Noéli Boscato

CARACTERIZAÇÃO CERÂMICA E AVALIAÇÃO FRACTOGRÁFICA DA INTERFACE ADESIVA COM RESINA, APÓS DIFERENTES TRATAMENTOS DE SUPERFÍCIE

Tese de Doutorado apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas para obtenção do Título de Doutor em Clínica Odontológica – Área de Prótese Dental

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Dedico este trabalho....

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SUMÁRIO

RESUMO

Este estudo avaliou o efeito do tratamento de superfície na resistência adesiva à tração (σ) entre resina e as cerâmicas IPS Empress®(E1) e VITAVM7®(V7) e o modo de falha nessa interface adesiva. A metodologia proposta teve por finalidade testar a hipótese de que a σ entre resina e cerâmica é controlada pelo tratamento de superfície das cerâmicas. Foram confeccionados 10 blocos de uma cerâmica a base de leucita, (E1) e de uma cerâmica feldspática com duas fases vítreas (V7), que foram polidos até a granulação de 1 µm. Os blocos de cada cerâmica foram divididos aleatoriamente em dois grupos e tiveram suas superfícies tratadas como segue (n=5): Grupos E1HF e V7HF: aplicação de ácido hidrofluorídrico a 9,5% (HF - Ultradent) aplicado por 60 s; Grupos E1CS e V7CS: jateamento com partículas de alumina modificadas por sílica (CS – Cojet System, 3M-Espe), aplicado por 15 s. As superfícies cerâmicas tratadas foram lavadas, secadas e o silano foi aplicado deixando-o evaporar. Aplicaram-se duas camadas finas de adesivo (Single Bond, 3M), seguido da aplicação de camadas de 2 mm de resina composta (Z250, 3M), que foram fotopolimerizadas durante 40 s cada uma. Os blocos cerâmica-adesivo-resina composta foram seccionados em dois eixos, x e y, obtendo-se corpos-de-prova em forma de barras (n=30), com área adesiva média de 1,04 mm^2. Os corpos-de-prova foram armazenados em água destilada a 37°C por uma semana antes do teste de tração em uma máquina de ensaios universal com velocidade de carga de 1.0 $mm.min^{-1}$, seguido da análise microscópica da superfície fraturada. A análise estatística foi realizada pela análise de variância, teste de Tukey (α=.01) e análise de Weibull. As médias e desvio padrão da σ (MPa) foram: E1HF: 29,8±4,5(a); E1CS: 24,6±5,6(b); V7HF: 22,3±4,0(b); V7CS: 15,7±6,9(c). Os valores médios de σ do Grupo E1HF foram significativamente maiores que os valores médios dos demais grupos (p=0,0001). As duas cerâmicas apresentaram valores médios de σ significativamente maiores quando tratadas com HF do que com CS (p=0,0001). Todas as fraturas ocorreram dentro da zona adesiva. O módulo de Weibull *(m)* foi mais alto para o Grupo E1HF (7,66), e o Grupo V7CS mostrou o valor mais baixo de *m* (2,54). Os resultados confirmam a hipótese inicial de que a σ da resina à cerâmica é controlada, primariamente, pelo tratamento de superfície do material cerâmico.

Palavras – chave: cerâmica – fractografia – tratamento de superfície

ABSTRACT

This study evaluated the effect of ceramic surface treatments on tensile bond strength (σ) and the mode of failure of a resin bonded to two types of ceramics, testing the hypothesis that σ of ceramics to resin is controlled by the ceramic surface treatment. **Methods:** Ten blocks of each the hot-pressed leucite-based ceramic (E1- IPS Empress) and the two-phase glassy feldspathic ceramic (V7-VITAVM7) were fabricated, polished through 1 μm alumina abrasive, and divided into two groups per ceramic (n=5): Groups E1HF and V7HF, 9.5% hydrofluoric acid (HF) was applied for 60 s; Groups E1CS e V7CS, silica coating (CS) using Cojet System (3M-Espe) for 15 s. The treated ceramic surfaces were washed and dried. Silane was applied and let to evaporate. An adhesive resin (Single Bond, 3M) followed by a resin composite (Z250, 3M) were applied on the ceramic treated surfaces and light cured. The composite-ceramic blocks were cut to produce bar-shaped specimens with a mean bonding area of 1.04 mm^2 (n=30). Specimens were stored in 37°C distilled water for 1 week before tensile loading to failure in a universal testing machine with cross-head speed of 1.0 $mm.min^{-1}$. Fracture surfaces were examined under scanning electron microscope (SEM). Results were statistically analyzed using one way ANOVA, Tukey's test and Weibull analyses. **Results:** Mean σ and standard deviation (MPa) values were as follows: E1HF: 29.8±4.5(a); E1CS: 24.6±5.6(b); V7HF: 22.3±4.0(b); V7CS: 15.7±6.9(c). Mean σ value of Group E1HF was statistically higher than the other Groups mean values (p=0.0001). HF treatment produced significantly higher mean σ value than CS treatment, independent of the ceramic material (p=0.0001). All fractures occurred within the adhesion zone. E1HF showed the highest Weibull modulus (*m*) value (7.66) and V7CS exhibited the lowest *m* value (2.54). **Conclusion:** Results confirmed the testing hypothesis that σ of ceramics to resin is controlled primarily by the ceramic surface treatment.

Key-word: ceramic – fractography – surface treatment

1. INTRODUÇÃO GERAL

A opção pelo uso de restaurações totalmente cerâmicas por pacientes e dentistas é baseada nas propriedades únicas desses materiais, incluindo biocompatibilidade e estética. Entretando, falhas mecânicas freqüentemente ocorrem devido à fragilidade desses materiais quando submetidos a forças de tração. O desafio de pesquisadores e fabricantes de produtos odontológicos, têm sido produzir materiais cerâmicos que combinem suficiente resistência com estética (Albakry *et al*., 2003).

A introdução de sistemas cerâmicos com diferentes composições, combinada com o uso de novas técnicas laboratoriais, tem resultado em melhorias nas propriedades mecânicas e estéticas desses materiais (Cattel *et al.*, 1997; Höland *et al*., 2000). Dentre esses materiais encontra-se a IPS Empress, uma cerâmica vítrea reforçada por leucita, fabricada por um sistema de termo-injeção e comercializada na forma de pastilhas pré-ceramizadas. Assim, esse material é aquecido e injetado, por pressão, para dentro de um molde, resultando em diminuição de porosidade nas restaurações confeccionadas a partir desse processamento laboratorial (Cattel *et al.*,1997; Anusavice, 1997; Höland *et al*., 2000; Della Bona *et al.*, 2003a).

