



Shear Bond Strength of Translucent Zirconia and Lithium-Disilicate Glass Ceramic to Dentin using Different Surface Treatments

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Abstract

Purpose: The aim of this study is to access shear bond strength of translucent zirconia and lithium disilicate to dentin using different ceramic surface treatments.

Material and Methods: twenty discs of translucent zirconia were obtained by cutting InCoris TZI blocks into discs (4mm diameter, 2mm thickness). Twenty lithium disilicate discs (E-max) of the same dimensions were obtained by pressing technique. Each group of discs (n=2) were subdivided into 4 groups according to the type of surface treatment received; air abrasion, hydrofluoric acid etching, tribochemical silica coating and control group which didn't receive any surface treatment. Self-adhesive universal resin cement RelyX U200 was used without any pretreatment steps, shear bond test was done at the ceramic dentin interface and the load required for debonding was recorded. The mode of failure of each specimen was determined by inspecting the bonding surfaces of each specimen using stereomicroscopy.

Results: Comparing both materials with the different surface treatments revealed that the maximum mean load was recorded in lithium disilicate with HF surface treatment, whereas the lowest value was obtained with translucent zirconia with HF surface treatment. ANOVA test revealed that the difference between both materials with different treatments was extremely statistically significant. Failures were classified as adhesive if less than 25% remained on the tooth structure or the disc and mixed if certain areas exhibited adhesive fracture.

Conclusion: the surface treatment can significantly affect the shear bond strength of translucent zirconia to dentin. The best surface treatment can be used to achieve a strong reliable bond to translucent zirconia is tribochemical silica coating. Hydrofluoric acid etching achieved a strong bond when used with lithium disilicate glass ceramic. Failure mode analysis revealed an increase in the adhesive failures corresponded to a decrease in bond strength.

Key words: Translucent zirconia, Lithium disilicate, Cojet, Primer, Adhesion

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Introduction

Dental restorations using all ceramic materials in association with adhesive cements have become popular to replace tooth structure lost by dental disease in an esthetic manner. Strength of the ceramic material is one of the parameters that determine the longevity of an all-ceramic restoration^[1] Adhesion between tooth structure and the restoration is one of the most important factors determining the success of a restoration^[2]. The adhesion techniques used in all-ceramic restorations depend on the chemical composition of the ceramic system, and surface treatments are necessary to ensure adhesion between the luting agent and the ceramic surface. In addition, the composition of the ceramic determines which surface treatment is appropriate. For example, hydrofluoric acid etching and silanization are obligatory steps for silica ceramics. However, ceramics with high alumina or zirconia cannot be roughened by hydrofluoric acid etching because they do not contain a silicon dioxide (silica) phase^[3]. Zirconia cores are composed of glass-free, polycrystalline microstructure, and consequently show off outstanding long-term stability^[4]. Nevertheless, most previous studies have examined hydrofluoric acid-etched zirconia in terms of its mechanical properties and its surface bond strength with the resin cement. Specifically, these studies have investigated various surface treatments applied to improve bonding to the zirconia

ceramic, including selective infiltration etching (SIE), laser etching, alumina coating, silica ceramic coating, tribochemical silica coating, and airborne-particle abrasion (also called sandblasting)^[5]

Different types of surface treatments can be used in order to produce micromechanical retention on the ceramic surface^[5]. Etching the inner surfaces of ceramics with glassy matrix using hydrofluoric acid followed by the application of a silane coupling agent is an efficient conditioning method for bonding resin composite^[6]. For ceramic surface treatment, the acid reacts with the glass matrix that contains silica and forms hexafluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of ceramic becomes rough, which is expected for micromechanical retention on the ceramic surface^[5].

Air-particle abrasion is a prerequisite for achieving sufficient bond strength between the resins and high-strength ceramics that are reinforced either with alumina or zirconia 6. The air abrasion systems rely on air-particle abrasion with different particle sizes ranging from 30 to 250 μm ^[6,7]. The abrasive process removes loose contaminated layers and the roughened surface provides some degree of mechanical interlocking or 'keying' with the adhesive^[6,8]. It can be argued that the increased roughness also forms a larger surface area for the bond^[6,7]. Thus, sandblasting with aluminium oxide particles was proposed as a surface treatment option that produces irregularities in acid-resistant ceramics^[9]. Another modern surface conditioning method which can be used with high crystalline ceramic material is Silica coating. In this technique, the surfaces are air abraded with alumina particles coated with silica. The blasting pressure results in the embedding of silica particles in the ceramic surface, rendering the silica modified surface chemically more reactive to the resin through silane coupling agents^[7].

