Original Research
Dental Materials

The effect of hydrofluoric acid and resin cement formulation on the bond strength to lithium disilicate ceramic

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Abstract: To investigate how the hydrofluoric acid (HF) concentrations applied to a lithium disilicate glass-ceramic (EMX) affects the surface morphology and microtensile bond strength (μTBS) of ceramics to dentin, using light-cured resin cements with or without UDMA. Sixty-three EMX square ceramic blocks were etched for 20 seconds using different HF concentrations (1%, 5% and 10%) and luted to dentin using two types of resin cement combinations: BisGMA/TEGDMA and BisGMA/TEGDMA/UDMA (n = 10). Each bonded EMX-dentin block was sectioned to obtain 1 mm^2 sticks for μTBS evaluation. Half of the sticks were tested after 24 hours and the other half was assessed after 6 months of water storage. Data were statistically assessed using split-plot three-way ANOVA and multiple comparisons were performed using the Tukey's post hoc test ($\alpha = 0.05$). One EMX sample from each HF concentration was analyzed using field-emission scanning electron microscope (FE-SEM) to characterize the etching pattern. According to the FE-SEM images, increasing the concentration of HF from 1 to 5 and then to 10% led to increased removal of glassy matrix and greater exposure of lithium disilicate crystals. The 10% HF concentration yielded higher μTBS when compared to 1% for BisGMA/TEGDMA formulation ($p < 0.05$); whereas HF 1% and 5% showed similar μTBS values when compared to 10% HF for BisGMA/TEGDMA/UDMA resin matrix ($p > 0.05$) at both storage times. Water aging decreased the μTBS values ($p < 0.05$), except when 10% HF was associated with BisGMA/TEGDMA resin cement. Resin cement formulation and hydrofluoric acid concentrations can interfere with the immediate and long-term glass-ceramic bond strength to dentin.

Keywords: Hydrofluoric Acid; Resin Cements; Dentin; Electron Microscope Tomography.

Introduction

Due to their optimal mechanical/optical properties, chemical durability and survival rates,[1,2] dental glass-ceramics are one of the most adopted indirect restorative materials for reestablishing function, shape and esthetics of affected dentition. The lithium disilicate glass-ceramic is noteworthy among glass-ceramics due to its outstanding natural look-like,[3] translucency and high mechanical strength.[1,4]

As lithium disilicate glass-ceramic is suitable to be adhesively bonded to dental tissues, the bond between glass-ceramics and resin cements is one of the key factors for long-term clinical success.[5] Although hydrofluoric acid (HF) etching followed by silane application is recognized as the most widely accepted procedure before luting glass-ceramic with resin cements,[3,5,6,7,8] the ideal etching protocol is still not clear.[4] The manufacturer of IPS e.max Press (EMX) (Ivoclar Vivadent, Schaan, Liechtenstein), a pressable lithium disilicate glass-ceramic, recommends etching EMX with 4.8% HF for 20 seconds. On the other hand, *in vitro* studies and clinical case reports have demonstrated concentrations of up to 10%.[3,6,7,8]

The hazardous nature of HF[9] has led researchers[10,11] to assess the effects of HF concentrations lower than 5% applied at room temperature on EMX, which showed underwhelming bond strength results. Most *in vitro* studies[6,7,10,11,12,13,14,15] have focused on the bond strength of lithium disilicate to the ceramic-resin cement or resin composite interfaces..

Resin cements are responsible for mechanically/chemically bonding the glass ceramics to tooth. Those materials must have high mechanical properties, adequate bond strength to tooth tissues and structures, high resistance to dissolution and satisfactory bonding to non-retentive tooth preparations to withstand the constant incidence of tensile/oblique/compressive masticatory loads found in the oral environment.[16,17] Previous reports[8,18,19,20] assessed the bond strength of lithium disilicate ceramics etched with only one specific HF concentration and then luted to dentin using different chemical-physical setting modalities available for commercial resin cements. As the main components of resin matrix are methacrylate-based materials, such as Bis-GMA (bis-phenol A diglycidyl dimethacrylate), TEGDMA (tri-ethylene glycol dimethacrylate) and UDMA (urethane dimethacrylate),[21,22] their role on the bonding between EMX etched with different HF concentrations to dentin has not been investigated so far. As not all commercially available resin cements present UDMA (such as RelyX Veneer, 3M ESPE, St. Paul, MN, USA) and considering the distinct chemical and physical properties of UDMA compared to BisGMA and TEGDMA,[23,24,25] it becomes necessary to investigate the role of UDMA on the bonding characteristics to EMX as well.

