

# Surface Treatments for Repair of Feldspathic, Leucite- and Lithium Disilicate-Reinforced Glass Ceramics Using Composite Resin

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The aim of this study was to evaluate the efficacy of different surface conditioning methods on the microtensile bond strength of a restorative composite repair in three types of dental ceramics: lithium disilicate-reinforced, leucite-reinforced and feldspathic. Twelve blocks were sintered for each type of ceramic (n=3) and stored for 3 months in distilled water at 37 °C. The bonding surface of ceramics was abraded with 600-grit SiC paper. Surface treatments for each ceramic were: GC (control) - none; GDB - diamond bur #30 µm; GHF - hydrofluoric acid (10%); GT- tribochemical silica coating (45-µm size particles). Treatments were followed by cleaning with phosphoric acid 37% for 20 s + silane + adhesive. The composite resin was used as restorative material. After repair, samples were subjected to thermocycled ageing (10,000 cycles between 5 °C and 55 °C for 30 s). Thereafter, the samples were sectioned into 1.0 mm<sup>2</sup> sticks and tested for microtensile bond strength with 0.5 mm/min crosshead speed. Data were compared by two-way ANOVA and Tukey's test ( $\alpha=0.05$ ). The superficial wear with diamond bur proved to be suitable for feldspathic porcelain and for leucite-reinforced glass ceramic while hydrofluoric acid-etching is indicated for repairs in lithium disilicate-reinforced ceramic; tribochemical silica coating is applicable to leucite-reinforced ceramic. Predominance of adhesive failures was observed (>85% in all groups). In conclusion, the success of surface treatments depends on the type of ceramic to be repaired.

Key Words: dental ceramics, repairs, surface treatments.

## Introduction

Ceramic restorations have been employed for their innumerable advantages such as color stability, low thermal conductivity, resistance to wear and biocompatibility. However, ceramics without metallic support are prone to crack propagation (1). The feldspathic ceramics used as veneer are commonly affected by shipping, fracture or excessive wear, mainly when supported by zirconia frameworks (2). In this way, direct repair with a composite resin appears as an attractive alternative due to the low cost, fast resolution, and preservation of supporting structures (3-5).

Glass-reinforced ceramics have been also emphasized. Actually, the most popular are the leucite- and the lithium disilicate-reinforced ones. Both may be used to obtain veneers, inlays, onlays and crowns. Monolithic application of these ceramics for crowns has been employed to provide higher resistance than the bi-layer restorations (6), although fractures and wear may also occur in this type of restoration.

Surface preparation in fractured ceramic must be performed in the repair procedure that involves mechanical or chemical surface preparations to create irregularities on the surface. Bonding components are also required for the

adhesion to restorative material. Traditionally, surface treatment for ceramics involves roughening with diamond burs (7-9), etching with hydrofluoric acid (8,10-12) or tribochemical process based on silica-coated aluminum-oxide particles (11-17). For restorative material bonding, use of silane is recommended for glasses and porcelains in order to obtain a mesh of siloxane with the silica on the ceramic surface, to improve the bond strength between the ceramic and luting material and to increase the surface energy for adhesive application (18).

The aim of this study was to evaluate the microtensile bond strength after composite resin repairs in glass-ceramics after different surface treatments. The null hypothesis of the study was that there is no difference among the tested techniques.

## Material and Methods

The following ceramics were used in this study: feldspathic ceramic (Vita VM7, Vita Zahnfabrik, Bad Säckingen, Germany), leucite-reinforced glass-ceramic (IPS Empress, Ivoclar Vivadent, Schaan, Liechtenstein), lithium disilicate-reinforced glass ceramic (IPS E.max Press, Ivoclar Vivadent). Twelve blocks (10.0 x 7.0 x 3.0 mm) were obtained

for each ceramic and aged in distilled water for 3 months at 37 °C. The bonding surface of all ceramic blocks was abraded for 15 s with 600-grit silicon carbide paper and cleaned using ultrasound for 10 min.

