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Effect of different design and surface treatment on the load-to-failure of ceramic repaired with composite

Mauricio JM Tavares, Flavia LB Amaral, Roberta T Basting, Cecilia P Turssi, Fabiana MG França

Faculdade São Leopoldo Mandic, Instituto de Pesquisas São Leopoldo Mandic. Campinas/SP. Brazil.

ABSTRACT

Glass ceramics are widely used to manufacture esthetic veneers, inlays, onlays, and crowns. Although the clinical survival rates of glass-ceramic restorations are favorable, fractures or chips are common. Certain cases can be repaired with direct composite. ***Aim:*** *The aim of this study was to investigate the interaction effect of different designs and surface treatments on the load-to-failure of lithium disilicate glass-ceramic repaired with nanofilled composite.* ***Materials and Method:*** *Lithium-disilicate glass-ceramic slabs (IPS e.max Press, Ivoclar Vivadent) with three different designs of the top surface (flat, single plateau, or double plateau) (n=11) received 'no treatment', '5% HF etching', or "Al_2O_3 sandblasting". HF-etched and sandblasted slabs also received silane and universal one-step adhesive application. All slabs were incrementally repaired with nanofilled composite (Filtek Z350, 3M ESPE) up to 6 mm above the highest ceramic top plateau. Specimens were stored in artificial saliva at 37 °C for 21 days and then subjected to 1,000 thermocycles between 5 and 55 °C. The interface composite-ceramic of each specimen was tensile tested until failure in a universal testing machine and the mode of failure was determined under a stereomicroscope. The ceramic surface morphology of one representative tested specimen from each subgroup (design/surface treatment) was observed through scanning electron microscopy (SEM).* ***Results:*** *Regardless of ceramic design, the absence of surface treatment resulted in significantly lower load-to-failure values. No significant differences in load-to-failure values were observed between HF-etched and sandblasted specimens for the flat design; however, HF etching resulted in significantly higher load-to-failure values than sandblasting for both single plateau and double plateau designs. The majority (60%) of HF-etched specimens with single plateau or double plateau presented mixed failures. SEM photomicrographs showed that HF-etched specimens had smoother surfaces than sandblasted specimens.* ***Conclusion:*** *The surface treatment of a defective lithium disilicate glass-ceramic restoration has more influence than its macroscopic design on the retention of the composite repair. HF etching seems to provide higher bond strength to the composite repair.*

Keywords: *dental restoration repair - composite repair - ceramic repair - hydrofluoric acid - sandblasting*

Efeito de diferentes tratamentos de superfície e formas do preparo de cerâmicas na resistência de união de reparos em resina composta

RESUMO

Embora fraturas e lascamento de restaurações vitrocerâmicas sejam comuns, alguns casos podem ser reparados com compósito direto. ***Objetivo:*** *investigar o efeito da interação de diferentes formas e tratamentos de superfície na carga de ruptura de uma vitrocerâmica reforçada com dissilicato de lítio reparada com compósito nanoparticulado.* ***Materials e Método:*** *A superfície superior de espécimes de vitrocerâmica (IPS e.max Press, Ivoclar Vivadent) foi preparada com três formas (plana, platô único, ou duplo) e recebeu (n=11): 'nenhum tratamento', 'condicionamento com ácido hidrofluórídrico 5%', ou 'jateamento com Al_2O_3'. Ambos espécimes condicionados e jateados receberam silano e adesivo universal. Todos os espécimes foram reparados incrementalmente com compósito (Filtek Z350, 3M ESPE) até 6 mm acima do platô cerâmico mais alto, armazenados em saliva artificial à 37 °C por 21 dias, e submetidos à 1.000 termociclos (5 e 55 °C). A interface compósito-cerâmica de cada amostra foi testada à tração até sua falha em máquina universal e o modo de falha foi determinado com estereomicroscópio. A morfologia da superfície de uma amostra representativa de forma/tratamento de superfície foi observada através de microscopia eletrônica de varredura (MEV).* ***Resultados:*** *Independentemente da forma cerâmica, a ausência de tratamento superficial resultou em valores de carga de ruptura significativamente menores. Não foi observada differença significativa entre os espécimes planos condicionados ou jateados; no entanto, o condicionamento resultou em valores significativamente maiores que o jateamento para espécimes com platô único e duplo. A maioria (60%) dos espécimes condicionados e com platô único ou duplo apresentou falhas mistas. Imagens SEM demonstraram rugosidade superficial mais regular dos espécimes condicionados que os jateados.* ***Conclusões:*** *O tratamento superficial de uma restauração defeituosa de vitrocerâmica reforçada por dissilicato de lítio tem maior influência na retenção do reparo de compósito do que sua forma macroscópica; ainda, o condicionamento com ácido hidrofluórídrico parece proporcionar maior resistência de união ao reparo com compósito.*

