed with oil-free water and then air-dried. A thin coating of silane coupling agent (Bisco, Inc., Schaumburg) was then applied to the samples and allowed to sit for 60 seconds. This was followed by a gentle air blow for 5 seconds to finalize the process [24,29].

2. **Sandblasting:** The samples were subjected to sandblasting using a blaster device (Modulars, Silfradent) with 50 µm Al2O3 particles (Korox, Bego). This process was conducted at a steady distance of 10 mm for 15 seconds at 0.2 MPa pressure. Following the sandblasting, the samples were thoroughly rinsed under running water for 30 seconds and then air-dried to complete the preparation [27].

Composite resin (Calibra ceram adhesive resin cement, Dentsply Sirona, USA) was inserted into the openings of polyethylene tubes. These tubes, each with a diameter of 5 mm and a height of 3 mm [8], were then positioned atop the samples. Light polymerization was carried out for 20 seconds using an LED light-curing unit (Elipar S10, 3M Espe, St. Paul, MN), according to the manufacturer's guidelines. After this, the samples underwent thermocycling in a thermo-cycler device (SD-Mechatronik, Westerham, Germany). The process involved alternating temperatures between 5°C and 55°C over 5000 cycles, with each cycle having a 25-second dwell time and a 10-second interval between cycles [28].

Shear bond test

Each specimen underwent SBS testing using a universal testing machine (Instron model 3345, Instron Corp., Norwood, MA, USA). As depicted in Figure 3, the samples were fixed to a specially designed metallic sample holder with central cavity (fit to the dimensions of the ceramic slice, which was glued to the base with cyanoacrylate glue) and secured to the lower fixed compartment of the testing machine by tightening screws. The samples were subjected to a load until failure at a crosshead

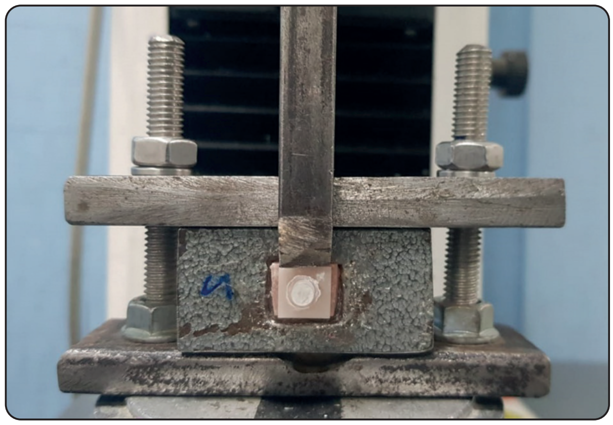

Fig. (3): samples subjected to shear bond strength test using universal testing machine

speed of 0.5mm/min, utilizing a 5kN load cell. The bond strength values were calculated by dividing the failure load (expressed in Newtons) by the bonded surface area (in square millimeters), with the results presented in Megapascals (MPa). The load and extension data collected during the tests were recorded and analyzed using Bluehill Lite software (Instron 3345, MA, USA), which was integrated with the testing frame [30].

Statistical analysis:

Statistical analysis was conducted using SPSS 20, Graph Pad Prism, and Microsoft Excel 2016. All quantitative data are presented as mean and standard deviation. The data was tested for normality using the Shapiro-Wilk and Kolmogorov-Smirnov tests. Independent t-tests made comparisons between different surface treatments and groups. The p-value < 0.05 was considered significant.

RESULTS

Statistical analysis:

Statistical analysis revealed insignificant p-values (>0.05), indicating the data followed normal distributions in both groups. Paired t-tests compared measurements before and after aging. Independent t-tests made comparisons between different surface treatments and groups.

Effect of thermocycling (comparison between before aging and after aging).