Entretanto, apesar de o sistema IPS Empress estar sendo bastante utilizado para fabricação de restaurações cerâmicas devido à precisão oclusal, adaptação marginal e translucidez, sua resistência flexural avaliada pelo teste de três pontos é de, aproximadamente, 110 MPa, o que o torna inadequado para confecção de próteses fixas totalmente cerâmicas, tendo seu uso indicado apenas para confecção de restaurações unitárias. Além disso, essa cerâmica apresenta alto coeficiente de expansão térmica (CET= 15,0 x $10^{-6}K^{-1}$), restringindo seu uso em conjunto com outros sistemas cerâmicos (Höland *et al*., 2000; Della Bona *et al.*, 2003a).

Outro material cerâmico lançado recentemente na Europa é o VITA**VM7**. Essa cerâmica foi idealizada para substituir a Vita Alpha para cobertura de infra-estruturas cerâmicas com valor de CET em torno de 7 x 10^{-6} K^{-1}, tais como os sistemas VITA In-Ceram® alumina, spinell e zircônia e o sistema procera (VITA, Zahnfabrik, 2004). A

resistência flexural, de acordo com o fabricante, é de 106 MPa. Essa cerâmica apresenta uma estrutura com partículas vítreas menores e distribuição mais homogênea, proporcionando um mínimo desgaste dos dentes antagonistas e melhor translucidez que a Vita Alpha (VITA, Zahnfabrik, 2004).

As restaurações produzidas pelo sistema VM7 são obtidas pela técnica da estratificação, diferentemente daquelas produzidas pela IPS Empress, que são confeccionadas pela técnica de volatilização da cera e prensagem em alta temperatura da cerâmica para dentro de um molde. O método de fabricação pode ser uma variável importante, com relação à quantidade e à localização dos defeitos estruturais (Anusavice, 1997; Tinschert *et al.*, 2000; Albakry *et al*., 2003; Della Bona *et al*., 2003a; Pallis *et al.*, 2004; Della Bona *et al.*, 2004a). A interação entre estresse e defeitos pode resultar na propagação catastrófica da falha e na fratura da restauração (Mecholsky, 1995; Ritter, 1995; Kelly *et al*., 1995).

A adesividade da cerâmica IPS Empress e VM7 à resina é baseada em mecanismos de retenção micromecânica (ação de ácidos e jatos com partículas a base de óxido de alumínio, Al_2O_3) e de união química (silanos). Esses tratamentos de superfície, quando devidamente utilizados, têm a propriedade de aumentar a energia de superficie e de diminuir o ângulo de contato, favorecendo o processo adesivo (Della Bona *et al*., 2004b). O silano faz a ligação entre a sílica contida na cerâmica e a matriz orgânica dos materiais resinosos (Della Bona *et al*., 2000; Jedynakiewicz & Martin, 2001; Hooshmand *et al*., 2001, 2002; Borges *et al*., 2003; Spohr *et al*., 2003; Della Bona *et al.*, 2004b).

A união entre cerâmicas ácido-sensíveis e resina em reparos intra-orais de estruturas cerâmicas, a partir do condicionamento com ácido hidrofluorídrico (HF), tem obtido resultados promissores de resistência adesiva (Della Bona & van Noort, 1995; Della Bona *et al*., 2000; Kato *et al.*, 2000; Blatz *et al.*, 2003; El-Zohairy *et al.*, 2003). Contudo, sabe-se que o contato do ácido hidrofluorídrico com o tecidos moles pode causar irritação (Szep *et al.*, 2000; Asvesti *et al*., 1997; Hoosmand *et al*., 2002; El-Zohairy *et al.*, 2003). Além disso, alguns autores sugerem que o HF pode fragilizar a superfície de algumas cerâmicas produzindo valores de adesão à resina inadequados clinicamente (Peumans *et al*., 2000; Della Bona *et al.*, 2000, 2003a), o que justifica a busca por outros meios que produzam

retenção micromecânica, como os jateamentos com Al_2O_3 (Della Bona *et al.*, 2000; Jedynakiewicz & Martin 2001; Hooshmand *et al*., 2002; Robin *et al.*, 2002; Oh & Shen, 2003; Özcan & Vallitu, 2003; Valandro *et al.*, 2005).

Entretanto, apenas recentemente, foi introduzida no mercado a tecnologia de jateamento de superfícies com partículas de óxido de alumínio modificadas por sílica. O objetivo deste sistema é produzir uma retenção micromecânica com deposição de sílica, favorecendo a união química com o silano, fenômeno conhecido como silicatização. O sistema Cojet (3M-Espe) foi o primeiro a possibilitar o uso dessa tecnologia, em consultório para cimentação e reparos de restaurações cerâmicas "fraturadas", constituindo-se numa nova alternativa clínica para esse procedimento (Frankerberger *et al*., 2000; Haselton *et al*., 2001; Jedynakiewicz & Martin 2001; Özcan 2002).

Dessa forma, para avaliar a integridade da interface adesiva *in vitro*, estudos sugerem que testes de resistência como microtração podem ser os mais apropriados, pois produzem uma distribuição mais uniforme do estresse nesta interface. Os testes de microtração, por apresentarem uma área de teste menor e, conseqüentemente, menor número de defeitos, tendem a produzir resultados ainda mais representativos, porque as falhas ocorrem quase que exclusivamente na interface adesiva, permitindo uma análise da real resistência de união às cerâmicas (Della Bona *et al.*, 2000; Wegner *et al*., 2002; El-Zohairy *et al.*, 2003; Oh & Shen, 2003).

A literatura científica envolvendo testes de resistência adesiva por microtração de resinas unidas às cerâmicas após diferentes tratamentos de superfície e posterior análise fractográfica ainda é insuficiente para inferências clínicas adequadas, pois são raros os estudos que consideram qualitativamente o modo de falha relativo aos valores quantitativos de resistência de união (Della Bona *et al.*, 2002; Della Bona *et al.*, 2003a; Della Bona *et al.*, 2003b). A caracterização do modo da fratura observado por meio da análise fractográfica, é muito importante para o entendimento e prognóstico de uma interface adesiva (Mecholsky, 1995; Della Bona *et al.*, 2000).