Studies performed in recent years have shown that sandblasting procedures do not have any negative effect and can be safely used on zirconia ceramic restoration surfaces. Sandblasting increases the roughness of the surface, and in turn, the surface energy and wetting capacity of the restoration^[10].

Cojet is an in-office silica coating system that uses 30- μm silica-modified Al_2O_3 particles (Cojet-Sand) blasted to the surface, followed by the application of a silane agent (ESPE-Sil). These silica coating systems have showed adequate bond strength values in several studies^[9, 11-13]. Establishment of a strong chemical bond between a ceramic surface and a resin luting agent can be achieved by treatment with a silane coupling agent^[8]. It is also evident that air-abrasion with Al_2O_3 particle, enhances the action of silane by generating more hydroxyl groups on the ceramic surface to react with the silanol groups of the silane. This mode of enhancement is in addition to the micromechanical retention that air-borne particle abrasion provides^[6,14].

Recently, several new ceramic primers have been introduced to the dental market to increase chemical bonding to zirconia ceramics. These include primers that contain a phosphoric acid monomer, 6-methacryloxyhexylphosphonoacetate (6-MHPA), (AZ, primer) or 3-trimethoxysilylpropyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), ethanol (Clearfil Ceramic Primer) and organophosphate monomer, carboxylic acid monomer and others (Z-Prime Plus). The application of MDP containing bonding agents can increase bond strength to zirconia because of an interaction between the hydroxyl groups of MDP and the cationic surface of zirconia.^[3, 15]

Glass ionomer cements from conventional luting agents are often used in the cementation of zirconia ceramic restorations; however, adhesive cementation is preferred due to inadequate retention and resin-bonded fixed dental prosthesis. Self-adhesive resin cements

have been developed to simplify bonding procedures. Nevertheless, the results from the literature on bond strength to zirconia ceramics remain very controversial.^[3]

Shear and micro tensile bond strength are methods frequently used in restorative dentistry and dental materials studies^[16]. These tests allow comparisons between products and techniques^[17,18]. Shear stresses are believed to be major stresses involved in in-vivo bonding failures of restorative materials^[19].

Thus, in this study shear bond strength of translucent zirconia will be assessed compared to lithium disilicate using the same surface treatments.

Material and Methods

Forty intact recently extracted human molars were selected with approximate similarity in shape and size to facilitate standardization of the samples. The occlusal surface of the teeth was flattened using a wheel diamond stone perpendicular to the long axis of the tooth. Guiding grooves were made to expose the dentin (1.5mm) and obtain at least 4mm diameter flat occlusal dentin surface. Forty cylindrical plastic tubes were prepared to be used as a holder for the tooth and acrylic resin. The flattened occlusal surface was placed on a clean glass slab and the plastic tube was placed around the tooth. Auto curing acrylic resin¹ was mixed and poured into the plastic tubes. The prepared teeth were left until complete curing of the resin then root amputation was done.

Forty ceramic discs were divided into 2 main groups according to the type of ceramic material into: Translucent Zirconium oxide ceramic (n=20) and Lithium disilicate glass ceramic (n=20). Translucent zirconia specimens were obtained by cutting InCoris TZI blocks (size 20/19) which was placed in the milling chamber of the MCXL unit. Lithium disilicate discs (E-max) were obtained by pressing technique (lost wax technique). The thickness of the discs was checked using a metal gauge caliber.

For each group (n=20), the discs were divided into 4 subgroups according to the surface treatment received as follows:

Air-borne particle abrasion:

Ceramic specimens were cleaned for five minutes in an ultrasonic bath containing distilled water and then air-dried. Air-borne particle abrasion was performed using 110 μm grain sized alumina particles at a pressure of 2 bar approximately with a distance of 2 cm for 5 seconds.

Hydrofluoric acid etching:

In hydrofluoric acid etched subgroups, the translucent zirconia discs were etched with 9% hydrofluoric acid gel for 90 s. As for the Emax discs, etching was performed for 60 s with 9% HF gel according to the manufacturer strict regulations. The ceramic surfaces were etched by expressing an even coating of the acid-etch onto the ceramic surface in the laboratory under ventilation, wearing acid-resistant gloves and protective glasses. The etching gel was removed by placing the vacuum tip next to the surface and then rinsing with water spray thoroughly for 30 s.

