

EFFECTS OF SURFACE TREATMENT ON THE MICROTENSILE BOND STRENGTH OF CERAMIC MATERIALS TO DENTIN

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ABSTRACT

This study evaluated the effects of distinct surface treatments on the micro-tensile bonding strength (μ TBS) of different ceramic materials. The occlusal surfaces of eighteen human maxillary molars were flattened perpendicularly to the long axis and divided in groups based on surface treatment (sandblasting: s; hydrofluoric acid: a; tribochemical silica coating: t): DP-s, DP-a, DP-t, IE-s, IE-a, IE-t, IC-s, IC-a, IC-t) and ceramic materials (Duceran Plus®: DP, IPS Empress 2®: IE, In-Ceram Alumina®, IC). Panavia F® luting resins were used according to the manufacturers' instructions to bond ceramic materials to the exposed dentin specimens under a load of 7.5 N. After 3-day

storage, μ TBS was tested at a cross-head speed of 1 mm/min. Data were analyzed with ANOVA and Tukey's test. ANOVA results showed that the μ TBS of DP and IC were significantly different. The μ TBS of DP-a was significantly higher than those of DP-s and DP-t. The μ TBS of IC-t was significantly higher than those of IC-s and IC-a. Ceramic materials with different chemical formulations and applications yielded significantly different bond strengths to human dentin and must receive distinct surface treatments accordingly.

Key words: surface treatment, bond strength, ceramic materials, dentin.

EFEITO DO TRATAMENTO DE SUPERFÍCIE DE MATERIAIS CERÂMICOS NA RESISTÊNCIA DE UNIÃO A MICROTRAÇÃO À DENTINA.

RESUMO

Este estudo avaliou os efeitos de diferentes tratamentos de superfície de diferentes materiais cerâmicos na resistência de união a microtração (μ TBS). A superfície oclusal de dezoito molares humanos foram cortados perpendicular ao eixo longitudinal e dividido em grupos baseado no tratamento de superfície (jateamento: s; ácido fluorídrico: a; recobrimento triboquímico com sílica: t): DP-s, DP-a, DP-t, IE-s, IE-a, IE-t, IC-s, IC-a, IC-t) e material cerâmico (Duceran Plus®: DP, IPS Empress 2®: IE, In-Ceram Alumina®, IC). Cimento resinoso Panavia F® foi empregado de acordo com as instruções do fabricante, para unir material cerâmico à dentina exposta sob carga de 7.5 N. Depois de armazenado

por 3 dias, μ TBS foi testado com uma velocidade de carregamento de 1,0 mm/min. Dados foram analisados com ANOVA e teste de Tukey. Os resultados de ANOVA mostraram que a μ TBS para DP e IC foram significativamente diferentes. μ TBS de DP-a foi significativamente maior que aqueles de DP-s e DP-t. μ TBS de IC-t foi significativamente maior que aqueles de IC-s e IC-a. Materiais cerâmicos com diferentes composições e processamento apresentaram diferença significativa na resistência de união a dentina humana e deve receber tratamentos de superfície distintos.

Palavras chave: tratamento de superfície, resistência de união, materiais cerâmicos, dentina

INTRODUCTION

The use of ceramic restorations has increased substantially, probably because of their improved biocompatibility and esthetic properties compared with those of other materials (1-4). Cementation is vital

for the clinical success of all-ceramic restorations and it depends on many factors such as ceramic material composition, surface treatment, luting agent (5, 6). As to bond strength, it must be analyzed as a function of the two interfaces formed:

between the ceramics and the luting agent and between the luting agent and the enamel and/or dentin (3, 7, 8). An increasing number of surface treatment methods have been introduced to bond ceramic materials and adhesive resins reliably (2, 4). Currently, sandblasting and silane application are one of the most used and less costly treatments. Other treatments use hydrofluoric acid and tribochemical silica coating (4, 5, 9-12).

Bond strength measurement in laboratory is one of the most effective methods of material characterization; however, it tends to exhibit cohesive fracture. The microtensile method introduced by Sano et al. (13) has become popular for testing adhesion to dentin because this technique presumably provides better stress distribution at the adhesive interface with fewer defects than in standard tensile tests due to the small bonding area. Also, this technique may be used to detect regional difference in resin-dentin bond strength due to the small bond areas used.

The aim of this study was to evaluate the effects of different surface treatments (sandblasting – s; hydrofluoric acid – a; tribochemical silica coating – t) on the μ TBS of different ceramic materials (DP - Duceran Plus® - DENTSPLY International, USA; IE - IPS Empress 2® - Ivoclar Vivadent AG, Liechtenstein; IC - In-Ceram Alumina® - VITA Zahnfabrik, Germany).