2. CAPÍTULO

Tensile bond strength and mode of failure of ceramics bonded to resin

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ABSTRACT

Statement of problem: Silica coating has been suggested to treat high-crystalline ceramics for bonding to resin. This bonding mechanism might be used to treat feldspathic ceramics, avoiding the potentially hazardous process of hydrofluoric acid etching.

Purpose: To evaluate the effect of ceramic surface treatments on tensile bond strength (σ) and the mode of failure of a resin bonded to a glass and a low-crystalline ceramics.

Material and Methods: Ten blocks of each the feldspathic glass (V7-VITAVM7) and the leucite-based ceramic (E1-IPS Empress) were fabricated and polished. Five blocks of each ceramic were treated as follows: HF, 9.5% hydrofluoric acid for 60 s; CS, silica coating using Cojet System for 15 s. After silane coating, an adhesive resin and a composite were applied and polymerized. The composite-ceramic blocks were cut to produce bar-shaped specimens (n=30) that were stored in distilled water at 37°C for 7 days before tensile loading to failure in a universal testing machine. Data were statistically analyzed using analysis of variance, Tukey's test (α=.01) and Weibull analysis. Fracture surfaces were examined to determine the mode of failure.

Results: The Weibull modulus (*m*) and mean σ value (MPa) of Group E1HF (29.8±4.5) were significantly higher than other Groups (*P*=.0001). There was no statistical difference between Groups E1CS (24.6±5.6) and V7HF (22.3±4.0). Group V7CS showed the lowest *m* and mean σ values (15.7±6.9) (*P*=.0001). All fractures occurred within the adhesion zone.

Conclusion: HF etching produces the highest *m* and σ values of resin bonded to both E1 and V7 ceramics.

CLINICAL IMPLICATIONS

Silica coating is not the ceramic treatment of choice for bonding to resin. HF etching produced the higher tensile bond strength of resin to both the glass and the low-crystalline content ceramic tested.

INTRODUCTION

The increasing acceptance of all-ceramic restorations by both dentists and patients is based on the unique properties of these materials, including biocompatibility and esthetics. However, mechanical failure often occurs because of the inability of ceramic materials to accommodate tensile forces by plastic deformation. The challenge for most researches and manufacturers has been the production of a ceramic material that combines sufficient strength with esthetic required in dentistry. [1]

The introduction of ceramics with different compositions combined with the use of novel laboratory techniques has resulted in materials with improved mechanical properties and heightened esthetics. [2, 3] One of these materials is IPS Empress (Ivoclar AG, Schaan, Liechtenstein), a hot-pressed leucite-based glass-ceramic, with properties well reported in the literature. [2-6]

Another commercially available material is VITA**VM7** ceramic (VITA Zahnfabrik, Bad Säckingen, Germany), that is a new veneering material, which is used on all-ceramic structures with coefficient of thermal expansion around 7 x 10^{-6} K^{-1}, including the Vita In-Ceram systems and the Procera system. [7]

IPS Empress and VITAVM7 ceramic restorations are fabricated via distinct processing methods. The IPS Empress is a hot-pressed leucite-based glass-ceramic while

VM7 is a sintered feldspathic ceramic applied on high crystalline content ceramic structures. The processing method can be an important variable in regarding the quantity and location of the defects. [1, 5, 6, 8, 9] The interaction between stress and defects can result in a catastrophic propagation of a critical crack. [10-12]

The bond strength of a resin to a ceramic substrate is traditionally based in mechanisms of micromechanical retention (airbone-particle abrasion and acid etching) and chemical adhesion via organosilanes. [5, 6, 13-21]

The use of hydrofluoric acid (HF) is the most popular ceramic surface treatment used for resin bonded restorations and repair of acid-sensitive ceramic restorations. This procedure, followed by silane application, produces a clinically acceptable resin bond to silica-based ceramics. [16, 22-25] Yet, it is known that the HF is extremely caustic to soft tissues and requires much caution for clinical use. [15, 26-28]

Furthermore, some studies suggested that HF may weaken the surface of some ceramics producing clinically inadequate bond strength values to resin. [5, 16, 29] Therefore, it seems appropriate to investigate alternative intraoral mechanisms for producing mechanical retention on ceramic surfaces, such as the airbone-particle abrasion using silica modified Al_2O_3 particles, the so called silica coating procedure. In this technique the ceramic surface is air abraded with 30-μm Al_2O_3 particles modified by silica followed by a silane application. [13, 30-32]

To assess the quality of the interfacial bond between ceramic and resin, it has been suggested the use of tensile bond strength tests coupled with fractographic analysis of the fracture surfaces. This quantitative and qualitative assessment of the adhesion zone should produce a more consistent and complete description of the bond and fracture phenomena,

reducing the risk of data misinterpretation. Scientific literature on such approach to investigate the adhesion mechanisms of resin bonded to ceramic is unusual but it should provide adequate clinical prediction of the success of bonding procedures for repairing and resin luting ceramic restorations. [5, 10, 16] Therefore the objective of the present study was to evaluate the effect of ceramic surface treatments on tensile bond strength and the mode of failure of a resin bonded to no- and low-crystalline ceramics, testing the hypothesis that the bond strength of ceramics bonded to resin is controlled by the ceramic surface treatment.

MATERIALS AND METHODS

Ten ceramic blocks (8 mm × 8 mm × 8 mm) each of the hot-pressed leucite-based ceramic (IPS Empress (E1); batch no. F6493, Ivoclar AG, Schaan, Liechtenstein) and the feldspathic glass (VITAVM7 (V7); batch no. 7318, VITA Zahnfabrik, Bad Säckingen, Germany) were fabricated according to the manufacturer's instructions, polished through 1200-grit metallographic paper (3M-ESPE, St. Paul, Minn) using a polishing machine (APL-4, Arotec Inc, São Paulo, SP, Brazil) and finished with 1 μm polishing diamond paste. All ceramic blocks were ultrasonically cleaned in distilled water for 10 min and treated as follows. For Groups E1HF and V7HF, five blocks of each ceramic material were randomly sampling and their polished surface was treated with 9.5% hydrofluoric acid (HF- batch no. 3Q5Y, Ultradent Poducts, Inc, South Jordan, UT) for 60 seconds.