Palavras-chave: *reparo de restauração dental; reparo com compósito; reparo cerâmico; ácido hidrofluórídrico; jateamento.*

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Corresponding Author:
Fabiana Mantovani Gomes França
biagomes@yahoo.com

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INTRODUCTION

Glass ceramics are widely used to manufacture esthetic veneers, inlays, onlays, and crowns[1]. However, they are brittle and often become cracked or chipped due to secondary caries, trauma, parafunctional habits, manufacturing flaws, or stress concentration induced by occlusal adjustments[2-5]. Several factors such as cost, time, wear of sound tooth structure, and risk to pulp vitality must be considered before replacing a defective ceramic restoration[6]. Removing restorations luted with adhesive inevitably enlarges the new preparation and weakens the tooth[7,8]. However, certain cases can be repaired with direct composite, which is a minimally invasive, low-cost, less time-consuming procedure[2,5].

The advantages of restorative repair have been routinely included for more than 10 years in most European and North American dental school syllabuses[19-11]. Some longitudinal clinical trials indicate that repaired composite restorations can remain clinically acceptable for up to 12 years[12-15]. Despite the lack of long-term evidence, the repair of ceramic restorations has shown a success rate of 89% and a survival rate of 3 years, which makes the approach feasible in certain cases[16].

Several factors, including ceramic type, composite type, aging condition, and surface treatment protocol can influence the composite repair bond strength to ceramic restorations[6]. The success of adhesion depends on the roughness of the surface to which the composite is bonded[21]. Different protocols for intraoral repair of chipped and/or fractured ceramic restorations have been suggested to increase the bond strength to the composite: roughening by diamond burs[22], etching with hydrofluoric acid (HF)[23,24] sandblasting with aluminum oxide (Al2O3) microparticles[15], laser irradiation, and tribochemical silica coating[25]. HF etching has been widely reported as a reliable extraoral surface treatment for glass-ceramic restorations prior to adhesive luting[26]. However, in an intraoral repair scenario, HF is highly toxic and may cause severe damage to oral tissues, and its use is forbidden in dental clinics in several countries[27]. Although lower bond strength values have been reported, sandblasting with Al2O3 microparticles at adequate pressure does not harm soft tissues nor decrease the flexural strength of lithium disilicate glass-ceramics[28].

Though most studies investigate the effect of microscopic changes promoted by different surface treatments on the ceramic surface to be repaired, it would be also relevant to address the question of whether the macroscopic design of the ceramic restoration could affect adhesive bonding to the composite repair. The aim of this study was therefore to investigate the interaction effect of different designs and surface treatments on the load to failure of lithium disilicate glass-ceramic repaired with nanofilled composite. The null hypothesis was that different macroscopic ceramic designs did not influence the load-to-failure of composite repairs, regardless of different previous surface treatments.

MATERIALS AND METHOD

Ceramic slabs 4 mm thick (10 x 10 mm) with three different designs of the top surface (flat, single 2-mm-deep plateau, or double 2-mm-deep plateau) were prototyped virtually using computer aided-design software, milled in wax using a computer aided-manufacturing unit, invested, and then heat-pressed with lithium disilicate glass-ceramic ingots (IPS e.max Press, Ivoclar Vivadent, Liechtenstein). The injection sprues were removed with diamond burs (#881 and #881F, Jota do Brasil, Florianópolis, SC, Brazil) mounted on a high-speed water-cooled air turbine, and the ceramic slabs were cleaned ultrasonically in distilled water for 30 sec (Fig. 1).

Fig. 1: Ceramic slabs with different macroscopic designs: flat, single plateau, and double plateau.

