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Heat treatment of silanized feldspathic ceramic: Effect on the bond strength to resin after thermocycling

Viviane Maria Gonçalves de Figueiredo [a], Pedro Henrique Corazza [b], Laura Soares Souto Lepesqueur [a], Geraldo M. Miranda [c], Clovis Pagani [c], Renata Marques de Melo [a], Luiz Felipe Valandro [d,*]

[a] Department of Dental Materials and Prosthodontics, Institute of Science and Technology of São José dos Campos, São Paulo State University (UNESP), Street Francisco José Longo, No. 777, Jardim São Dimas, São José dos Campos, CEP 12245-000 São Paulo, Brazil
[b] Post-graduation Program in Dentistry, Dental School, University of Passo Fundo, BR 285, Km 171, Passo Fundo, CEP 99001-970 Rio Grande do Sul, Brazil
[c] Department of Restorative Dentistry, Institute of Science and Technology of São José dos Campos, São Paulo State University (UNESP), Av. José Longo, No. 777, Jardim São Dimas, São José dos Campos, CEP 12245-000 São Paulo, Brazil
[d] Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Brazil Street Floriano Peixoto, 1184, 97015-372 Santa Maria, Rio Grande do Sul, Brazil

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ABSTRACT

Purpose: This study aimed to evaluate the effect of heat treatment (at 77 °C) of a silanized feldspathic ceramic on microtensile bond strength (μTBS) with a resin cement before and after being aged by thermocycling.
Material and methods: Twenty-four blocks ($12 \times 10 \times 4$ mm^3) of a CAD/CAM feldspathic ceramic (Vita-blocks Mark II, Vita) were obtained and randomly divided into three groups, according to the surface treatment prior to the cementation: Group AS – hydrofluoric acid 10%+silane; Group S77 – silane+heating at 77 °C for 60 s; and Group AS77 – hydrofluoric acid 10%+silane+heating at 77 °C for 60 s. Ceramic blocks were cemented to composite resin blocks with a resin cement. The sets were subsequently cross-sectioned into 1 mm^2 beams for μTBS testing. The beams of each group were randomly divided into two subgroups: aging (thermocycling, 12,000 cycles between 5 °C and 55 °C) and non-aging (tested immediately). One-way ANOVA and Tukey's test (α=0.05) and Weibull analysis (95% CI) were used to analyze the data.
Results: Group AS77 had the lowest pre-test failure number during the cutting among the groups. There was no significant difference (p=0.255) between the μTBS mean values of the non-aged groups. After aging, the mean value of S77 was significantly lower than those of AS77 and AS (p=0.005). There was no difference in the Weibull modulus (m) and characteristic strength (σ_0) of the aged and non-aged groups for all comparisons. Before aging, heat treatment of silanized feldspathic ceramic (non acid-etched surface) demonstrated bond strength similar to that achieved with hydrofluoric-acid-etching treatment however, it had lower bond strength after aging.
Conclusion: The combination of hydrofluoric-acid-etching treatment with heat treatment silanized feldspathic ceramic did not improve the bond strength of the interface.

1. Introduction

Feldspathic ceramic has been used in dental restorations mainly due to the excellent long-term esthetics provided by its color stability. The established surface treatment for this class of ceramic to bond with resin cements is the 'hydrofluoric acid etching+silane coupling agent application' [1–3]. Surface etching with hydrofluoric acid promotes roughness on the ceramic [1], favoring micromechanical interlocking, and also improves the wettability of the silane on the ceramic surface, by energy surface modification [4]. Nevertheless, the use of hydrofluoric acid has been questioned in some studies, mainly due to its chemical toxicity and hazardous effects, but also due to the formation of insoluble salts that can affect bond strength [3,5–11]. Therefore, alternative treatments for bonding to these ceramics have been proposed, as silane heating in air stream [5,6,12,13] or oven [14–17], and bath in hot water [17]. These temperature-based treatments have the ability to eliminate solvents of the material and enhance the crosslink reaction, favoring the bonding [17].

* Corresponding author. Tel.: +55 55 3220 9276; fax: +55 55 3220 9272.
E-mail address: lfvalandro@hotmail.com (L.F. Valandro).

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The silane coupling agent consists of a bifunctional molecule with an organic side (reacts with resins and promotes the formation of oligomers) and an inorganic side (reacts with metal oxides and silica of the ceramic glass phase). This characteristic is responsible for cement/ceramic chemical bonding [1,3,4,6,18]. The greater the interaction of the silane molecules on the ceramic surface, the better the bond strength of the system and the resistance to degradation of the silane film [18,19]. After final condensation, the silane film is basically composed of three structurally and physicochemically different layers: (a) an outer layer, in which silane molecules are weakly attached, with few siloxane bonds that are easily hydrolyzed and removed; (b) a middle layer, in which molecules have strong siloxane bonds that are more resistant to hydrolysis and can be removed only with a hot water bath; and (c) an inner layer, in which the silane is considered chemically reactive [20].