Tribochemical silica-coating:

Silica-coating process was then achieved using an intra-oral air abrasion device Cojet 3 (fig.36) filled with cojet-sand (30 μm SiO_x particles) from a distance of approximately 2 cm at a pressure of 2 bar for 5 seconds. Following the surface conditioning, the remnants of sand particles were gently air blown, and ultrasonic cleaning was done for 5 minutes. The substrates of the three groups were air dried and silane coupling agent was applied with a mini brush and left for 60 sec followed by air dryness to ensure silane evaporation

Ceramic specimens of the control group didn't receive any surface treatment.

A specially designed loading device was constructed for standardization of load during cementation. It was composed of metal rectangular base 20 cm in length, 12 cm in width and 2 cm thickness, a fixed projecting arm 13cm length, a lever arm 37 cm length connected to the fixed projected arm through a joint to permit its vertical movement. The lever arm was designed to carry the load 1 Kg and a piston to fit inside the assembling copper sleeve cylinder. On the base of the device there is a fixed projecting part 45mm in diameter and 3mm in height on which teeth blocks and the guiding cementation device would be seated after the ceramic disc was cemented.

Self-adhesive universal resin cement RelyX U200 was used, without any pretreatment steps, i.e. without etching, priming or bonding. The mixed cement was applied evenly to the working area then the disc was seated firmly. Immediately after applying the luting cement on the disc, the cemented disc and the tooth block were placed in the

guiding cementation device and were placed in their position on the base of the loading device and immediately received a static cementing load of 1 Kg magnitude and maintained for 3 minutes. Light curing was done for 20 seconds per surface to insure polymerization according to the manufacturer instructions.

Shearing test was done by compressive mode of load applied at ceramic-substrate interface using a mono-beveled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min. The load required for debonding was recorded in Newton. The mode of failure of each specimen was determined by inspecting the bonding surfaces of each specimen using stereo photomicroscopy 4 with magnification 1.5x for the disc and 1x for the tooth structure. Failure mode was classified into four types: (1) adhesive failure between resin luting agent and dentin; (2) adhesive failure between resin luting agent and ceramic; (3) cohesive failure within the resin luting agent; and (4) mixed mode of failure. Photographs of representative specimens were taken.

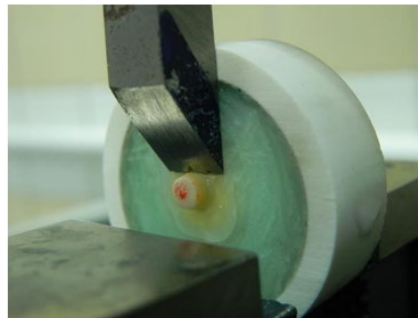


Fig 1: Sample mounted on the testing machine

Results

Comparison between both materials used with the same surface treatment was carried out by Student t-test of two independent samples, while analysis of variables (ANOVA) test and Tukey's post hoc test were applied to compare the 4 surface treatments within each material. Results were expressed in the form p-values that were differentiated into: Non-significant when $p\text{-value} > 0.05$, Significant when $p\text{-value} \leq 0.05$, Highly significant when $p\text{-value} \leq 0.01$, and Extremely significant when $p\text{-value} \leq 0.001$.

-Comparing both materials with the different surface treatments revealed that the maximum mean load was recorded in lithium disilicate

with HF surface treatment, whereas the lowest value was obtained with translucent zirconia with HF surface treatment. ANOVA test revealed that the difference between both materials with different treatments was extremely statistically significant.

Comparing each modality of surface treatment within the 2 materials revealed a greater mean maximum load in the translucent zirconia groups treated as control or with CJ. On the other hand, greater mean maximum loads were observed in lithium disilicate groups treated with AB or HF. Unpaired Student's t test revealed the difference in mean maximum load of the materials in relation to the surface treatment was statistically significant except in the control group.