The ceramic slabs of each design were assigned to subgroups (n=11) according to the following top surface treatments:

- No treatment.
- Etching with 5% HF (Condac Porcelain, FGM, Joinville, SC, Brazil) for 20 sec, followed by water rinsing and air-drying. One layer of silane (Relyx Ceramic Primer, 3M ESPE, Saint Paul MN, USA) was applied and air-dried after 60 sec. Then, one layer of a universal one-step adhesive

(Single Bond Universal, 3M ESPE, Saint Paul, MN, USA) was applied for 20 sec, air-dried for 5 sec, and light-cured for 10 sec using an LED unit with an output of 1000 mW/cm^2 (VALO, Ultradent, South Jordan, UT, USA).

– Sandblasting with 50-µm Al2O3 particles for 10 sec from a distance of 5 mm. Then, both silane and adhesive were applied as described above.

Each slab was placed in a polyvinyl chloride mold and incrementally repaired with nanofilled composite (Filtek Z350, 3M ESPE) up to 6 mm above the highest ceramic top plateau. The molds had marks every 2 mm to guide the thickness of each composite layer (Fig. 2), which was light-cured for 20 sec on each side using the abovementioned LED unit. All specimens were stored in artificial saliva at 37 °C for 21 days (ECB 1.3 bacteriological oven, Odontobrás, Ribeirão Preto, SP, Brazil). Then, they were subjected to 1,000 thermocycles between 5 and 55 °C (30 sec dwell time) and stored in distilled water at 37 °C before testing.

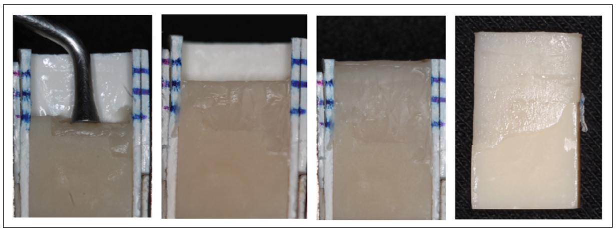

Fig. 2: Ceramic slab placed in a polyvinyl chloride mold and repaired with 2-mm-thick layers of nanofilled composite.

The upper and the lower edges of each specimen were attached to a tensile test setup (Fig. 3) and the interface composite-ceramic was tested until failure in a universal testing machine with a 200-kgf load cell (DL2000, EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 1 mm/min. The load-to-failure of each specimen was recorded in Newtons (N).

The mode of failure was determined under a stereomicroscope with 40x magnification (EK3ST, Eikonal, São Paulo, SP, Brazil) and classified as 'adhesive' (at the interface between ceramic and composite), 'cohesive in composite', 'cohesive in ceramic', or 'mixed' (combination of interfacial failure and cohesive in composite). One representative tested specimen of each subgroup (design/surface treatment) was sputter-coated with gold and the ceramic surface morphology was observed through scanning electron microscopy (SEM; VEGA3, Tescan, Brno, Czech Republic) at magnifications of 100X, 160X, and 500X.