The results showed a significant reduction in bond strength ($P<0.05$) across all groups, except for the lithium disilicate ceramic (IPS e.max CAD) treated with HF, which showed no significant change ($P=0.48$). For the zirconia ceramic (IPS e.max ZirCAD prime) treated with sandblasting, there was a decrease in bond strength with an average difference of 9.89 MPa (P=0.0001). When treated with HF, the bond strength of zirconia ceramic dropped with an average difference of 7.44 MPa (P=0.0005). Similarly, lithium disilicate ceramic with sandblasting showed a decrease in bond strength. For detailed information, refer to Table 1 and Figure 1 in the study.

Effect of surface treatment (comparison between Sandblasting and HF acid).

The results indicated a significant disparity in the performance of these two treatments across all groups ($P<0.05$). In the case of Zirconia ceramic (IPS e.max ZirCAD prime), specimens treated with sandblasting exhibited notably higher bond strength values than those treated with HF acid. On the other hand, for Lithium disilicate ceramic (IPS e.max CAD), the bond strength values were significantly lower for specimens treated with sandblasting compared to those treated with HF acid. Following the aging process, surface treatment with sandblasting (4.87 ± 2.06) was significantly lower than that with HF acid (8.83 ± 6.66) as $P=0.015$, despite the material used. These variances in bond strength between the two surface treatments for each type of ceramic are detailed numerically in Table 1 and illustrated graphically in Figure 1.

Effect of material (comparison between IPS e.max and IPSe.max ZirCAD prime):

Independent t-tests were employed to analyze the differences between Zirconia ceramic (IPS e.max ZirCAD prime) and Lithium disilicate ceramic (IPS e.max CAD) in various surface treatments, both before and after aging, thereby assessing the impact of the material type. There were notable differences observed between the two types of ceramics in all

TABLE (1) Shear bond strength (SBS) before and after aging in sandblasting and hydro-fluoric acid surface treatment in both Zirconia ceramic (IPS e.max ZirCAD prime) group and Lithium disilicate ceramic (IPS e.max CAD) group.

		Before aging		After aging		Paired t test					
						MD	SD	SEM	95% CI		P value
		M	SD	M	SD				L	U	
Zirconia ceramic (IPS e.max ZirCAD prime)	Sand- blasting	13.01	3.89	3.12	0.96	9.89	3.59	1.13	7.32	12.46	0.0001*
	Hydro- fluoric acid	9.8	3.02	2.36	0.35	7.44	3.27	1.03	5.1	9.78	0.0005*
	P value Independent t test	0.054		0.03*							
Lithium disilicate ceramic (IPS e.max CAD).	Sand- blasting	14.16	3.93	6.62	1.09	7.54	4.39	1.39	4.4	10.68	0.0004*
	Hydro- fluoric acid	16.15	3.5	15.3	0.64	0.85	3.69	1.17	-1.79	3.49	0.485ns
	P value Independent t test	0.24		0.0001*							

M: mean SD: standard deviation MD: mean difference SEM: standard error mean CI: confidence interval L:lower arm U:upper arm *Significant difference as P<0.05 Ns: non-significant difference as P>0.05.

TABLE (2) Evaluation of material effect on Shear bond strength of Zirconia ceramic (IPS e.max ZirCAD prime) and Lithium disilicate ceramic (IPS e.max CAD) before and after aging in both surface treatments using Independent t test:

Material	Thermocycling	Zirconia ceramic (IPS e.max ZirCAD prime)		Lithium disilicate ceramic (IPS e.max CAD).		Difference (Independent t test)				
						MD	SEM	95% CI		P value
		M	SD	M	SD			L	U	
Sand- blasting	Before aging	13.01	3.89	14.16	3.93	1.15	1.75	-4.82	2.52	0.52ns
	After aging	3.12	.96	6.62	1.09	3.50	0.46	-4.47	-2.53	0.001*
Hydro- fluoric acid	Before aging	9.80	3.02	16.15	3.50	6.36	1.46	-9.43	-3.28	0.001*
	After aging	2.36	.35	15.30	.64	12.95	0.23	-13.43	-12.46	0.001*
Overall	Before aging	11.40	3.77	15.15	3.77	3.75	1.19	-6.16	-1.34	0.001*
	After aging	2.74	.80	10.96	4.54	8.22	1.03	-10.31	-6.14	0.001*