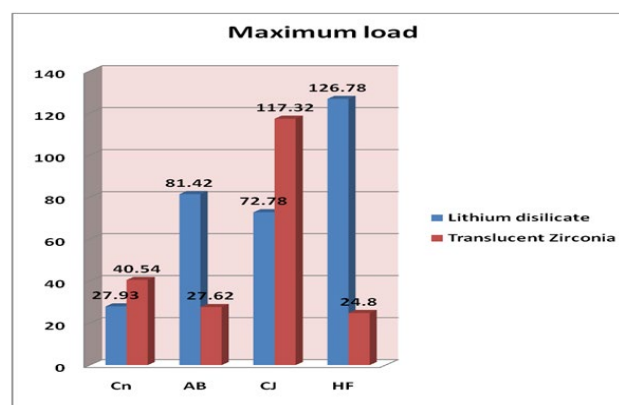


Fig 2: Maximum load in all groups

	Cn		AB		CJ		HF	
	L	Z	L	Z	L	Z	L	Z
Mean	27.93	40.54	81.42	27.62	72.78	117.32	126.78	24.8
Std Dev	5.54	32.32	34.98	14.36	22.51	15.04	66.78	11.60
Min	19.53	9.79	24.93	11.30	48.49	102.04	44.45	15.60
Max	34.53	87.40	114.59	43.35	104.80	142.40	216.50	43.98
T value	0.8599		3.1815		3.6789		3.3643	
P value	0.4149		0.01300		0.0062**		0.0099**	

Student's t test, *statistically significant, **very significant.

Table 1: Difference in maximum load in relation to the surface treatment

Type of ceramic	Mode of failure Surface treatment	1	2	3	4
		A	AB	1	1
	HF	4			1
	CJ	1			4
	Cn		5		
B	AB	4	1		
	HF		4		1
	CJ		1	1	3
	Cn		5		

A summary of the modes of specimen failure is presented in **Table (2)**

Abbreviations:

A: Lithium disilicate glass ceramic, B: Translucent Zirconia,

AB: Airborne particle abrasion, HF: hydrofluoric acid,

CJ: Cojet system, Cn: control group.

1: Adhesive failure between resin luting agent and dentin,

2: Adhesive failure between resin luting agent and ceramics

3: Cohesive failure within resin luting agent and

4: Mixed mode of failure

Failures were classified as adhesive if less than 25% remained on the tooth structure or the disc and mixed if certain areas exhibited adhesive fracture

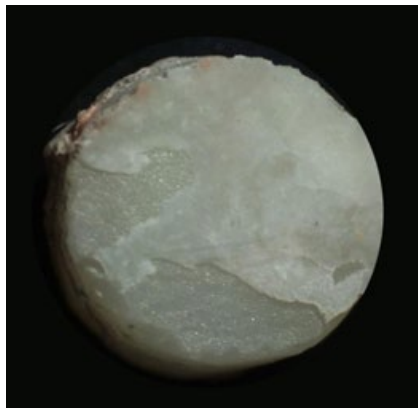


Fig 3: Translucent zirconia treated with air abrasion showing adhesive failure (more than 50% of the resin cement is adherent on the ceramic disc)



Fig 4: Translucent zirconia treated with hydrofluoric acid showing adhesive failure between resin and ceramic



Fig 5: Translucent zirconia treated by cojet system showing cohesive layer within the resin cement (cohesive failure)

Discussion

Zirconia ceramics have been widely used as a framework material for tooth-supported or implant-supported restorations owing to their excellent biocompatibility, enhanced strength, and inherent esthetic properties.^[20] Adhesive luting has been known to increase the fracture resistance of all-ceramic restorations^[21]. Thus, it is very important to select the best combination of resin cement and surface treatment for durability^[4].

Various surface treatments have been explored to improve bonding to zirconia ceramic, including SIE, laser etching, alumina coating, silica ceramic coating, tribochemical silica coating and sandblasting.^[22] Previous studies have found hydrofluoric acid etching to be incapable of roughening high-alumina and zirconia ceramics due to their lack of a silica phase. Despite this, several studies have investigated the effects of hydrofluoric acid etching on the mechanical properties of zirconia and on the bond strength between the zirconia's surface and resin cements^[3]. For example, Sriamporn et al. evaluated dental zirconia's surface morphology following hydrofluoric acid etching and examined changes to its crystal structure. Their results showed that hydrofluoric acid is able to etch dental zirconia ceramics by creating micro-morphological changes on its surface.^[23] Hydrofluoric acid is the most commonly used acid in dental clinical practice, and 4–10% concentrations of hydrofluoric acid have been found to be safe and are preferred in dental applications.^[3]

Etching the inner surface of glass-ceramic restoration with hydrofluoric acid followed by the application of a silane coupling agent is a well-known and recommended method to increase the bond strength. The lithium disilicate glass ceramic is used in this study for comparing the results of the translucent zirconia.

Application of priming agents containing MDP yielded the durable bond strength of resin-based luting agent to zirconia ceramics^[3].