Therefore, the aim of the present in *vitro* study is to assess the effect of three HF concentrations (1%, 5% and 10%) on the etching morphology and microtensile bond strength (μTBS) of lithium disilicate glass-ceramic luted to dentin using light curing resin cements with and without UDMA at immediately after preparation and after 6-month of water storage. The null hypotheses tested were: 1) Different HF concentrations would not affect the μTBS; 2) Resin cement matrices would not influence the μTBS; and 3) Water storage would not decrease μTBS.

Methodology

Ceramic blocks

Sixty-three square ceramic blocks (8 mm x 8 mm x 3 mm thick) were fabricated from IPS e.max Press ingots (Ivoclar Vivadent, Schaan, Liechtenstein, shade LTA2), according to the manufacturer`s instructions and as described in a previous study.[11] After divestment, the EMX blocks were wet-polished with 1000-, 2500- and 4000-grit silicone carbide abrasive papers (Buehler, Lake Buff, USA) to obtain a flat surface.

Hydrofluoric acid etching of IPS e.max Press

A person that was not involved in the study and blinded to the groups randomly divided the EMX blocks into 3 groups according to the hydrofluoric acid (HF) concentrations: 1%, 5% and 10% (Fórmula & Ação, São Paulo, Brazil) (n=21). The etching time was fixed at 20 seconds and, following etching, the HF was removed using air/water spray for 1 minute and the specimens were ultrasonically cleaned with deionized water for 20 minutes and air dried.

Field-emission scanning electron microscopy (FE-SEM) evaluation

One random etched EMX specimen was selected (in the same manner adopted for the groups distribution) from each group to characterize the resultant etching pattern. The etched EMX specimens were mounted on coded brass stubs, sputter coated with gold-palladium for 60 seconds at 45 mA (Denton Vacuum Desk II, Moorestown, USA) and submitted to FE-SEM analysis (FEI Quanta 200 Environmental Scanning Electron Microscope, Hillsboro, USA) at 20 kV. Images were obtained with a 3,038 × magnification and 10 μm scale bars.

Resin cement formulation

After HF etching, specimens from each group were randomly distributed into 2 subgroups according to the resin cement formulation (n=10). The chemical components of the resin matrix were mixed using the following materials: bis-phenol A dyglycidyl dimethacrylate (Bis-GMA; Esstech, Essington, USA); tri-ethylene glycol dimethacrylate (TEGDMA; Esstech, Essington, USA); and urethane dimethacrylate (UDMA; Esstech, Essington, USA). The type 1 resin cement matrix was formulated using Bis-GMA and TEGDMA in a 1:1 mass ratio. The type 2 resin cement had the addition of UDMA, with the final resin matrix composition presenting Big-GMA, TEGDMA and UDMA in a 5:2:3 mass ratio. All the resin cements components are disclosed in Table 1.

Photoinitiators were added to the resin matrix as follows: 0.8 wt% of a tertiary amine (EDMAB, ethyl 4-dimethylaminobenzoate; Avocado, Heysham, England), 0.2 wt% of dl-camphoroquinone (CQ, Polysciences Inc., Warrington, USA) and 0.1 wt% inhibitor (BHT, 2,6-di-tert-butyl-4-methylphenol; SigmaAldrich, St. Louis, USA). Barium borosilicate glass filler (Esstech, Essington, PA, USA) was mixed into the resin matrix using a mechanical mixer (DAC 150 Speed mixer, Flacktek, Landrum, USA) for 5 min at 2,500 rpm at a 1:1 mass ratio with the resin matrix. All the procedures were performed under yellow lights in order to prevent photoinitiator (CQ) degradation.