MATERIALS AND METHODS

Tooth preparation

Eighteen extracted third molars were collected. The teeth, which had to be free of caries and previous restorations, were stored in 0.2% timol solution 4°C for no longer than 4 weeks. Dentin was prepared by cutting occlusal enamel and dentin perpendicular to the tooth axis 1 mm below the dentino-enamel junction using a slow-speed diamond-coated disk saw (Isomet 1000®, Buehler, USA). Dentin specimens were wet polished with 600 grit SiC paper to produce a standard smear layer and stored in distilled water at 4°C (13-16).

Ceramic material preparation

The teeth were randomly divided to generate into three main groups based on ceramic material (n = 6 each) as follows: Duceran Plus® - DP; IPS Empress 2® – IE; and In-Ceram Alumina® – IC that was to be bonded to them. Impressions of all tooth preparations were made with polyvinyl-siloxane impres-

sion material and poured into a vacuum-mixed die material. Each of the 3 main groups was divided into 3 sub-groups based on surface treatment (n = 2 each). Sandblasting – s: was performed with 50-mm alumina particles. The tip of the micro etcher was kept 1 mm away from the specimen surface and applied for 3 s. Hydrofluoric acid 10% - a: etching with 10% hydrofluoric acid for 5 minutes. Tribochemical silica coating – t: micro etching was performed with 100-mm alumina particles (Rocatec pre® - 3M ESPE AG, Germany) and 100-mm alumina particles modified with silica (Rocatec plus® - 3M ESPE AG, Germany).

After surface treatment, all specimens were rinsed under running tap water to remove debris and a silane solution (Dentsply, Brasil) was applied onto the ceramics specimens for 5 minutes. Luting resin Panavia F® - Kuraray Medical Inc, Japan was mixed according to the manufacturer's directions and a weight of 7.5 N was applied. Excess resin was removed from the bonding margin with cotton pellets and an oxygen-blocking gel was applied. The samples were stored in saline solution at 37 °C for 24 hours. The specimens were cut in "sticks" approximately 12 mm high with a rectangular cross-sectional area of 1.44 mm² by using an Isomet 1000® machine. Twenty "sticks" were selected from each group and were stored in saline solution at 37°C from 8 to 12 hours (6).

The cross-sectional area of each specimen was measured for calculation of bond strength after fracture. μ TBS was performed in a universal testing machine MTS® - Material Test System, USA at a cross-head speed of 0.5 mm min⁻¹.

Analysis of variance (ANOVA) and Tukey's test were performed with bond strength as the dependent variable. Ceramic materials and surface treatments were treated as between-subject factors. Samples of each group were examined in a scanning electron microscope to establish the characteristics of the fracture region surfaces.

RESULTS

The means and standard deviations of all microtensile bond strength tests are compiled in Table 1. As seen in Table 2, two-way analysis of variance indicated that μ TBS was significantly affected by surface treatment and ceramic system factors and significant interaction between factors. Tukey's test indicated significantly different bond strengths for

TABLE 1. Means and standard deviations of microtensile bond strength (MPa) of each group.

GROUP	Treatment		
	Sandblasting (s)	Hydrofluoric acid (a)	Tribochemical Silica Coating (t)
Duceram Plus (DP)	4.54 (2.23)	18.71 (6.33) ^a	9.99 (3.29) ^b
IPS Empress 2 (IE)	14.03 (5.52) ^c	16.05 (4.32) ^{acd}	16.76 (5.20) ^{acd}
In Ceram (IC)	9.03 (3.03) ^{be}	8.53 (4.21) ^{be}	12.03 (3.71) ^{bc}

Groups identified with the same superscript letter are not significantly different ($p > 0.05$).

TABLE 2. Analysis of variance of microtensile bond strength results.

Source	df	Sum of Squares	Mean Squares	F value	P value
Surface treatment	2	869.8775	434.9388	22.67	< 0.001
Ceramics	2	1101.7087	550.8544	28.72	< 0.001
Surface treatment X Ceramics	4	1398.1642	349.5410	18.22	< 0.001

feldspatic ceramics Duceram Plus[®] for distinct surface treatments. The bond strength of DP-a (18.71 MPa) was significantly higher than those of DP-s and DP-t. For DP-t (9.99 MPa), the bond strength was significantly higher than that of DP-s (4.54 MPa). For ceramics IPS Empress 2[®], bond strength was significantly similar for distinct surface treatments. As to In-Ceram Alumina[®], bond strength was significantly different for distinct surface treatments. Group IC-t (12.03 MPa) showed bond strength significantly higher than those of the other groups (IC-s and IC-a).