For Groups E1CS and V7CS, the remaining five blocks of each ceramic material had their polished surface treated with airborne-particle abrasion with 30-μm Al_2O_3 particles modified by silica (Cojet-Sand (CS); batch no. 004, 3M-ESPE, Seefeld,

Germany). The abrasion was applied (Micro-Etcher; Danville Inc, San Ramon, Calif) perpendicular (90º) to the surface at a distance of 10 mm, for 15 seconds, and at a pressure of 2.8 bars. [32]

All treated ceramic surfaces were washed under running water for 30 seconds and dried. The surfaces were coated with a silane coupling agent (Batch no. 124, ESPE-Sil, 3M-ESPE), which was allowed to air dry for 5 minutes. [30, 32-34]

An adhesive resin (Single Bond, batch no. 8BJ, 3M-ESPE) was applied onto the treated ceramic surfaces and polymerized for 20 seconds (XL 3000; 3M ESPE; light output = 500 mW/cm^2). The ceramic blocks were placed into a mold made of an addition silicone impression material (Elite HD, batch no. Bo1.01.B; Zhermack, Badia Polesine, Rovigo, Italy) and four 2-mm thick incremental layers of resin composite (Filtek Z250, Batch no. EXI-127, 3M-ESPE) were condensed on the treated ceramic surface to build a composite block. Each composite layer was polymerized for 40 seconds (XL 3000; 3M ESPE).

The composite-ceramic blocks were bonded with cyanoacrylate (Zapit, Dental Ventures of America Inc., Corona, CA) to an acrylic base, which was attached to a low-speed, automatic precision cutting machine (Minitom, Struers, Copenhagen, Denmark). Slices approximately 1.02 mm thick were obtained using a slow-speed diamond wheel saw (Sultrade, Com. Exp. Ltda, São Paulo, SP, Brazil) under water cooling. The peripheral slices were discarded because the results could be influenced by either an excess or an insufficient amount of resin composite and/or adhesive at the interface.[16,32,35] Nontrimmed specimens were obtained directly from the cutting machine, meaning, neither polishing nor finishing were performed. This procedure was used to avoid stress concentration at the adhesive interface by polishing materials with different elastic modulus. [16] Six non-

trimmed bar specimens with a bonding area of approximately 1.04 mm^2 were obtained per block (n = 30). [16, 32, 36-39]

Specimens were stored in distilled water at 37°C for 7 days before testing. Each specimen was attached to the flat grips of the Bencor Multi-T device (Danville Engineering, San Ramon, Calif) using cyanocrylate adhesive (Zapit, Dental Ventures of America Inc., Corona, Calif) and loaded to failure in tension at a crosshead screw speed of 1 $mm.min^{-1}$ using a universal testing machine (EMIC DL2000, EMIC, São José dos Pinhais, Brazil). [16, 32, 38, 40]

The bonding area of all specimens was measured individually with a digital caliper (Digimatic caliper, Mitutoyo Co., Kawasaki, Japan) immediately after testing and used to calculate the bond strength. Tensile bond strength (σ) values were calculated using $\sigma = L/A$, where "L" is the load at failure (N) and "A" is the adhesive area (mm^2). [16] The results were analyzed using one-way ANOVA and Tukey's test (α=.01) and statistical software (Statistix 8.0 for Windows, Analytical Software Inc., Tallahassee, FL, USA). As the size of the bonded cross-sectional area can affect the calculated bond strength, a linear regression analysis was performed to determine if such a relationship existed for the experimental data of this study. Weibull analysis was also performed to evaluate the structural integrity of the adhesion zone. [5] Fractured surfaces were examined using scanning electron microscopy (SEM- JEOL–JSM–5600 LV, Jeol Ltd, Tokyo, Japan) to determine the mode of failure based on the fracture origin and factrographic principles. [5, 10, 41] In preparation for SEM examination (Jeol – JSM – 5600 LV, Tokyo, Japan), the specimens fracture surfaces

were sputter-coated (Balzers-SCD 050, Liechtenstein, Germany) with gold-palladium for 3 minutes, at a current of 10 mA, and vacuum of 130 mTorr.

Additional HF- and CS-treated V7 ceramic samples were prepared for surface topography investigation and examined under the SEM as mentioned above. These analyses were not done for ceramic E1 since these results are reported in previous studies. [5, 6, 42, 43] Some CS-treated ceramic specimens were analyzed for the silica content. Silica mappings were generated using energy-dispersive spectroscopy (EDS) at 20 Kv. [32] Representative images and spectra were recorded.

RESULTS

One-way ANOVA, described in Table I, was used to statistically analyzed the data. As statistically differences were found among groups, Tukey's test was used (α=.01). The mean bond strength values, standard deviations, and Tukey grouping are presented in Table II.

Table I. One-way ANOVA

Source	*df*	SS	MS	F	P
Groups	3	3079.4276	1026.7559	35.13	0.0001
Error	116	3389.10833	29.21645		
Total	119	6468.53592			

df, degrees of freedom; SS, sum square; MS, mean square

The mean bonding area of the specimens was 1.04 ± 0.01 mm^2. Linear regression analysis showed that tensile bond strength values were statistically independent of the size of the bonding area.

Table II. Mean tensile bond strength (σ), standard deviation (SD), Tukey grouping, characteristic strength (σ_o), strength value at 5% failure rate ($\sigma_{0.05}$), Weibull modulus (m) and the mode of failure (percentage per mode) for microtensile bond strength tested specimens.

Experimental groups	σ (SD)* (MPa)	σ_o (MPa)	$\sigma_{0.05}$ (MPa)	m	Mode of failure
E1HF	29.8 (4.5)[a]	31.7	21.5	7.7	**5**: 90.0%; **2**: 10.0%
E1CS	24.6 (5.6)[b]	26.8	14.8	5.0	**5**: 96.7%; **3**: 3.3%
V7HF	22.3 (4.0)[b]	24.0	14.7	6.1	**5**: 90.0%;**4**: 3.3%; **2**: 6.7%
V7CS	15.7 (6.9)[c]	17.7	5.5	2.5	**5**: 76.7%; **2**: 6.7%; **1**: 16.6%

*Means not statistically different share same letters; Coefficient of variance is 21.3%
E1: IPS Empress; V7: VitaVM7; HF: hydrofluoric acid; CS: CoJet system

One-way ANOVA showed that the mean σ value of E1HF was statistically higher than the means of the other groups (p=0.0001). HF-treated specimens (Groups E1HF and V7HF) produced significantly higher mean σ value than the corresponding CS-treated specimens (Groups E1CS and V7CS) (p=0.0001). Group V7CS showed the lowest mean tensile bond strength (p=0.0001) and the highest standard deviation.