M: mean SD: standard deviation MD: mean difference SEM: standard error mean
CI: confidence interval L: lower arm U: upper arm *Significant difference as P<0.05
Ns: non-significant difference as P>0.05

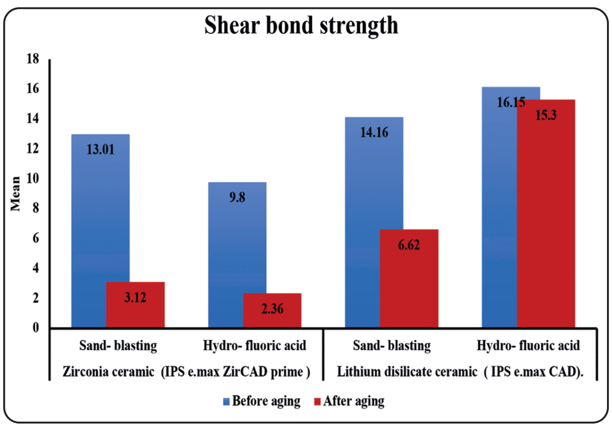

Fig. (4) Bar chart representing shear bond strength before and after aging in sandblasting and hydro-fluoric acid in both groups.

groups before and after aging (P<0.05), with the exception of the pre-aging phase with sandblasting where P=0.52. Distinctions in bond strength between the two types of ceramics, considering different surface treatments and aging conditions, are detailed in Table 2 of the study.

DISCUSSION

Achieving a strong bond is a main prerequisite for ensuring the restoration durability. Thus, it is crucial to know the best combination of material, surface treatment, and type of cement used to reach this goal [31]. Otherwise, a crack will form within the restorative material, propagating through the cement interface leading to permanent fracture [32].

Ceramic samples were created for the current investigation using IPS e.max CAD HT and IPS e.max ZirCAD Prime. This study chose lithium disilicate glass ceramic as a commercial comparison to the new zirconia. Because of its strong mechanical qualities, it may be employed in thin parts without detracting from the final restoration's appearance or usefulness [33, 34].

The second ceramic type used in this investigation was IPS e.max ZirCAD Prime, a freshly released monolithic zirconia. In order to increase the clinical uses of aesthetic zirconia restorations, this material is manufactured utilizing a gradient technique that combines very transparent 5Y-TZP in the enamel/outer area with high strength 3Y-TZP in the interior dentin/body [35, 36].

Two different surface treatments were applied to ceramic plates before SBS evaluation:1) HF acid etching followed by silane primer [37-39], 2) Sandblasting [9, 40]. Resin cement was applied for a sealed interface [41]. Thermal cycling procedure was used extensively in previous studies to simulate the intra-oral conditions [41, 42].

SBS testing was conducted on specimens' post preparation, enabling effective protocol screening, substrate profiling, and material composition using a universal testing machine [27,44,45].

Regarding the effect of thermocycling on SBS, the present results showed a significant decrease in values for all test groups following artificial aging. This led to rejection of that portion of the null hypothesis predicting no difference between aged and non-aged specimens. The reductions in bond strength due to thermal cycling aligned with previous studies by Kilinc et al.[46] and Subasi et al.[47], confirming the SBS between different ceramic types can be significantly impacted by simulated aging processes.

Debonding of restorations occurs when the bonding interface cannot withstand integrity within the moist oral environment. The likelihood of this event is associated with the level of water sorption by the ceramic material under wet aging conditions[47-49].

Our findings showing decreased bond strength post-thermocycling, agreed with similar studies by Lee and Lee [49] Fathpour et al., [50] and Ozcan et al. [51]. These studies also concluded that thermocycling universally reduced shear bond values across all tested groups.

