Does the Ultrasonic Cleaning Medium Affect the Adhesion of Resin Cement to Feldspathic Ceramic?

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Purpose: To evaluate the effect of different cleaning media on the adhesion of resin cement to feldspathic ceramic after etching.

Materials and Methods: The cementation surfaces of ceramic blocks (N = 20, n = 5 per group) were etched with 10% hydrofluoric acid (HF) gel for 20 s and rinsed for 60 s. They were then randomly assigned to 4 groups: G1: air-water spray+drying (control); G2: ultrasonic cleaning in distilled water for 4 min+drying; G3: ultrasonic cleaning in 99.5% acetone for 4 min+drying; G4: ultrasonic cleaning in 70% alcohol for 4 min+drying. The ceramic blocks were silanized and cemented (RelyX ARC) to the composite blocks. Subsequently, the microtensile bond strength test (μTBS) was performed. In addition, EDS analysis was made to assess the elemental composition of the conditioned and cleaned ceramic surfaces.

Results: A significantly higher mean μTBS was obtained when specimens had been ultrasonically cleaned in distilled water (G2: 18.8 ± 0.4 MPa) ($p < 0.05$) compared to other groups (G1: 16.6 ± 0.5; G3: 16.1 ± 0.9; G4: 15.8 ± 1.4) (one-way ANOVA). EDS analysis indicated the presence of F^- only in G1. Dissolved precipitates after HF etching were removed by ultrasonic cleaning.

Conclusion: Cleaning the HF-etched ceramic surface ultrasonically in distilled water is recommended, instead of rinsing it with air-water spray only.

Keywords: adhesion, cementation, dental materials, microtensile bond strength, ultrasonic cleaning.

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Adhesive cementation of feldspathic ceramics increases their clinical survival but requires a sequence of procedures. The first step is etching with hydrofluoric acid (HF), which selectively dissolves the glassy matrix, followed by silanization.[5] As it is a weak acid, hydrogen fluoride in water is not completely ionized. Its conjugate base, the fluoride ion F^-, can re-associate to form HF in solutions with low pH.[4] Strong bonds are formed between resin-based materials and dental ceramics that are etched with HF because the affinity of fluoride to silicon is higher than to oxygen: $4HF + SiO_2 \rightarrow SiF_4 + 2H_2O$.[4] However, HF etching leads to precipitates of organic debris or fluorosilicate salt. Elimination of such precipitates by air-water spray or in an ultrasonic cleaner is expected to increase the adhesion.[2] Moreover, removal of F^- is especially desirable due its hazardous effects.[4] Cleaning in an ultrasonic cleaner can be performed in distilled water,[1] acetone, or alcohol.[3]

The objectives of this study were to evaluate the effect of the ultrasonic cleaning medium on the adhesion of resin cement to feldspathic ceramic and to perform

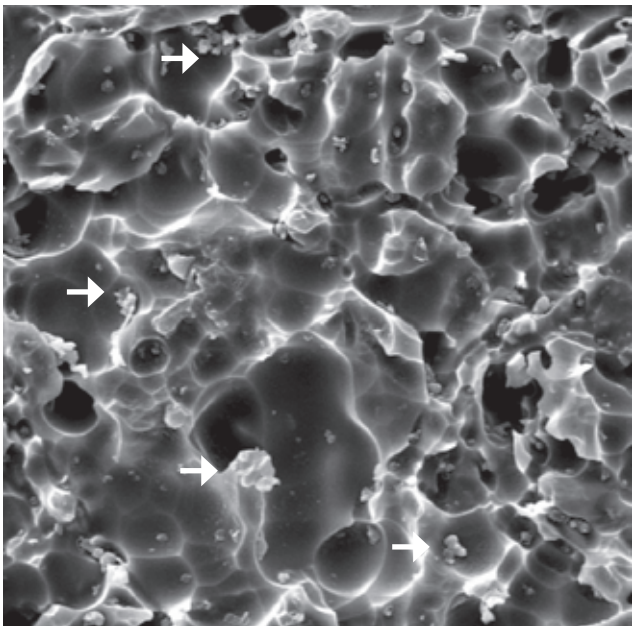

Fig 1a SEM image (2000X) of the HF-etched ceramic surfaces after air-water spraying. Note the precipitates after air-water spraying indicated by arrows.