Bonding procedures

Dentin surface treatment

Sixty-five freshly extracted human third molars were obtained from the Oregon Health & Science University and stored in 0.5% chloramine at 4°C until use. The coronal third was removed to expose the mid-dentin portion, wet-polished with -600 SiC abrasive papers for 60 seconds to produce a smear layer, and rinsed. Dentin was further etched with 32% phosphoric acid (Scotchbond Universal Etchant, 3MESPE, St. Paul, USA) for 15 seconds and air-water sprayed for 30 seconds to remove the phosphoric acid. Later, excess of water was removed using a dry cotton-pellet to leave a moist dentin.

The primer of a three-step etch-and-rinse adhesive system (Scotchbond MultiPurpose, 3MESPE, St. Paul, USA) was applied to the dentin surface and air-dried for 15 seconds. A thin layer of the bonding agent (Scotchbond MP, 3MESPE) was also applied to the dentin and light cured for 10 seconds using an LED curing device (Valo, Ultradent Inc., South Jordan, USA), with an irradiance of 1,000 mW/cm^2.

IPS e.max press surface treatment

After HF etching, a silane coupling agent (RelyX Ceramic Primer, 3MESPE, St. Paul, USA) was applied onto all specimen surfaces and allowed to air dry for 1 minute, followed by air-heat drying (60°C ± 5) for 1 minute. A thin layer of a bonding agent (Scotchbond MP, 3MESPE) was applied to the ceramic surface for 10 seconds.

Luting the IPS e.max press to dentin

The resin cement was poured onto the dentin surface and the etched EMX surface was pressed against it under a vertical static load of 1 kilogram for 120 seconds. Light-activation was performed for 40 seconds at each of the EMX/dentin sides (four activations) and a final 60 seconds of light curing through the bulk of the ceramic. All bonded specimens were stored in deionized water for 24 hours at 37°C before trimming.

Table 1. Resin cements chemical composition.

Resin cement type	Chemical composition
Type 1	Organic matrix: BisGMA and TEGDMA (1:1 mass ratio). EDMAB (0.8 wt%), CQ (0.2 wt%), BHT (0.1 wt%) and barium borosilicate glass filler (1:1 mass ratio with the resin matrix)
Type 2	Organic matrix: BisGMA, TEGDMA and UDMA (5:2:3 mass ratio). EDMAB (0.8 wt%), CQ (0.2 wt%), BHT (0.1 wt%) and barium borosilicate glass filler (1:1 mass ratio with the resin matrix)

BisGMA bis-phenol A dyglycidyl dimethacrylate, TEGDMA tri-ethylene glycol dimethacrylate, UDMA urethane dimethacrylate, EDMAB ethyl 4-dimethylaminobenzoate, CQ dl-camphoroquinone, BHT 2,6-di-tert-butyl-4-methylphenol

Microtensile bond strength (μTBS) evaluation

Cuts perpendicular to the bonded interface were made using a water-cooled diamond blade (Dia. Wafer Blade, Esstech Corp., Enfiled, USA) in the 'X' and 'Y' directions using a precision sectioning machine (Accutom-5, Struers, Cleveland, USA) to obtain 1mm^2 EMX/dentin sticks. Each stick was fixed to the grips of a μTBS device using a cyanoacrylate adhesive (Zap CA superglue, Ontario, Canada) and the μTBS was determined using a universal testing machine (MTS Criterion, Model 42, Eden Prairie, USA) at 0.5 mm/min crosshead speed until failure. Half of the obtained EMX/dentin sticks were stored in deionized water for 24 hours before the μTBS, while the other half were stored in deionized water for 6 months for further bond strength evaluation (water was replaced every 15 days).

Failure analysis

The fractured specimens were observed using an optical microscopy at 30× and 100× magnifications and failure modes were classified as: adhesive, cohesive within ceramic, cohesive within dentin, and mixed, which involved ceramic, resin cement, adhesive interface and dentin. A representative sample from each failure mode was subjected to FE-SEM analysis. The fractured specimens were prepared as described in the topic "Field-emission scanning electron microscopy (FE-SEM) evaluation".

Statistical analysis

The experimental unit considered for μTBS test was the EMX/dentin blocks. The data of μTBS at immediate (24 hours) and after 6-month water-storage were submitted to split-plot three-way analysis of variance (hydrofluoric acid concentration × resin cement formulation × storage time) and multiple comparisons were performed using the Tukey's post hoc test ($\alpha = 0.05$).