The blocks were randomly divided (n=3) in four groups according surface treatments, as follows: GC (control group) - no surface treatment; GDB: surface wear by 30- $\mu$ m-grit diamond bur during 20 s under water cooling; GHF: hydrofluoric acid (10%) during 90 s for feldspathic ceramic, 60 s for leucite-reinforced glass-ceramic, and 20 s for lithium disilicate-reinforced glass ceramic; GT: tribochemical process: sandblasting with silica-coated aluminum oxide (45- $\mu$ m size particles) for 20 s, at a distance of 10 mm, perpendicular to the adhesion surface, under 2.8 bar pressure.

In all ceramic blocks, the surface treated by the different protocols was cleaned with 37% phosphoric acid (Condac 37, FGM Dental Products, Joinville, SC, Brazil) for 30 s, followed by silane application (Angelus Dental Products, Londrina, PR, Brazil) for 1 min. An adhesive system was used as bonding agent (Adapter Singlebond 2; 3M/ESPE, St. Paul, MN, USA) and light-cured for 20 s using an irradiance of 800 mW/cm (BluePhase LED; Ivoclar Vivadent). Composite resin (Filtek Z350; 3M/ESPE) was used as restorative material by the incremental technique and light-cured for 30 s as previously described until obtaining a 3-mm thickness. A single operator performed the complete repair process.

Samples were submitted to thermocycling process (10,000 cycles, between 5 °C and 55 °C for 30 s in each bath). After the ageing procedure, the specimens were sectioned in serial slabs (1 mm thick) using a diamond-embedded blade under continuous water irrigation (Buehler, Lake Bluff, IL, USA) and subsequently in 1 mm<sup>2</sup> match-sticks and subjected to the microtensile bond strength ( $\mu$ TBS) evaluation in a universal testing machine (EZ-test, Shimadzu Co., Kyoto, Kansai, Japan) at a crosshead speed of 0.5 mm/min. Data were obtained in MPa. The failure pattern was evaluated by a stereomicroscope (Leica Microsystems, Wetzlar, Germany) at 60x magnification and failures were

Table 1. Mean values (MPa) and standard deviations for the different groups in each ceramic

Groups	Feldspathic	Leucite-reinforced	Lithium disilicate-reinforced
GC	9.0 (10.0) B ab	17.9 (3.4) A a	4.6 (1.3) B b
GDB	13.9 (1.99) B a	22.4 (4.4) A a	4.4 (2.6) C b
GHF	14.4 (4.44) B a	3.7 (0.4) C b	21.1 (5.2) A a
GT	0 (0) C b	21.7 (2.5) A a	8.3 (5.5) B b

Different uppercase letters indicate in rows and lowercase letters in columns indicate statistically significant difference ( $p=0.05$ ).

classified as adhesive, mixed or cohesive.

Data were processed using SPSS software (version 20; IBM, Armonk, NY, USA) by two-way ANOVA. Post-hoc tests were calculated using the Tukey's test. All tests were conducted at 95% confidence interval ( $\alpha=0.05$ ).

## Results

Mean values (MPa) and standard deviation for all groups are presented in Table 1. Statistical analysis revealed that for feldspathic ceramic no experimental treatment increased  $\mu$ TBS in comparison with GC. However, the group subjected to tribochemical process had significantly lower results than the other groups ( $p<0.001$ ), presenting pre-test failures in all sticks. For the leucite-reinforced glass ceramic (IPS Empress), GDB and GT did not differ from GC, while GHF showed lower  $\mu$ TBS values ( $p<0.001$ ). For the lithium disilicate-reinforced glass ceramic (IPS E.max press), GHF showed significantly higher  $\mu$ TBS values than GC, GDB and GT ( $p<0.001$ ), which were similar among them. Interaction between the factors (ceramic type and surface treatment) was statistically significant ( $p<0.001$ ).