However, in a study conducted by Altan et al, HF acid etching group showed higher bond strength compared to control groups regarding Vita YZ HT, Sirona inCoris TZI and IPS e.max ZirCAD. It can be associated that even HF acid etching does not change surface morphology of zirconia, it increases wettability and surface energy. HF acid etching treatment resulted in the highest bond strength values for Vita Suprinity blocks. These results are in agreement with the findings of Ataol et al. and Sato et al., who stated that HF acid etching group showed the highest bond strength for Vita Suprinity. These results may be explained by the fact that HF acid etching enhanced micromechanical retention by dissolving the glassy matrix of Vita Suprinity.^[22]

The success of a zirconia restoration depends on the quality, strength, and durability of the bond between the resin cement and the restoration.^[24] Owing to zirconia's opacity, the polymerization of light-cure resin cement may be impaired; therefore, dual- and chemical-cure resin cement are recommended for luting zirconia ceramics.^[3] Self-adhesive resin cement doesn't demand tooth structure pretreatment, therefore simplifying the clinical steps.^[25] It was also found that RelyX Unicem produced bond strength to dentin which was not significantly different from the other resin based luting agents^[26]. The dual-cured nature of RelyX Unicem is preferable thus removing the potential for limited light transmission through zirconia^[27].

Human teeth have been commonly used for the in-vitro testing for simulating the clinical conditions^[21]. Recently extracted human molars were used in this study for the purpose of standardization.

This study is aimed at assessing the shear bond strength of dentin to translucent zirconium oxide ceramic compared to that of lithium-disilicate glass ceramic using three types of ceramic surface treatments: air abrasion (sandblasting), hydrofluoric acid etching and tribochemical silica-coating. In addition to control group, where there is no surface treatment applied.

The results of the present study revealed that the greatest mean of maximum load of translucent zirconia was recorded in the group treated with cojet system, followed by control group, and then followed by air abraded group, whereas the group treated with hydrofluoric acid showed the least values. The results revealed that the difference between the 4 surface treatments was statistically extremely significant regarding the maximum load. However, neither hydrofluoric acid etching nor silane coupling agent is enough to improve the bond strength between zirconium oxide ceramics and resin cements because zirconia is polycrystalline, silica free in nature and thus resistant to acid etching^[4]

However, Sandblasting enhances bond strength by increasing surface area and roughness. Zhang et al. claimed that sandblasting causes formation of microcracks which decrease strength of zirconia. However, it was proven that resin cement flowed into microcracks and therefore significantly strengthened the ceramic. Moreover, in CoJet system, silica particles not only roughen the surface, they also support chemical retention by bonding silane and silica-coated zirconia surface. The present studies reported that CoJet application increased bond strength values more than did sandblasting^[22].

The results of our study are in compliance with the results of a study conducted by Ozcan et al^[28] which showed that acid etching did not improve the bond strength to zirconium oxide ceramic; however, tribochemical silica coating enhanced the bond.

Also, the results are in agreement with that of a study conducted by Zrilie et al^[29] which showed that cojet system exhibited highest bond strength values and also showed that the control group exhibited higher values than the hydrofluoric acid etched group.

In the present study, the control translucent zirconia group had a mean maximum load which was extremely significantly greater than the air abraded group. These results may be attributed to the fact that air abrasion might compromise the mechanical strength of the ceramic itself by initiating surface defects that can be stress concentration sources, leading to failure. Therefore, reducing the pressure during air-abrasion or omitting air-abrasion completely might be advantageous in reducing the negative surface effects caused by air-abrasion^[30].

Opposing to these findings, Aboushelib^[31] showed that the control zirconia group demonstrated spontaneous failure and relative bond stability was achieved when the surface of the specimens was air-borne particle abraded. This may be attributed to the difference in the test where microtensile bond strength test was used and artificial aging was done in that study.

It was also shown in this study that the greatest mean of maximum load of lithium disilicate glass-ceramic e.max was recorded in the group treated with hydrofluoric acid, followed by air abraded group, and then followed by tribochemical silica coated group (cojet system), whereas the control group showed the least values. The results also revealed that the difference between the 4 surface treatments was statistically significant regarding the maximum load.

These results are in coincidence with that of a previous study conducted by Salvio et al^[32] which showed that air abrasion could not provide a mechanically retentive surface as satisfactorily as etching with hydrofluoric acid can with the lithium-disilicate glass ceramic.

Our results are in compliance with another study conducted by Ozcan et al^[38] which showed that acid etching demonstrated higher results for glass ceramics. A different study conducted by Menezes et al^[58] showed that results were superior with hydrofluoric acid etching, lower with air-borne particle abrasion and least with the control group.