The Weibull and fracture analyses of the experimental groups are summarized in Table II. The highest and lowest Weibull modulus (m) values were associated, respectively, with groups E1HF (7.7) and V7CS (2.5).

Representative SEM images of HF- and CS-treated V7 ceramic specimens are shown in Figure 1. Specimens in Group V7HF revealed a typical retentive surface pattern with the formation of grooves (Figure 1, A and B). CS-treated V7 ceramic specimens showed a deposition of particles onto the surface (Figure 1, C and D). EDS analysis confirmed the presence of silica in the deposited surface particles. The initial composition of V7 ceramic was Si(K) 19.6%; Al(K) 4.9%; K(K) 4.0%; Na(K) 2.4%; Ca(K) 0.7%; C(K)

25.7%; O(K) 42.2%. The amount of silicon (SiK) after silica coating the V7 ceramic was 20.3%.

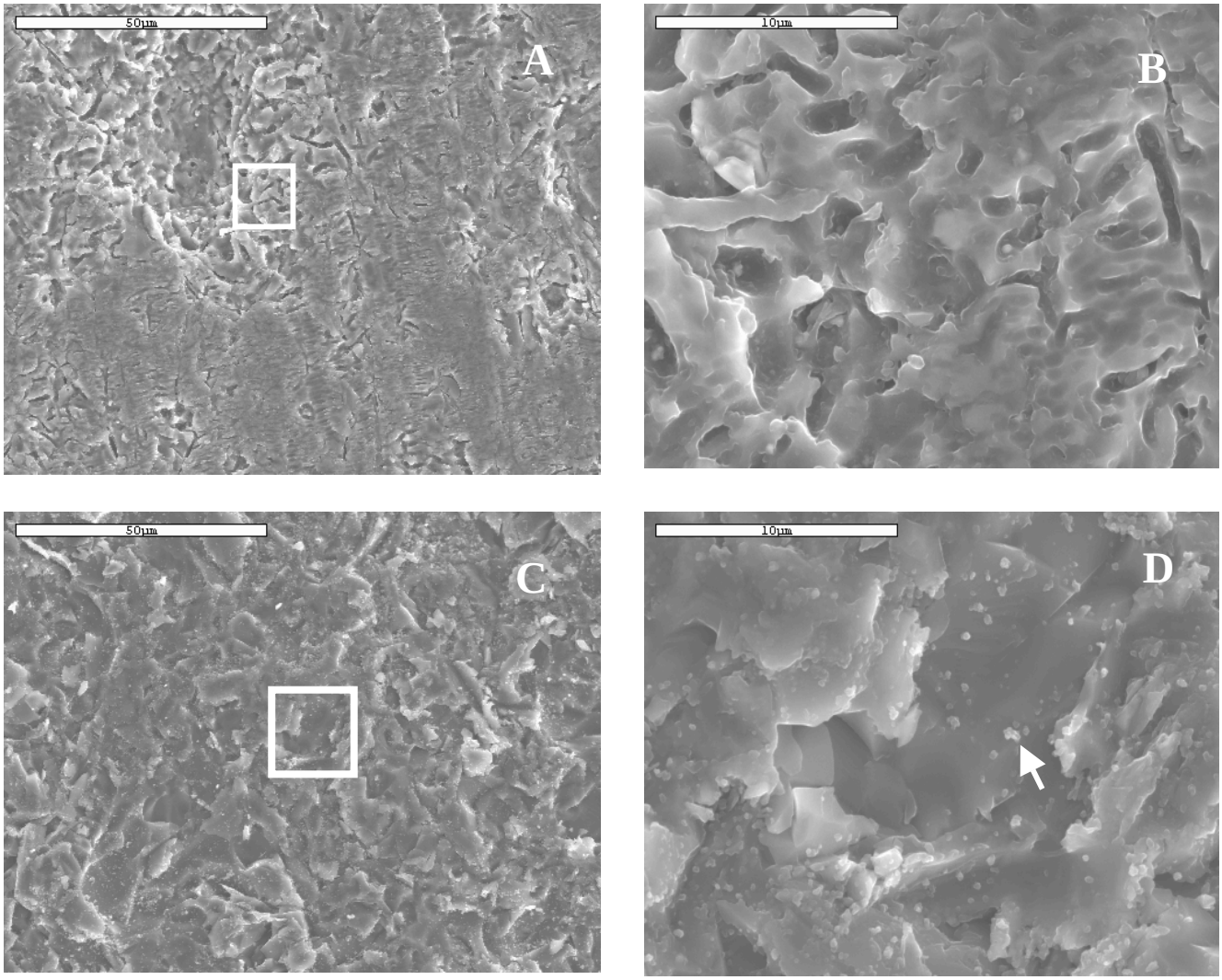

Figure 1. Representative SEM images of HF- and CS-treated V7 ceramic specimens. **A,** HF-treated V7 ceramic surface (Group V7HF) showing the production of retentive grooves (original magnification X1000); area within the white square is magnified in **B** (original magnification X5000). **C**, CS-treated V7 ceramic surface (Group V7CS) showing a deposition of silica modified alumina particles from the airborne-particle abrasion with Cojet system (original magnification X1000); area within the white square is magnified in **D** (original magnification X5000), white arrow shows particle from the Cojet system.