Results

IPS e.max press etching pattern

The etching patterns of IPS e.max Press are represented in Figure 1. The HF concentrations of 1% and 5% (Figures 1A and 1B, respectively) resulted in

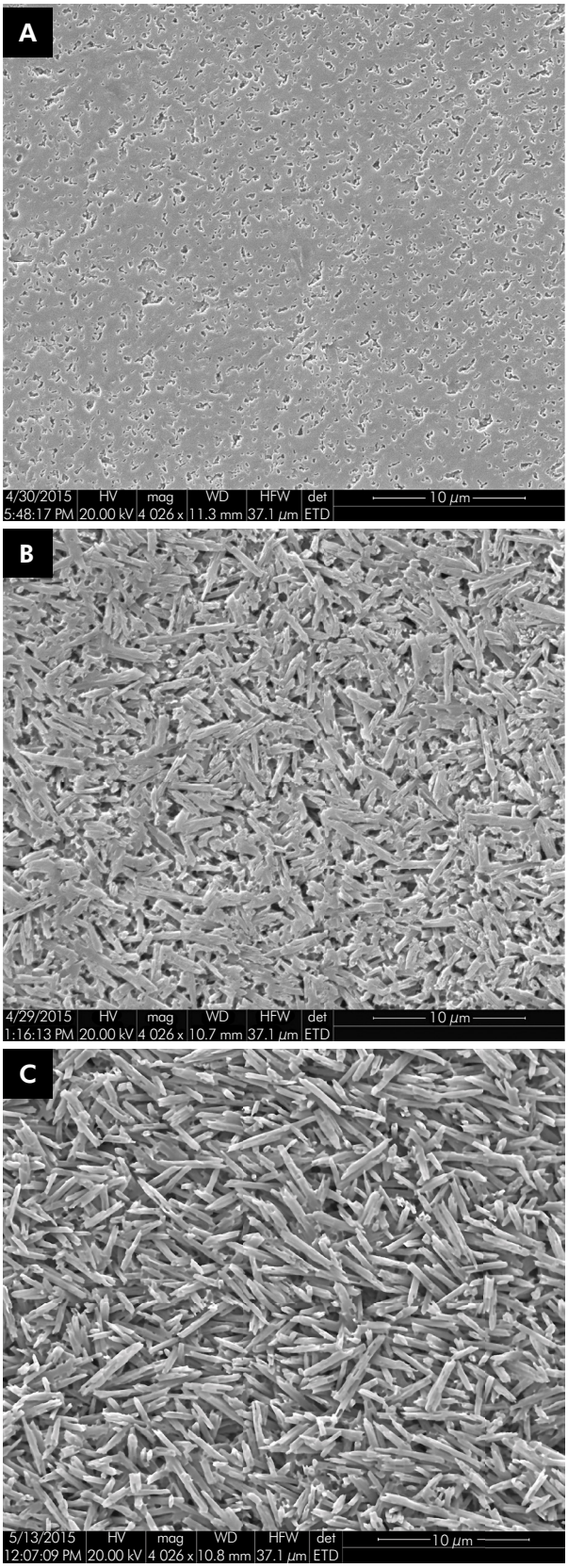

Figure 1. Resulted etching pattern on IPS e.max Press with 1% (A), 5% (B) and 10% (C) hydrofluoric acid applied for 20 seconds.

a superficial etching pattern when compared to 10%, as they removed a lower amount of vitreous phase associated with less lithium disilicate crystal exposure. The highest HF concentration (10%, Figure 1C) showed the greatest removal of vitreous phase and exposure of the lithium disilicate crystals.

Microtensile bond strength (μTBS)

The mean μTBS values are shown in Table 2. HF concentration × resin cement matrix (p = 0.1107), HF concentration × storage time (p=0.5375), resin cement matrix × storage time (p = 0.7587) and the triple interaction 'HF concentration × resin cement matrix × storage time' (p = 0.1877) did not show significant interactions between factors. Significant differences for resin cement matrix (p = 0.0312), HF concentration (p = 0.014) and storage time (p = 0.0002) were detected.