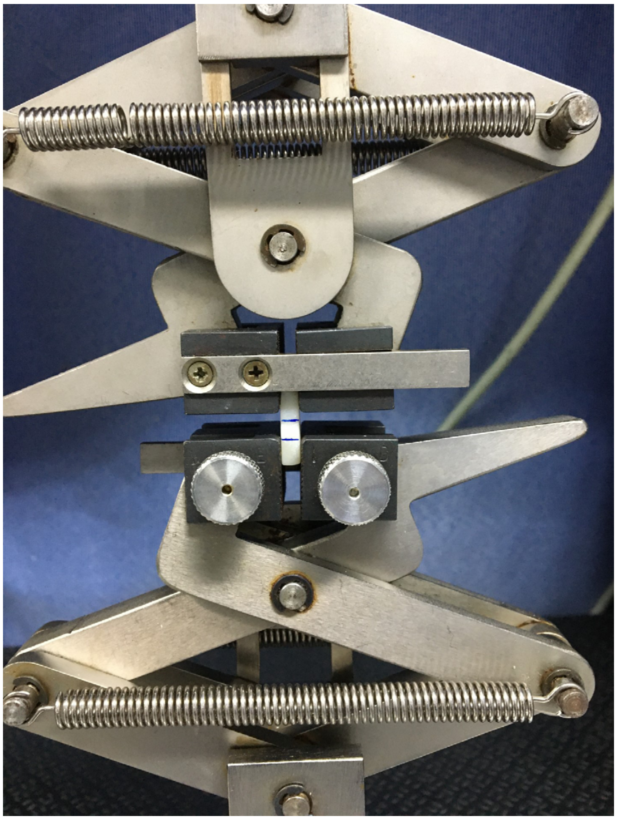

Fig. 3: Composite-repaired ceramic specimen attached to a tensile test setup in a universal testing machine.

Since the data did not meet the assumptions of normality and homoscedasticity, the ceramic design and surface treatment effects were analyzed using the Kruskal-Wallis test followed by Dunn's multiple comparisons test. The failure modes were compared using G-tests. The data were analyzed with statistical software at a significance level of $p<0.05$ (SPSS 23.0, IBM Corp., Chicago, IL, USA; BioEstat 5.0, Mamirauá Institute, Belém, PA, Brazil).

RESULTS

Considering the flat ceramic design, no significant differences in load-to-failure values were observed between HF-etched and sandblasted specimens (Tables 1 and 2). However, HF etching resulted in significantly higher load-to-failure values than sandblasting for both single plateau and double plateau designs (Tables 3 and 4).

Regardless of ceramic design, the absence of surface treatment resulted in significantly lower load-to-failure values between lithium disilicate glass-ceramic and composite repair (Table 1).

Table 1. Mean (± standard deviation) and median load to failure values (N) between lithium disilicate glass-ceramics and composite repair for different ceramic designs and surface treatments.

Ceramic design	No treatment	HF etching	Sandblasting	p-value
Flat	2.19 (±4.62); 0.00^{Ba}	148.62 (±66.06); 144.81^{Aa}	61.29 (±50.96); 33.74^{Aa}	< 0.001
Single plateau	5.49 (±10.99); 0.00^{Ba}	171.02 (±84.40); 208.64^{Aa}	16.84 (±12.58); 16.98^{Bb}	< 0.001
Double plateau	8.90 (±17.78); 0.0^{Ba}	127.59 (±64.10); 116.39^{Aa}	34.34 (±35.17); $19.53B^{ab}$	< 0.001
p-value	0.733	0.550	0.031	—

Values with identical superscript uppercase letters for each ceramic design within the same rows are not significantly different (p>0.05). Values with identical superscript lowercase letters in the same columns for each surface treatment are not significantly different (p>0.05).

Table 2. Dunn's multiple comparison test for flat specimens with different surface treatments.

Comparisons	Rank difference	calculated Z	critical Z	p<0.05
No treatment vs. HF etching	18.0	4.5720	2.394	Yes
No treatment vs. Sandblasting	10.2	2.5908	2.394	Yes
HF vs. Sandblasting	7.8	1.9812	2.394	No

* Rank sum: No treatment 61.0; HF etching 241.0; Sandblasting 163.0.
* Mean rank: No treatment 6.1; HF etching 24.1; Sandblasting 16.3.

Table 3. Dunn's multiple comparison test for single plateau specimens with different surface treatments.

Comparisons	Rank difference	calculated Z	critical Z	p<0.05
No treatment vs. HF etching	17.95	4.5593	2.394	Yes
No treatment vs. Sandblasting	5.90	1.4986	2.394	No
HF vs. Sandblasting	3.0607	2.394	2.394	Yes

* Rank sum: No treatment 75.5; HF etching 255.0; Sandblasting 134.5.
* Mean rank: No treatment 7.55; HF etching 25.5; Sandblasting 13.45.

Table 4. Dunn's multiple comparison test for double plateau specimens with different surface treatments.

Comparisons	Rank difference	calculated Z	critical Z	p<0.05
No treatment vs. HF etching	18.00	4.5720	2.394	Yes
No treatment vs. Sandblasting	8.10	2.0574	2.394	No
HF vs. Sandblasting	9.90	2.5146	2.394	Yes

* Rank sum: No treatment 68.0; HF etching 248.0; Sandblasting 149.0.
* Mean rank: No treatment 6.88; HF etching 24.8; Sandblasting 14.9.