The results of the present study are in agreement with the results of Panah et al^[8] which showed that highest bond strengths of lithi-

um-disilicate glass ceramic was obtained in the group treated by both air-abrasion and hydrofluoric acid etching which were in turn not significantly different from the bond strength of the group treated with hydrofluoric acid only. And both groups are significantly higher than group treated with air-borne particle abrasion alone.

Analysis of Zirconia Ceramic Surfaces with Different Surface Treatments where the latest studies have reported that the long-term success levels of zirconia-based all-ceramic restorations depend on the preparation techniques of inner ceramic surfaces before cementation, characteristics of the attaching cement, and the durability and the strength of the attachment between the cement and the ceramic^[10,34]. To this respect, changes in the surface morphology of zirconia ceramic samples, to which different surface treatments were applied, were analyzed with stereomicroscope in our study. Some researchers have stated that roughening the inner surfaces of ceramic restorations increase the surface area and enable the wetting capability of ceramic surfaces of resin-based materials to increase. Some studies have reported that the sandblasting method creates rougher surfaces^[10].

Failure mode analysis revealed that the results of the shear bond tests of the present study are consistent with the failure modes observed. An increase of adhesive failure mode corresponded to a decrease in bond strength^[35]. It seems that the higher the shear bond strength value a specimen records, the higher the rate of cohesive failure rather than adhesive failure is observed^[4,36].

In this study, the air abraded zirconia group showed adhesive failure between adhesive resin and dentin. These results are in agreement with other studies^[37,38] where most failures were adhesive involving the dentin surface. Therefore, the interface between the resin cement and the dentin surface was the weak line.

The results of this study showed adhesive failure between resin cement and ceramic in the hydrofluoric acid etched zirconia group. This may be due to the fact that hydrofluoric acid etching does not produce effective retention in zirconia-based ceramics as it is polycrystalline silica-free ceramics.

Fracture mode analysis also showed mixed mode of failure in the zirconia group treated by tribochemical silica coating. This can be attributed to the relatively higher bond achieved by increased surface roughness and increased silica content resulting from silica coating.

Altan et al showed that SEM images of CoJet groups have micro retentive grooves with finer texture than sandblasting groups. SEM analysis supports the bond strength values. Similar to Altan's study, Elsaka stated that specimens treated with CoJet exhibited significantly higher bond strength than sandblasted specimens. These results may be explained by the fact that CoJet enhanced mechanical and chemical bonding^[22].

Results of this study showed that zirconia control group tends to fail at the zirconia-ceramic-resin cement interface. These results came coincident with a study conducted by Yun^[4]. This may be attributed to the low bond achieved with the lack of surface treatment which is important to achieve a reliable bond between ceramic and the cement. Fracture mode analysis of this study revealed that air abraded lithium disilicate glass ceramic showed mixed mode of failure. These results are in agreement with the results obtained by Nagai et al^[39] which also showed mixed mode of failure. The results of this study showed adhesive failure between resin cement and dentin in the hydrofluoric acid etched lithium disilicate glass ceramic group. These results are in compliance with a study conducted by Kumbuloglu et al^[40] where fracture mode analysis revealed adhesion debondings.

Fracture mode analysis of this study did not show cohesive failure in all the lithium disilicate glass ceramic groups. Also, fracture within the ceramic material was not observed in any substrate specimens.

This came in coincidence with Abo-Hamar^[26] and Nagai et al^[39] where fracture mode analysis showed nearly 60% adhesive failures and 40% mixed failures with no cohesive failures.

Among the limitations of this study is that shear bond strength studies do not simulate the clinical conditions and the cementation process. Thus, this testing design does not reflect the factors that may affect the performance of the cement^[41]. It is sometimes difficult to compare results obtained due to the lack of standardization of the techniques and materials used in the literature.

Conclusion

Within the limitations of this study, the following conclusions can be drawn:

1. Surface treatment significantly affected the dentin shear bond strength for both lithium disilicate glass ceramic and translucent zirconia.
2. Hydrofluoric acid etching achieved a strong bond when using lithium disilicate glass ceramic, while it produced a poor bond with translucent zirconia.
3. The best surface treatment can be used to achieve a strong reliable bond to translucent zirconia is tribochemical silica coating.
4. Failure mode analysis showed adhesive, cohesive and mixed mode of failure. However, most of failures were adhesive even with high results and this reflects negatively on bond strength.

Conflicts of interest:

The authors declare that there is no conflict of interest related to this study.

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