The SEM analysis revealed that all fractures occurred within the adhesion zone (Figure 2). The "adhesion zone" is defined as the region in which the adhesive interacts with the two substrates to promote bonding. The adhesion zone in this study consists of the following: (1) the interfacial region between the adhesive and the resin composite within which molecular interaction and chemical bonding occur between the two materials; (2) the adhesive; (3) the interfacial region between the adhesive and the dental ceramic, including the surface region treated with the HF or CS and coated with silane such that micromechanical and chemical bonding occurs. [5]

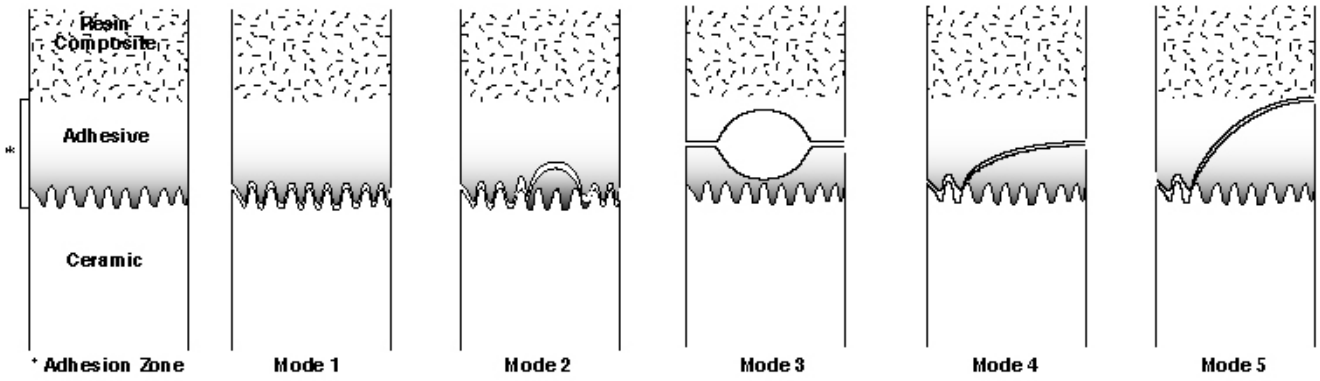

Figure 2. Schematic representation (side view) of the modes of failure for the microtensile bond strength test of ceramic bonded to resin composite. **Mode 1**: adhesive separation at the ceramic - adhesive resin (C-A) interface. **Mode 2**: failure starts at the C-A interface, progresses into the adhesive resin (A) and returns to the C-A interface (C-A-C). **Mode 3**: failure originates from an internal flaw (penny-shape internal crack). **Mode 4**: failure starts at the C-A interface and propagates through the adhesive resin (A). **Mode 5**: failure starts at the C-A interface, propagates though the adhesive resin (A) to reach the adhesive - resin composite (A-R) interface (C-A-R). With permission of and adapted from Della Bona *et al.*, 2003. [5]

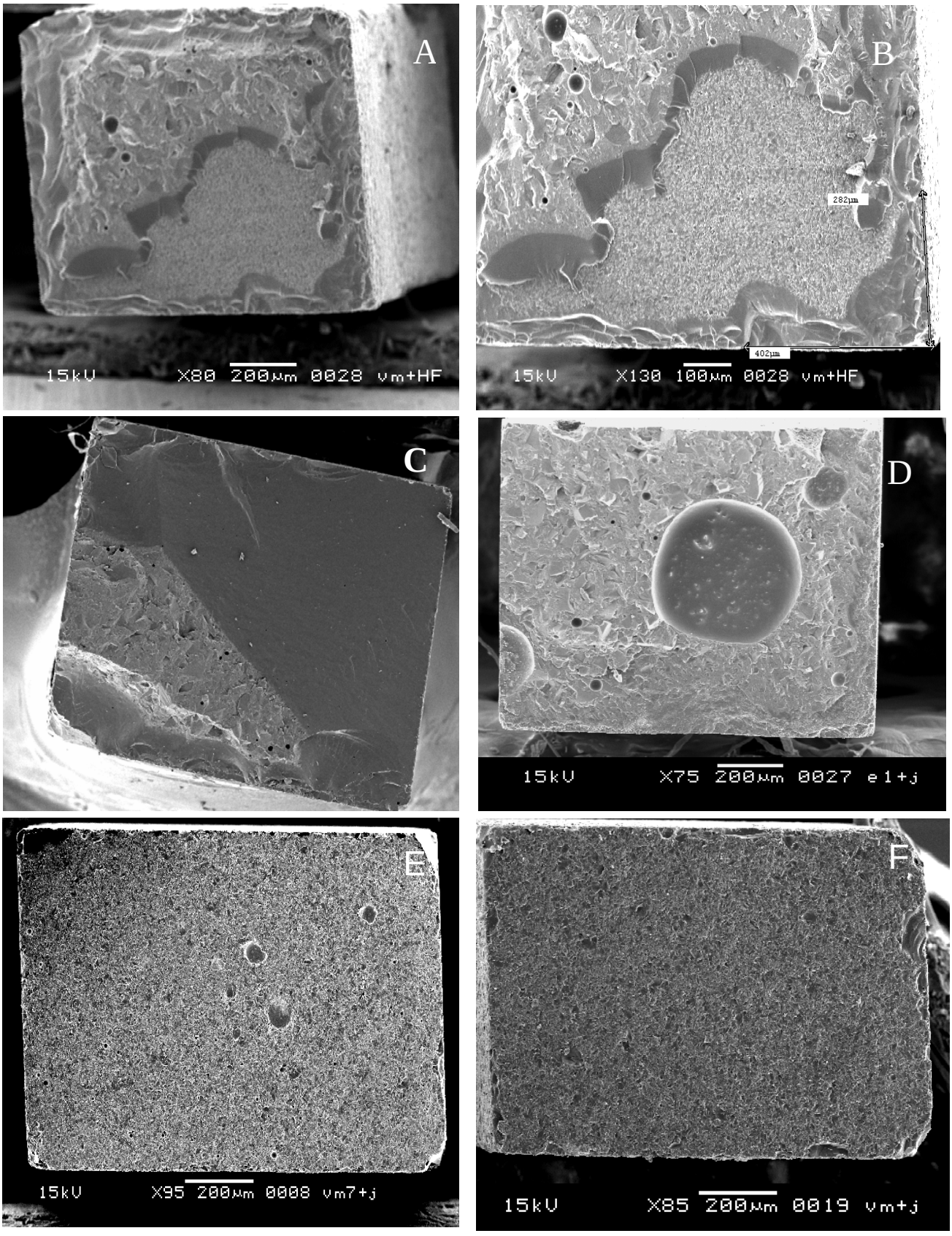
A
15kU X80 200µm 0028 vm+HF
B
282µm
402µm
15kU X130 100µm 0028 vm+HF
C
D
15kU X75 200µm 0027 e1+j
E
15kU X95 200µm 0008 vm7+j
F
15kU X85 200µm 0019 vm+j

Figura 3. Representative SEM micrographs of fracture surfaces corresponding to the modes of failure found in this study and schematically illustrated in Figure 2. **A**, fracture surface of specimen from Group V7HF that failed in Mode 5 (original magnification X80). **B**, fracture origin (measuring arrows) of specimen in image **A** (original magnification X100). **C**, specimen from Group V7HF that failed in Mode 4 (original magnification X80). **D**, Mode 3 (internal flaw) was reported for a specimen from Group E1CS (original magnification X75). **E**, fracture surface of specimen from Group V7CS that failed in Mode 2 (original magnification X95). **F**, specimen from Group V7CS that failed in Mode 1, adhesive failure (original magnification X85).