Approximately 80%, 70%, and 60% of the untreated specimens of flat, single plateau, and double plateau ceramic designs, respectively, had pre-testing failures during thermocycling, and their respective load-to-failure values were recorded as zero. Only 20% of sandblasted specimens with flat and single plateau designs presented pre-testing failures. Conversely, HF-etched specimens did not present failures during thermocycling.

The load-to-failure values of HF-etched specimens did not differ significantly according to the ceramic design. For specimens with sandblasted surface treatment, the load-to-failure values of flat specimens were significantly higher than for specimens with a single plateau. The double plateau design resulted in intermediate load-to-failure values, which did not differ significantly from the other two ceramic designs (Table 5).

Table 5. Dunn's multiple comparison test for sandblasted specimens with different ceramic design.

Comparisons	Rank difference	calculated Z	critical Z	p<0.05
Flat vs. Single plateau	10.25	2.6035	2.394	Yes
Flat vs. Double plateau	6.4	1.6256	2.394	No
Single plateau vs. Double plateau	3.85	0.9779	2.394	No

* Rank sum: Flat 210.5; Single plateau 108.0; Double plateau 146.5.
* Mean rank: Flat 21.05; Single plateau 10.8; Double plateau 14.65.

Significant differences were found among failure modes (p<0.001) Regardless of the design, untreated and sandblasted lithium disilicate glass-ceramic specimens, respectively, presented only adhesive failures and cohesive failures in composite. The percentage of cohesive failures in composite was high (80%) for HF-etched flat specimens, while most (60%) of the HF-etched specimens with single plateau or double plateau presented mixed failures. Cohesive failure in ceramic was observed only in HF-etched with single plateau design (10%) (Fig. 4). SEM photomicrographs showed that the surface was smoother in HF-etched specimens than in specimens sandblasted with Al2O3 microparticles (Fig. 5).

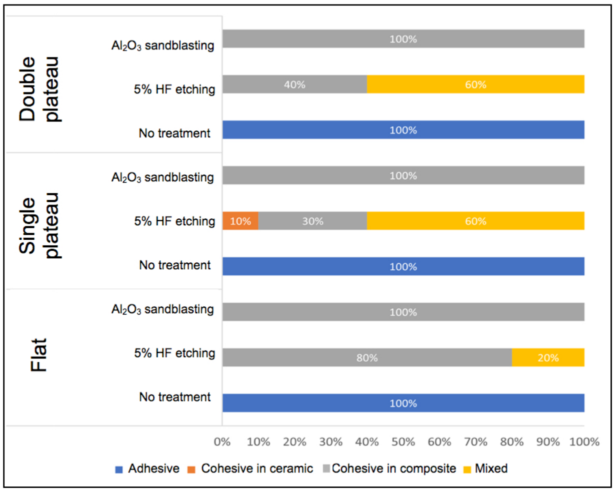

Fig 4: Failure mode percentages of composite-repaired lithium disilicate glass-ceramic specimens after tensile testing for each ceramic design and surface treatment.