Failure patterns are presented in Table 2. Predominance of adhesive failures (>85% in all groups) was observed. Mixed failures presented a small proportion (<8% in all groups) and cohesive failures did not exceed 5.2% in all groups.

## Discussion

This study showed that the surface treatment influenced the  $\mu$ TBS of all tested ceramics. Therefore, the null hypothesis

Table 2. Failure patterns (%) for the different surface treatments and ceramics features

Group	Ceramic	Failure mode (%)		
		Adhesive	Mixed	Cohesive
GC	Feldspathic	95.3	4.7	0
GC	Leucite	94.8	5.2	0
GC	Disilicate	93.3	6.7	0
GDB	Feldspathic	89.8	5.8	4.4
GDB	Leucite	91.3	7.6	1.1
GDB	Disilicate	93.9	6.1	0
GHF	Feldspathic	87.7	7.1	5.2
GHF	Leucite	91.8	6.0	2.2
GHF	Disilicate	96.5	3.5	0
GT	Feldspathic	100.0	0	0
GT	Leucite	95.5	3.1	1.4
GT	Disilicate	96.4	2.4	1.2

was rejected. Additionally, it was observed that the success of surface treatment depends on the ceramic type. For the lithium disilicate-reinforced glass ceramic, the etching with hydrofluoric acid promoted the highest  $\mu$ TBS values, which is in agreement with Colares et al. (10). This ceramic has the lowest vitreous proportion among the ceramics tested in this study. Therefore, it seems that the chemical conditioning is most efficient to infiltrate and remove the vitreous phase, creating irregularities in the surface. For leucite-reinforced glass ceramic, the etching with hydrofluoric acid was the only tested surface treatment that showed lower  $\mu$ TBS compared with control and the other tested groups. Since this ceramic has a higher vitreous proportion in comparison to lithium disilicate, it was suggested that mechanical treatments such as the use of diamond burs are more efficient to create irregularities. The success of tribochemical process for leucite-reinforced glass ceramic, which involves the creation of irregularities by sandblasting with aluminum oxide silica-coated particles and chemical improvement by silica deposition on the ceramic surface, is also in agreement with previous reports (14).

For the feldspathic ceramic, the tribochemical process was the only tested surface treatment that promoted lower values of  $\mu$ TBS in comparison with other tested groups. In fact, this type of ceramic showed pre-testing failures during the tribochemical process for all samples during the stick preparation, like weak bond strength, which lead to failures previous to the testing method, even during stick preparation or handling them, characterizing a bond strength equal to zero in the results section. This process is characterized by silica deposition on the ceramic surface, used mainly for bonding on crystalline ceramics as zirconia and alumina. Therefore, it may be suggested that silica deposition on feldspathic ceramic has a long-term low stability. Nevertheless, the present results are different from those of Attia (7) who found that the tribochemical treatment showed similar values of  $\mu$ TBS in relation to diamond bur preparation and also diverge from the study of Melo et al. (11) where tribochemical process was similar to etching with hydrofluoric acid. However, it is important to highlight that those studies did not use the ageing process for the bonded interface, as the thermocycling used in this study, which may have caused the interface degradation. As regards the success of other surface treatments (groups GHF and GDB) used in this study to repair feldspathic ceramics, they are in agreement with other studies (7,8,11).

These observations for efficacy of mechanical or chemical methods for conditioning depending on the ceramic's type are also supported by the data of control group for all ceramics. The silicone grit paper used in all samples to remove the superficial ceramic layer simulating the repair and also used to standardize the surface

roughness probably acts as a mechanical preparation providing roughness on the ceramic surface. In this way, the results of this study showed  $\mu$ TBS values in GC of feldspathic and leucite-reinforced ceramic comparable to those of other mechanical surface treatments. However, for lithium disilicate ceramic the GC was similar to other mechanical treatments, which were significantly lower than HF etching.