Studies testing the bond strength of the ceramic/resin interface have shown positive results of heat treatment of the silane layer before cementation [5,6,8,12,15,17,21]. Specifically, silane heat treatment at 77 °C (solvent evaporation temperature) increased the bond strength between the ceramic and resin cement compared with that achieved by conventional treatment, even after mechanical aging, showing the stability of the technique after short-term mechanical cycling [17]. However, the literature lacks studies concerning the real effect of temperature (hot air without the association of an air spray), mainly after aging by thermocycling, on the bond strengths between feldspathic ceramic and resin cement. Moreover, the association of heat treatment with the conventional protocol after aging, designed to improve bond strength, should be tested. Therefore, the aim of this study is to evaluate the effect of a 77 °C heat treatment of a silanized feldspathic ceramic on the microtensile bond strength (μTBS) with a resin cement before and after being aged by thermocycling. The null hypotheses are: (1) the heat treatment does not affect the bond strength; (2) the combination of the hydrofluoric-acid-etching treatment with heat treatment does not improve the bond strength; and (3) the thermocycling does not affect the reliability of the interfaces.

2. Materials and methods

2.1. Materials of study

The materials used in this study are described in Table 1.

2.2. Specimen preparation

Six blocks of a feldspathic ceramic for CAD/CAM (Vitablocks Mark II) with dimensions of 12 × 10 × 17 mm^3 were sectioned in a cutting machine (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA), resulting in 24 ceramic blocks (12 × 10 × 4 mm^3). All blocks were ground-finished for surface standardization with 600-grit wet silicon carbide papers (3M ESPE, St. Paul, MN, USA). The blocks were molded using a silicone impression material (Elite HD, Zhermack, Badia-Polesine, Rovigo, Italy), and the mold was used to the construction of the composite resin blocks (Z100, A1 shade) which were cemented onto the ceramic surfaces. The composite resin was incrementally applied to the mold (2 mm thickness for each increment), and each increment was light-cured for 40 s (XL 3000, 3M/ESPE; light intensity = 500 mW/cm^2) at a distance of 10 mm, until the total fulfillment of the mold. Light intensity was verified by radiometry so that it was no lower than 500 mW/cm^2. The composite resin blocks were made on the day of cementation. The final dimension of the composite resin blocks was similar to the ceramic blocks.

2.3. Surface treatment

The ceramic blocks were ultrasonically cleaned in distilled water for 5 min, and randomly divided into three groups, according to the surface treatment:

- Group AS – The ceramic surface was etched with 10% HF for 60 s, washed, and air-dried for 30 s. The silane coupling agent was applied by means of a microbrush (3M ESPE) for 60 s and gently dried with air spray for 15 s.
- Group S77 – The silane coupling agent was applied to the surface by means of a microbrush for 60 s and gently dried with air spray for 15 s. The silanized block was placed in a 77 °C preheated oven for 60 s.
- Group AS77 – The ceramic surface was etched with 10% HF for 60 s, washed, and air-dried for 30 s. The silane coupling agent was applied by means of a microbrush for 60 s and gently dried with air spray for 15 s. The silanized block was placed in a 77 °C preheated oven for 60 s.

2.4. Specimens cemetation

After the treatments, the blocks of resin composite were cemented onto the ceramic blocks by means of a resin cement (Variolink II), with a constant pressure of 750 g for 60 s. The excess cement was removed, and the set was light-cured for 40 s on each side. All sets were stored in distilled water at 37 °C for 24 h before the sticks obtaining.

2.5. Beams preparation

The ceramic/resin sets were embedded in chemically cured acrylic resin, positioned in a cutting machine (Isomet, Buehler), and sectioned at low speed with water cooling. Each set was longitudinally cut into a series of 1.0-mm-thick slices and then rotated 90° for a second sectioning. Approximately 68 beams with (cross-section, area of 1.0 mm^2) were obtained from each set. The edge sections (measuring approximately 1 mm) were discarded due to the possibility of excess or no cement at the interface,

Table 1
Brand name, material type, manufacturer and composition of the materials used in the study.

Brand name	Material type	Manufacture	Composition
Vitablocks Mark II	Feldspathic ceramic	VITA Zanhfabrik; Bad Säckingen, Germany	Feldspathic cristaline particles (SiO_2, Al_2O_3, Na_2O, K_2O) in glassy matrix
Monobond S	Silane coupling agent	Ivoclar Vivadent; Schaan, Leichtenstein	1% 3-methacryloxypropyl trimethoxysilane (3-MPS) ethanol/water-based solvent
Porcelain Conditioner	Acid etchant	Dentsply; Petropolis, Brasil	Hydrofuoric acid (HF) 10%
Variolink II	Resin Cement	Ivoclar Vivadent, Schaan, Leichtenstein	Monomer matrix: Bis-GMA, urethane, dimethacrylate and triethylene glycol dimethacrylate. Inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass and spheroid mixed oxide
Z100	Composite resin	3M ESPE; St Paul, MN, USA	Bis-GMA, TEGDMA, zirconia/silica (0,6 μm)