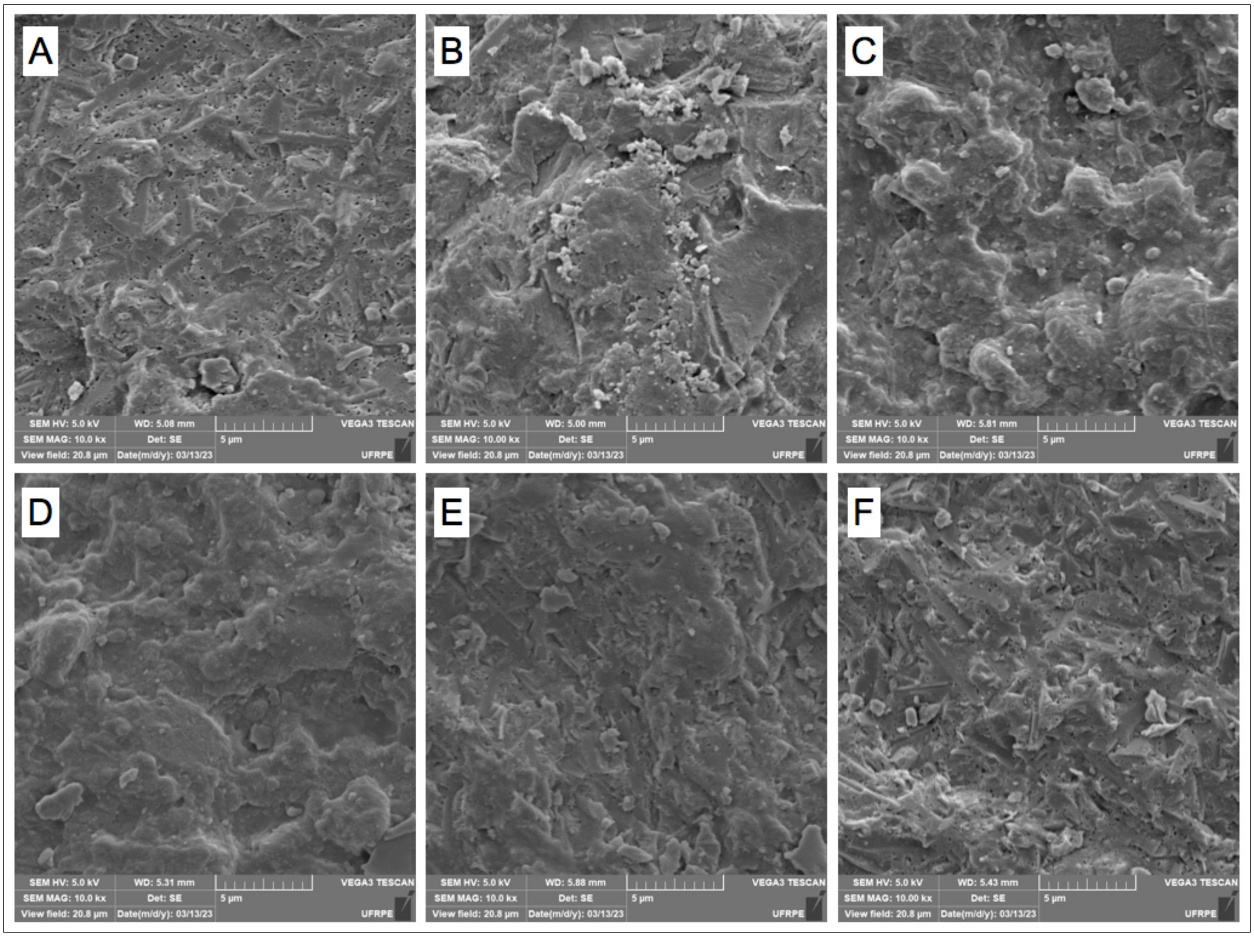

Fig. 5: SEM photomicrographs of tensile tested surfaces: A) Untreated specimen with single plateau design; B) Sandblasted specimen with single plateau design; C) Sandblasted specimen with double plateau design; D) HF-etched specimen with flat design; E) HF-etched specimen with single plateau design and F) HF-etched specimen with double plateau design.

DISCUSSION

Since repairing defective ceramic restorations with direct composite can be a valuable approach due to its reliability, low cost, and conservative characteristics[29], this study addressed the effect of macroscopic design and surface treatment on the load-to-failure of lithium disilicate glass-ceramic repaired with nanofilled composite. The null hypothesis was rejected because there was no significant difference between macroscopic ceramic design and load-to-failure values.

The bonding effectiveness of composite to ceramic depends strongly on micromechanical retention[6], so the lithium disilicate glass-ceramic surface was roughened by HF etching or Al2O3 sandblasting. Regardless of the ceramic design, the highest values of load-to-failure were observed for specimens etched with 5% HF before composite repair. Although the microstructure of lithium disilicate glass-ceramic has high crystal content, HF etching dissolves the glassy matrix of ceramic, creating a superficial porous microretentive surface which increases the surface free energy and wettability for adhesive bonding[30,31]. Both HF concentration and etching time were within the acceptable range that does not jeopardize the bond strength to lithium disilicate glass-ceramic[15,24].