The failure patterns observed in this study showed a great prevalence of adhesive failures for all groups, ranging from 85 to 100%. Based on these results, it was not possible to establish a relation of failure pattern with  $\mu$ TBS for the used surface treatments. Other analyses showed that adhesive failures are the most observed for repaired ceramic interfaces (16), some studies presenting 100% prevalence (12,14). This information may suggest that bond strength of repairs on ceramic surfaces are not comparable to the bulk strength of the materials, thus its clinical prognostic success may be related to application in areas of low occlusal load.

Ceramic fractures may result from several factors, such as inadequate occlusal adjustment, failure in the bonding interface, internal porosities, parafunctional habits, and internal stress from the manufacturing process (1). The repair is characterized by a faster and low-cost method if compared to replacement of the entire restoration (3-5). This study has shown that using the correct surface treatment for each ceramic is the key for success in repair procedure. The methods here employed are traditional treatments used in dental clinic. Roughness with diamond bur might be considered the most practical among them because it does not require any additional precaution or protection of the patient than those traditionally used and does not need the use of other apparatus than the traditional burs. Etching with hydrofluoric acid needs complete rubber dam isolation of the teeth to be applied on, as it is irritating to oral tissues. The tribochemical process also needs rubber dam isolation for protecting the mouth from the sandblasted silica-coated aluminum oxide particles.

Within the limitations of this study, considering the evaluated materials and techniques, it may be concluded that diamond burs can be used as surface treatment for repairs in feldspathic and leucite-reinforced ceramics; hydrofluoric acid etching is indicated for repair of lithium disilicate-reinforced ceramic and tribochemical process in successfully used for repairs of leucite-reinforced ceramic. The success of surface treatment depends on the type of ceramic to which it is applied.

## Resumo

O objetivo deste estudo foi avaliar a eficácia de diferentes

condicionamentos de superfície na resistência de união de reparos de compósitos restauradores em três tipos de cerâmicas odontológicas: reforçada por dissilicato de lítio, reforçada por leucita e feldspática. Foram confeccionados 12 blocos para cada tipo de cerâmica (n=3) e armazenados por 3 meses em água destilada a 37 °C. A superfície de união das cerâmicas foi regularizada com lixa de granulação 600 por 15 s e lavadas em ultrassom por 10 min. Os tratamentos de superfície para cada cerâmica foram: GC (controle) - nenhum; GPD - ponta diamantada com 30 µm de granulação; GAF - ácido hidrófluorídrico a 10%; GJ - jateamento com partículas de óxido de alumínio revestido por sílica (45 µm - tamanho das partículas). Após, foi realizada a limpeza da superfície com ácido fosfórico a 7% por 20 s, seguido de silano e adesivo. Como material restaurador foi utilizada resina composta. Após o reparo, as amostras foram submetidas a ciclagem térmica (10,000 ciclos entre 5 °C e 55 °C, por 30 s). Na sequência, as amostras foram seccionadas em palitos de aproximadamente 1,0 mm<sup>2</sup> e levadas ao teste de tração em uma máquina de ensaios universal à velocidade de 0,5 mm/min. Os dados obtidos foram comparados estatisticamente por ANOVA de dois fatores e teste de Tukey ( $\alpha=0,05$ ). Sugere-se que o desgaste da superfície com ponta diamantada é mais indicado para a cerâmica feldspática e cerâmica reforçada por leucita, enquanto o condicionamento com ácido fluorídrico é indicado para reparos em cerâmica reforçada por dissilicato de lítio. O jateamento com partículas de óxido de alumínio revestido por sílica mostrou-se aplicável à cerâmica reforçada por leucita. Predominância de fraturas adesivas acima de 85% foi observada para todos os grupos. Este estudo demonstrou que o sucesso dos tratamentos de superfície depende do tipo de cerâmica a que são aplicados.

## Acknowledgements

The authors would like to thank the Meridional Laboratory, Passo Fundo, RS, Brazil, for the valuable assistance with the sintering process of ceramics.

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Received February 12, 2014

Accepted October 28, 2014