Examination of the fracture surfaces showed no bulk fracture at the origin of failure for either the resin composite or the dental ceramics. The mode of failure was determined using fractographic principles and classified as shown in Figure 2 and Table II.

The Mode 5 was the predominant type of failure for specimens in all Groups (Figure 3, A and B). Mode 4 was the mode of failure in one specimen of Group V7HF (Figure 3, C). Mode 3 (internal flaw) was the mode of failure in one specimen of Group E1CS (Figure 3, D). Mode 2 was the mode of failure in two specimens of groups V7HF and V7CS, and in three specimens of group E1HF (Figure 3, E). The purely adhesive failure (Mode 1) was found in five specimens of Group V7CS (Figure 3, F).

DISCUSSION

Incorrect design of the metal frame, defects in the ceramic-core interface or local overload may cause fracture of porcelain veneering. [11, 10] These fractures are remarkably frequent within the first few months after the incorporation of the restoration and failure rates are up to 9%. [30]

Complete removal of the fractured restoration is unpleasant and expensive for the patient; therefore, the possibility to repair metal- and all-ceramic restorations intraorally is a worthwhile clinical challenge. [26, 30-33]

The clinical success of a repaired ceramic restoration will depend on the quality and durability of the bond between the ceramic and the resin composite. The quality of this bond will depend upon the bonding mechanisms that are controlled in part by the specific surface treatment to promote micromechanical and/or chemical retention with the substrate and by the substrate microstructure. [5, 43]

Studies suggest that a tensile bond strength test may be more appropriate to evaluate the bond strength of adhesive interfaces because of more uniform interfacial stresses distribution. [16, 39, 40] The non-uniform interfacial stress distribution generated for conventional tensile and shear bond strength tests initiates fractures from flaws at the interface or in the substrate in areas of high stress concentration. [5, 22, 35]

The results of this study showed that the mean tensile bond strength (σ) values of HF-treated ceramics were significantly higher than the mean σ values of the corresponding ceramics treated with CS ($p=0.0001$). These results are in agreement with other studies in which HF produced higher bond strength values suggesting that the use of this ceramic surface treatment is the method of the choice to promote bonding between resin composite and silica-based ceramics. HF selectively attacks the glassy phase, phase boundaries and material defects, producing a porous, irregular surface that increases the surface area and facilitates the penetration of the resin into the microretentive etched ceramic surface. [14, 16-19, 23, 24, 28]

For each ceramic surface treatment, the mean σ values were statistically higher for E1 than for V7. These differences in bond strength can be explained by the difference in ceramic composition and microstructure. E1 has about 40% of leucite crystals, which improve the mechanical properties, [2, 3, 6] while V7 is a feldspathic glass with no crystalline phase. [7]

The processing method can be an important variable regarding defect quantity and location. [2, 4, 8, 9] The interaction between stress distribution and defects can result in catastrophic propagation of a critical flaw. [10-12, 41] The higher mean tensile bond strength values of E1 ceramic bonded to resin suggest that processing, microstructure and composition of the ceramic substrate play an important role in the adhesion process between ceramics and resins, which is in agreement with previous reports. [5, 16, 24] The E1 is hot-pressed ceramic system provided as core ingots that are heated and pressed until the ingot flows into a mold, producing a relatively pore-free restorations, [3] V7 is a feldspathic glass fabricated by vacuum sintering of the ceramic powder, which is more prone to create processing defects.

The Weibull analysis gives values for the shape parameter or Weibull modulus (*m*) and for the scale parameter or characteristic strength (σ_o). The *m* gives an indication of the reliability of the bond strength, describing the relative spread of strength values in the asymmetrical distribution with higher values indicating narrower distribution of the bond strength. The σ_o represents the value at which 63.21% of the test specimens fracture. [8, 11, 42, 21] The scale and the shape parameters correspond to the mean value and the standard deviation for materials with a Gaussian strength distribution, respectively. The Weibull

modulus compensates the lower range of values whose asymmetry is typical for ceramic materials. [8, 42, 21]

Group E1HF exhibited the highest *m*, σ_o and strength value at 5% failure rate ($\sigma_{0.05}$). Yet, HF-treated ceramic specimens (Groups E1HF and V7HF) revealed fracture surfaces with several fracture events starting at the specimen edges of the ceramic-resin adhesive interface (Figure 3, A, B, and C). These observations suggested that HF may have a weakening effect on the surface of ceramics E1 and V7, which agrees with previous reports. [5, 16, 29]

Based on microscopy and bond strength data analyses, the CS-treated V7 ceramic produced an insufficient micromechanical retentive surface (Figure 1, C and D) and, as a consequence, specimens of Group V7CS showed the lower mean σ and the highest standard deviation of all groups. The adhesive failure (mode 1) was found for five specimens in this Group (Figure 3, F). In addition, the V7CS group showed the lowest *m*, σ_o and $\sigma_{0.05}$ values, suggesting a poor bonding reliability (Table II). SEM and EDS analyses, along with the bond strength results, showed that silica coating the high silica content ceramics tested (E1 and V7) is not the procedure of choice for bonding to resin.