DISCUSSION

Feldspatic ceramics Duceram Plus[®] was used as a control because its bond process to resin cements is established. The results showed significant difference in bond strength for surface treatments (Table 1 and 2). The bond strength obtained with hydrofluoric acid etching was significantly higher than those of other treatments. This result is in agreement with that of Stewart et al. (3), who studied the influence of different surface treatments on shear bond strength to dentin. They observed significantly high bond strength for hydrofluoric acid etching and silane. Using shear test, Shahverdi et al. (17), also found large bond strength values for hydrofluoric acid in comparison to those for sandblasting. However, Jedyakiewicz & Martin (18) did not observe difference in the bond strength of feldspatic ceramic blocks treated with either hydrofluoric acid

or tribochemical silica coating. Borges et al. (5) evaluated morphologic alterations on the surface of feldspatic ceramics treated with hydrofluoric acid and evidenced that this conditioning results in surface alterations, creating a beehive-like topography. This alteration may be explained by the chemical reaction of hydrofluoric acid with the silica of feldspatic ceramics to form hexafluorosilicates. These silicates are removed by rinsing, resulting in an ideal surface for micromechanical retention. IPS Empress 2[®] ceramics results did not show any significant difference in bond strength for the distinct surface treatments (Table 1). The relative increase in bond strength values for the specimens may be explained by the passivity to the surface treatments presented by this ceramic system, which may be verified in the findings of Borges et al. (5), whose sandblasting with aluminum oxide modified the surface of IPS Empress 2[®] by increasing the number of surface depressions. Elongated crystals and flat irregularities were clearly observed when the surface of IPS Empress 2[®] ceramics was treated with hydrofluoric acid.

For the In-Ceram Alumina[®] ceramic system, bond strength presented a significant difference for the different surface treatments, the highest values being associated with tribochemical silica coating process, which was significantly higher than those of the other tested conditions (Table 1). Considering shear strength, Ozcan et al. (8) evidenced that silica coating of restoration surface of In-Ceram

cemented with Panavia F® provided the largest bond strength values (21.54 MPa), followed by sandblasting (12.9 MPa) and hydrofluoric acid conditioning (5.5 MPa). Ozcan et al. (8) also pointed out that the effect of hydrofluoric acid conditioning and sandblasting on bond strength of In-Ceram Alumina® and opposite to the results of feldspatic ceramics. Low bond strength values associated to the conditioning can be associated to the fact that hydrofluoric acid does not to create micro-retention in the surface of the In-Ceram due to the high alumina content. The findings of Madani et al. (12) had confirmed the results in this study; bond strength to In-Ceram is higher when sandblasting than when conditioning with 9.5% and 5.0% hydrofluoric acid solutions. Our results corroborate those by Kern & Strub (10), who found that hydrofluoric acid conditioning associated with silane application increases the bond strength of ceramics with high silica content such as feldspatic ceramics, and decreases it for ceramics with high alumina content. However, silica coating such ceramics increases the surface silica content (Kern & Thompson (10), by forming a silica layer for interaction with the silane agent, which increases strength. Effective bond by silica coating In-Ceram® ceramics was verified in a clinical study carried out by Kern & Strub (10) in which the success of restorations was 94.1% after 5 years. Borges et al. (5) found that sandblasting In-Ceram Alumina® and In-Ceram Zirconia® systems with aluminum oxide generated flatter surface irregularities than those of other control ceramic surfaces, which may be due to the high alumina content of these ceramics and to glass infiltrated in the structure. The aluminum oxide crystals used in abrasion have similar hardness to that of aluminum oxide

crystals in the ceramics structure. The authors also reported that sandblasting Procera® ceramics with high alumina content planned off alumina crystals. Considering that hydrofluoric acid etching of ceramics In-Ceram Alumina® and In-Ceram Zirconia®, whose alumina content represents 85% and 67% weight, respectively, no alteration was evidenced. Both structures are infiltrated by lantana-aluminum-silicate glass containing less than 5% weight silica. As the silica phase is the only one that may be conditioned by hydrofluoric acid, the conditioning process was inefficient (Borges et al.) (5). Laboratory research has shown that aluminum oxide sandblasting and/or hydrofluoric acid conditioning followed by silane application increase the bond strength of ceramics that present silica, not provide lasting resistance to either aluminized or glass infiltrated ceramics. However, silica surface coating of aluminized ceramics is a possible surface treatment as it induces the formation of a surface silica layer onto which the silane agent may bond. (Kern & Thompson) (10), Jedynekiewicz & Martin (18) studied the influence of surface treatments on the bond strength of ceramic glass and evidenced that it may be increased by the application of a silicon oxide layer in detriment of acid conditioning.

In conclusion, μ TBS is influenced by the interaction between surface treatment and material composition. The bond strength of feldspatic ceramic Duceram Plus® is significantly high for hydrofluoric acid etching, while that of ceramic IPS Empress 2® is not significantly different for distinct surface treatments. The bond strength of In-Ceram Alumina® is significantly high for tribochemical silica coating process.

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