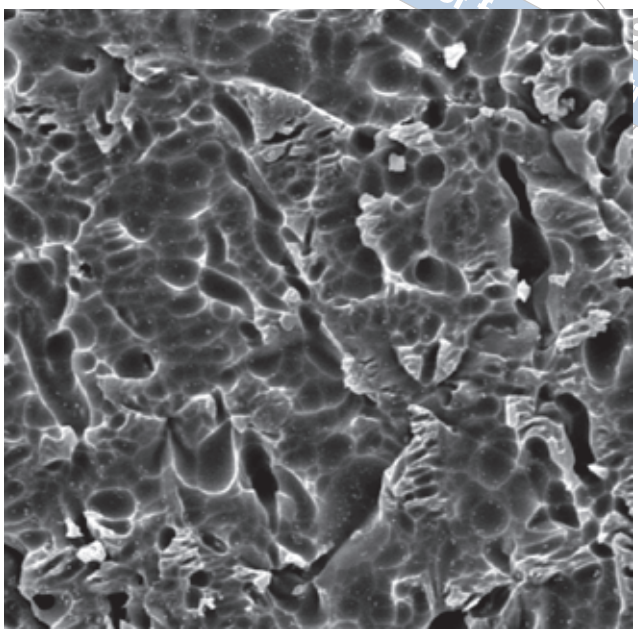

Fig 1b SEM image (2000X) of the HF-etched ceramic surfaces after ultrasonic cleaning in distilled water.

elemental analysis after HF etching and cleaning. The following hypotheses were tested: a) ultrasonic cleaning of the ceramic after HF etching yields higher bond strength than does cleaning with air-water spray, b) the ultrasonic cleaning medium does not influence bond strength.

MATERIALS AND METHODS

The cementation surfaces of ceramic blocks (N = 20, n = 5 per group) (Vita VM7, Vita Zahnfabrik; Bad Säckingen, Germany) (6 mm x 6 mm x 8 mm) were duplicated in resin composite (W3D Master, Wilcos do Brasil; Rio de Janeiro, Brazil). The cementation surfaces were wet ground with 1200-grit SiC paper and cleaned in an ultrasonic bath in distilled water for 5 min. After etching with 10% HF gel for 20 s and rinsing for 60 s with water spray, they were randomly assigned to 4 groups: G1: only air-water spray for 4 min+drying (control); G2: ultrasonic cleaning in distilled water for 4 min+drying; G3: ultrasonic cleaning in 99.5% acetone for 4 min+drying; G4: ultrasonic cleaning in 70% alcohol for 4 min+drying. The ceramic blocks were silanized (Porcelain Primer, Dentsply; Rio de Janeiro, Brazil) using a microbrush, cemented to the composite blocks with resin cement (RelyX ARC, 3M ESPE; St Paul, MN, USA) under constant pressure (750 g), and photopolymerized for 40 s per side (XL3000, 3M ESPE) at 600 mW/cm^2.

The cemented blocks were sectioned with a diamond disk under water to obtain microbars (bonding area: 0.8 mm^2). Each bar was attached to an adapted device and the microtensile bond strength test (μTBS) was performed in a universal testing machine (Emic DL-1000; São José dos Pinhais, Brazil) (1 mm/min). Failure types were analyzed after debonding using an optical microscope (MC 80 DX, Zeiss; Jena, Germany) and a scanning electron microscope (SEM) (JSM-T330A, JEOL; Tokyo, Japan).

Additional ceramic blocks were obtained (n = 4 per group). They were conditioned and cleaned as described above. The topographies of the ceramic blocks were analyzed using SEM, and elemental compositions of the ceramic surfaces were assessed using energy dispersive x-ray spectroscopy (EDS).