High mean bond strength values of silica-coated high crystalline content ceramic bonded to resins have been reported, [19, 20] suggesting that the tribochemical adhesive mechanism is a promising technique for bonding to acid-resistant ceramics. Yet, it seems that the topography of the silica-coated ceramic surface varies depending on the matrix-crystal ratio and the crystal size distribution. [25] It is possible that silica coating a high silica content feldspathic glass, such as V7, does not produce an adequate retentive surface for bonding to resin because of the absence of a crystalline phase and, consequently, the

presence of fewer phase boundaries, which are more susceptible to the action of airborne particle abrasion and acids. Analogous to these findings, silica coating seemed to provide some micromechanical retention for E1 ceramic to resin, probably because of the crystalline content (leucite) in the microstructure. [6, 13, 30] Yet, the mode of failure was similar for groups E1CS and E1HF, which was predominantly mode 5. SEM observations of the fractured surfaces of E1 ceramic specimens treated with HF and CS (groups E1CS and E1HF) showed less edge fractures at the ceramic-resin interface, suggesting that both ceramic treatments did little weaken the E1 ceramic structure. In addition, the amount and nature of the crystalline components enhance the mechanical properties and fracture toughness of E1, hinder crack propagation. [1] These observations suggest that the lower the ceramic crystalline content the lower the bond strength to resin.

This rationale supports the testing hypothesis that the tensile bond strength of ceramic to resin is affected by the ceramic surface treatment, which has been also suggested by previous studies. [5, 13-15, 34, 42, 43] The results of the present study enforce the importance and the relationship of materials microstructure and composition, and the surface treatments for bonding ceramics to resins. Results are relevant to the materials and procedures used in the present study. Future studies should examine the effect of silica coating on the bond strength to other glasses and low-crystalline content ceramics.

CONCLUSIONS

The results of this study confirmed the test hypothesis that the tensile bond strength of ceramic bonded to resin is controlled by the ceramic surface treatment, which is directly related to the ceramic microstructure. In addition, the microtensile test appears to be an

adequate method to evaluate the bond strength of the resin-ceramic interface, since all fractures occurred within the adhesion zone.

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3. CONCLUSÃO GERAL

Os resultados deste estudo confirmam a hipótese experimental de que a resistência adesiva da resina à cerâmica é controlada, primariamente, pelo tratamento de superfície do material cerâmico, o qual é diretamente relacionado a microestrutura cerâmica. Além disso, o teste de microtração demonstrou ser um método adequado para avaliar a resistência de união da interface cerâmica-resina, uma vez que todas as fraturas ocorreram dentro da zona de adesão.

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ANEXOS

Anexo 1. Valores de resistência adesiva à microtração (σ).

Tabela 1. Grupo E1CS

Corpo- de- prova	(σ) MPa
1	31,0
2	32,9
3	18,6
4	23,3
5	27,2
6	23,2
7	33,8
8	28,6
9	35,7
10	23,5
11	15,6
12	21,2
13	16,4
14	19,1
15	18,2
16	27,9
17	16,4
18	17,5
19	28,0
20	29,2
21	27,4
22	28,3
23	23,5
24	18,6
25	26,6
26	28,3
27	21,7
28	30,4
29	20,1
30	26,3

Tabela 2. Grupo V7CS

Corpo-de-prova	(σ) MPa
1	21,1
2	26,6
3	17,5
4	25,0
5	16,1
6	23,7
7	13,8
8	6,8
9	7,6
10	15,5
11	21,1
12	14,9
13	9,0
14	26,9
15	6,2
16	13,7
17	19,4
18	5,7
19	5,9
20	23,1
21	22,1
22	12,0
23	8,1
24	11,2
25	21,3
26	9,0
27	22,4
28	16,2
29	6,6
30	22,5

Tabela 3. Grupo E1HF

Corpo-de-prova	(σ) MPa
1	21,9
2	19,8
3	25,7
4	27,2
5	29,0
6	28,8
7	35,2
8	32,7
9	36,2
10	33,4
11	33,7
12	38,5
13	24,2
14	36,2
15	29,2
16	31,7
17	20,9
18	30,6
19	32,1
20	30,2
21	35,9
22	28,0
23	26,9
24	27,9
25	31,2
26	28,2
27	29,6
28	28,7
29	32,8
30	27,8

Tabela 4. Grupo V7HF

Corpo-de-prova	(σ) MPa
1	28,2
2	20,3
3	24,4
4	23,2
5	22,6
6	27,3
7	22,2
8	29,6
9	29,3
10	17,6
11	24,2
12	23,7
13	18,0
14	20,1
15	18,9
16	18,9
17	20,7
18	23,0
19	18,1
20	17,3
21	26,4
22	25,0
23	29,4
24	17,1
25	22,7
26	19,1
27	25,8
28	16,7
29	16,7
30	23,1

Anexo 2. Análise Estatística.

Tabela 5. Análise de Variância – Variável Resistência.

Causas da Variação	Graus de Liberdade	Soma dos Quadrados	Quadrados Médios	Teste F	p-valor
Grupos	3	3079,4276	1026,7559	35,13	< 0,0001
Erro	116	3389,10833	29,21645		
Total	119	6468,53592			

Coeficiente de variação: 21,3%

Tabela 6. Valores médios de resistência adesiva à microtração (σ) e desvio padrão (DP), em cada grupo.

Grupos Experimentais	σ ± DP (MPa)
E1HF	29,81 ± 4,56 a
E1CS	24,62 ± 5,62 b
V7HF	22,32 ± 4,06 b
V7CS	15,70 ± 6,93 c

Médias seguidas por letras distintas, indicam que os grupos diferem estatisticamente entre si, pelo teste de Tukey (α=.01). Coeficiente de variação de 21,3%. HF: Ácido Hidrofluorídrico; CS: Sistema Cojet; E1: IPS Empress; V7: VitaVM7.

Tabela 7. Análise de Weilbull, parâmetro de forma (m), parâmetro de escala (σ_0) e resistência à fratura no índice de falha de 5% ($\sigma_{0.05}$), para cada grupo.

Estimativa dos Parâmetros	E1HF	E1CS	V7HF	V7CS
Forma (m)				
Estimativa	7,661	5,018	6,060	2,547
Desvio Padrão	1,081	0,7161	0,848	0,383
I.C. 95%				
Lim.Inf.	5,810	3,794	4,606	1,896
Lim.Sup.	10,103	6,6379	7,9735	3,4208
Escala (σ_0)				
Estimativa	31,702	26,827	24,014	17,757
Desvio Padrão	0,797	1,031	0,766	1,340
I.C. 95%				
Lim.Inf.	30,177	24,880	22,558	15,317
Lim.Sup.	33,303	28,926	25,565	20,587
Resistência à fratura no índice de falha de 5% ($\sigma_{0.05}$)	21,513	14,843	14,711	5,533