Significant differences were found between groups in terms of how various surface treatments influenced SBS after aging. Specifically, sandblasted IPS e.max ZirCAD prime showed higher post-aging bond strength compared to IPS e.max ZirCAD prime treated with silane and HF acid. However, when considering overall aged results across both ceramic types, HF acid etching proved to be a more effective surface treatment method than sandblasting in terms of maintaining higher SBS post-thermocycling. The two ceramics responded differently to the surface treatments in their ability to resist bond deterioration from artificial aging.

The current findings agree with those of Abed et al.[52], who stated that bond strength is substantially increased by 50 µm aluminum oxide sandblasting, particularly when combined with an MDP-containing bonding agent to achieve maximum mean SBS. This is due to enhanced wetting ability and bonding promotion via MDP. These findings corroborated those of Kulunk et al. [53] and Lee at al. [49], who found that the sandblasting protocol increased the bond strength endurance through increasing the micro-irregularities. Gerdzhikov et al. [54] study was also comparable to the present study.

Taking into account the contentious impact of HF acid etching protocol on zirconia ceramics, Altan et al's. [24] study elaborated that any surface treatment technique would impact positively the bond strength of zirconia-based materials to resin cement. It is related to the fact that HF acid etching enhances the surface energy and increases the wettability of zirconia, though it does not alter its surface morphology. On the other hand, Lee et al [49] and Sriamporn et al. [55] demonstrated the capability of HF acid surface treatment to optimize the bond stability, as the HF acid corroded the zirconia particles leading to their dislodgment thus creating porosities and roughness. Additionally, there was an increase in the inter-particle space and a decline in the particle size. This can be attributed to the greater chemical reactivity and faster dissolution of atoms outside the crystal compared to those inside, So the zirconia's crystallographic orientation determines its chemical reactivity. On the contrary, El Zayat et al.[29] experiment concluded that HF acid etching to zirconia revealed the lowest results regarding the bond strength. This conclusion was confirmed by several studies counseled by Pjetursson et al. [4], Inakuchi et al [14] and Saleh et al [7].

Our findings on HF acid etching and silane treatment of lithium disilicate ceramics aligned with previous studies by Hashem et al. [30] and Ozcan et al. [51] showing that acid etching optimizes glass ceramic bonding, allowing stable adhesion. Another agreement was seen with El-Zayat et al.'s [29] work concluding that combining acid etching and silane coupling agent application resulted in the highest SBS for glass ceramic substrates. Our lithium disilicate results reinforce these studies in that HF acid surface conditioning followed by a silane layer produced superior bonding durability compared to other surface treatments for this glass-containing ceramic.

Regarding the impact of ceramic type on the SBS value, the results of the current study revealed that the SBS value of IPS e.max ceramic was higher than IPS e.max ZirCAD prime ceramic before and after aging protocol. These results were in agreement with Ozcan et al. [51], Altan et al. [24], and Klinik et al.[46], investigations whose findings demonstrated that, both before and after ageing, the SBS values of lithium disilicate ceramic were greater than those of new zirconia, resulting in a more robust link between glass ceramics and resin cement. This finding might be explained by the fact that the glassy phase is selectively dissolved by HF acid etching, exposing the crystalline phase and producing a jagged, porous surface that increases the surface area penetrated by the resin. The microstructure of zirconia ceramics is far more uniform and less susceptible to acid etching; yet, manually roughening its surface by sandblasting significantly increases its SBS [24, 46, 51].

Kwon et al. [56] raised an opposite conclusion to the present study, stating that the bond strength of 5Y-TZP, 3Y-TZP and combined novel zirconia was comparable to lithium disilicate. This dissimilarity may be due to the variations in aging technique or the material brand.

CONCLUSION

Within the limitation of the current *in-vitro* study:

- Thermocycling decreased the shear bond strength of both ceramics regardless of the surface treatment protocol applied.
- Sandblasting surface treatment was better than HF acid with silane when applied to zirconia ceramic, while in Lithium disilicate sandblasting surface treatment showed lower results than HF acid treatment protocol.
- IPS ZirCAD prime zirconia ceramic still revealed weaker bond stability before and after aging in comparison with IPS e.max CAD lithium disilicate ceramic.

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