RESULTS

No pre-test failures occurred in any of the specimens. One-way ANOVA revealed that the mean μTBS values were significantly affected by the surface treatment ($p = 0.001$). Significantly higher mean μTBS values were obtained in G2 (18.8 ± 0.4 MPa) ($p < 0.05$), where specimens had been ultrasonically cleaned in distilled water, than in the other groups (G1: 16.6 ± 0.5; G3: 16.1 ± 0.9; G4: 15.8 ± 1.4).

All groups showed exclusively mixed failure types, where remnants of the resin cement were visible on the ceramic surface after debonding. SEM images showed porous surfaces of etched ceramic with apparent precipitates, confirmed by EDS analysis, when specimens had only been cleaned with air-water spray. After ultrasonic cleaning, the surfaces were free of these precipitates (Fig 1). Air-water spraying was not sufficient for removing F^-, but after ultrasonic cleaning in distilled water, F^- was not detected on ceramic surfaces (Fig 2).

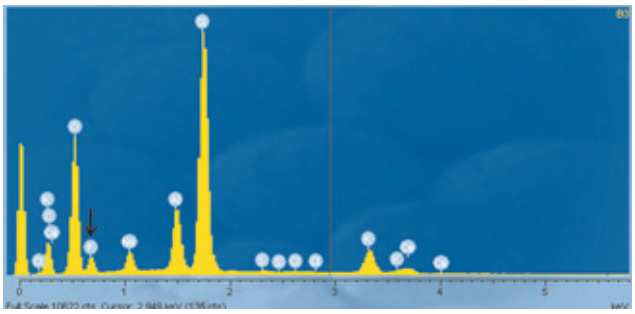

Fig 2a EDS analysis of the HF-etched ceramic surface after air-water spraying. Note the presence of F^- on the ceramic surface in the EDS analysis after water-spraying indicated by the arrow.

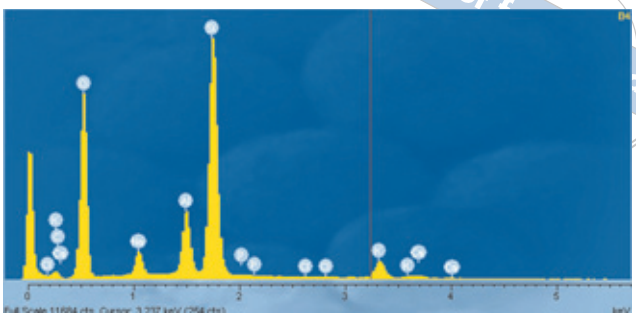

Fig 2b EDS analysis of the HF-etched ceramic surface after ultrasonic cleaning in distilled water. Note absence of F^- on the ceramic surface after ultrasonic cleaning in distilled water.

DISCUSSION

Since ultrasonic cleaning in acetone or alcohol presented bond strength values similar to those after cleaning with air-water spray, the first hypothesis was rejected. However, EDS measurements indicated that F^- was not removed completely with air-water spray, demonstrating the necessity of ultrasonic cleaning. Nevertheless, ultrasonic cleaning in distilled water presented significantly higher bond strength values than did ultrasonic cleaning in acetone or alcohol. Therefore, the second hypothesis was rejected. The non-polarity of acetone and alcohol could be responsible for their lower ability to dissolve the precipitates. Future studies should consider prolonged cleaning durations. The lack of cohesive failures in ceramic indicates less favorable adhesion of the resin cement. The bond strength results and failure types may change with other resin cements.

CONCLUSIONS

After etching feldspathic ceramic with HF gel, rinsing the ceramic surfaces with air-water spray did not completely remove F^- ions and precipitates, whereas ultrasonic cleaning in distilled water, acetone, or alcohol did. Ultrasonic cleaning of feldspathic ceramic after HF etching seems to be essential, and distilled water as the cleaning medium provided the highest bond strength between the resin cement and the ceramic tested.

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Clinical relevance: Hydrofluoric acid gel remnants after etching of feldspathic ceramic are best removed ultrasonically in distilled water.