The results also demonstrated that Al2O3 sandblasting created a certain amount of micromechanical retention on lithium disilicate glass-ceramic. Flat ceramic surfaces resulted in significantly higher load-to-failure values than did single plateau design, suggesting that Al2O3 sandblasting is less effective when applied on angulated ceramic walls. Moreover, sandblasting was significantly less effective than HF etching for both single plateau and double plateau ceramic designs; however, the difference was not significant between HF-etched and sandblasted flat ceramic surfaces. Sandblasting increases the surface roughness and surface area of glass-ceramics; however, surface roughness above certain microlevels can form microcracks that reduce micromechanical retention and decrease bond strength; in addition, deep irregular pits on the ceramic surface do not provide retentional features[32].

The application of a silane coupling agent produces a chemical link between the silicate in the ceramic surface and the polymer-based hydrophobic components in the composite through covalent siloxane bonds[4,33]. Thus, the combination of mechanical and chemical retention increases the bond strength of ceramic and repair composite[34]. The results of the current study corroborate some other studies that recommend HF etching followed by silanization as the gold standard surface treatment for silica-based glass-ceramics [4,5,35].

The specimens were stored in artificial saliva for 21 days and thermocycled between 5 and 55 ^{o}C (1,000 cycles) because these techniques are widely accepted to simulate aging of the interface between ceramic and composite repair[6,8]. Regardless of the ceramic design, 80% of the specimens that did not receive surface treatment presented pre-testing failure during thermocycling, which indicates the importance of creating microretention on the ceramic surface before composite repair.

In the HF-etched specimens, the load-to-failure values were higher for single plateau ceramic design than for flat design. Although the double plateau was expected to provide even more retention for the composite repair, it had the lowest load-to-failure values. Since the load-to-failure values of all ceramic designs were relatively high and did not differ significantly, it seems that the failures (mostly cohesive in composite or mixed) occurred due to intrinsic characteristics of the composite. Furthermore, the relatively high standard deviations presented by most of the groups may be related to the macro design of the specimens, in which the occurrence of internal gaps along the ceramic/composite interface as well as stress accumulation, particularly at the internal corners of single and double plateau specimens, may have influenced the overall results[36].

All lithium disilicate glass-ceramic specimens that did not receive previous surface treatment failed at the interface between ceramic and composite, which was not observed in any HF-etched or sandblasted specimen. Regardless of the ceramic design, all sandblasted specimens presented cohesive failure in composite, which suggests that the interfacial bond strength provided by sandblasting is higher than the cohesive strength of the nanofilled composite. Moreover, the higher load to failure values of specimens with flat design in comparison to both single plateau and double plateau indicates that a thick, uniform layer of repair composite when sandblasting is used as ceramic surface treatment.

In contrast to flat specimens, HF-etched specimens with single or double plateau presented the most mixed failures, which suggests better stress distribution throughout the composite and the lithium disilicate glass-ceramic. The optical profilometry analysis conducted by Lima et al. (2021) showed that both sandblasting with 50-µm Al2O3 particles and silica coating with 30-µm Al2O3 particles resulted in the most pronounced alterations on the ceramic surface. The authors reported that although sandblasting created the highest surface roughness, it also promoted surface damage in all evaluated ceramic types. Moreover, 10% HF etching increased flexural strength, particularly when applied for 20 sec.

Strasser et al. (2018) reported that HF provided strong, homogenous etching patterns on lithium disilicate glass-ceramic, in which the glass phase was dissolved and the crystals were found to be relatively protruded. Nevertheless, sandblasting with 50-µm Al2O3 particles resulted in the highest values of surface roughness. Gul & Uygun (2020) reported that sandblasting caused the most remarkable alterations on ceramic surfaces. In the current study, the SEM images of sandblasted specimens also showed deeper, more irregular roughness than HF-etched specimens. In addition, the Al2O3 microparticles potentially abraded the lithium disilicate glass matrix and crystals to a certain level that weakened the surface.

The results of this study therefore indicated that the surface treatment of a defective lithium disilicate glass-ceramic restoration has more influence on the retention of the composite repair than its macroscopic design. Moreover, the highest load-to-failure values, the failure mode pattern, and the regular surface roughness observed for HF-etched lithium disilicate glass-ceramic specimens suggest higher bond strength to the composite repair.

CONFLICT INTERESTS

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article.

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