The different HF concentrations affected the μTBS values for resin cement type 1 (Table 2), with 10% HF showing a statistically higher μTBS value when compared to 1% at both storage times; whilst the HF concentrations revealed statistically similar μTBS values for resin cement type 2 at both storage times (Table 2). Both resin cement formulations showed decreased μTBS values after 6 months of water storage, except for 10% HF associated with resin cement type 1, which water storage did not decrease the μTBS value.

Failure modes analysis

A descriptive analysis of failure modes is shown in Table 3. At the 24 hour-storage time, a predominance of

Table 2. Mean microtensile bond strength values (MPa) ± standard deviation of type 1 and 2 resin cements. Within each resin cement type, means followed by different letters (uppercase letters in line and lowercase letters in column) indicate significant statistical differences according to Tukey`s test ($p < 0.05$).

Resin cement matrix	Hydrofluoric acid concentration	Storage time	
		24 h	6-month
BisGMA + TEGDMA (type 1)	1%	22.2 (± 5.7) Ab	19.6 (± 6.5) Bb
	5%	23.7 (± 2.9) Aab	19.8 (± 3.9) Bab
	10%	26.7 (± 2.9) Aa	26.2 (± 5.5) Aa
BisGMA + TEGDMA + UDMA (type 2)	1%	22.6 (± 2.1) Aa	19.8 (± 4) Ba
	5%	20.3 (± 3.5) Aa	19.4 (± 3.8) Ba
	10%	22.8 (± 2.5) Aa	20.6 (± 5.2) Ba

Table 3. Failure Modes Analysis (total number followed by % in parentheses) of the debonded specimens among groups.

Groups	Failure Modes			
	Adhesive	Cohesive within ceramic	Cohesive within dentin	Mixed
Storage time 24 h				
1% HF + Bis-GMA/TEGDMA	30 (43)	12 (17)	1 (2)	22 (38)
1% HF + Bis-GMA/TEGDMA/UDMA	29 (44)	15 (23)	3 (4)	19 (29)
5% HF + Bis-GMA/TEGDMA	16 (27)	10 (17)	15 (26)	17 (30)
5% HF + Bis-GMA/TEGDMA/UDMA	6 (10)	19 (31)	11 (17)	27 (42)
10% HF + Bis-GMA/TEGDMA	29 (45)	18 (28)	-----	18 (27)
10% HF + Bis-GMA/TEGDMA/UDMA	29 (47)	16 (25)	3 (5)	14 (23)
Storage time 6-month				
1% HF + Bis-GMA/TEGDMA	24 (35)	11 (16)	2 (3)	32 (46)
1% HF + Bis-GMA/TEGDMA/UDMA	19 (27)	13 (19)	2 (3)	35 (51)
5% HF + Bis-GMA/TEGDMA	26 (42)	8 (13)	3 (5)	25 (40)
5% HF + Bis-GMA/TEGDMA/UDMA	13 (20)	3 (5)	-----	47 (75)
10% HF + Bis-GMA/TEGDMA	17 (26)	12 (19)	3 (4)	33 (51)
10% HF + Bis-GMA/TEGDMA/UDMA	5 (9)	15 (28)	1 (2)	33 (61)

adhesive, cohesive within ceramic and mixed failures was verified. At the 6-month storage time, there was a prevalence of mixed failures for all tested groups and a decrease in the cohesive failures in dentin for the groups etched with 5% HF. Figure 2 shows the representative FE-SEM images of each failure mode obtained.

Discussion

The HF etching mechanism on lithium disilicate glass-ceramic basically consists of removing the glassy matrix due to the greater affinity of fluoride (present in the HF acid) reacting with silicon when compared to oxygen, which enables the ionized HF to dissolve the silicon-oxygen bonds (silanol) present in the glass ceramic.[9] Consequently, there is an exposure of lithium disilicate crystals that will be future sites for micromechanical interlocking for resin cements.[10,11,12] The FE-SEM images (Figure 1) depicted a superficial etching pattern for 1% HF and considerable removal of the glassy matrix and exposure of lithium disilicate crystals for 10% HF,

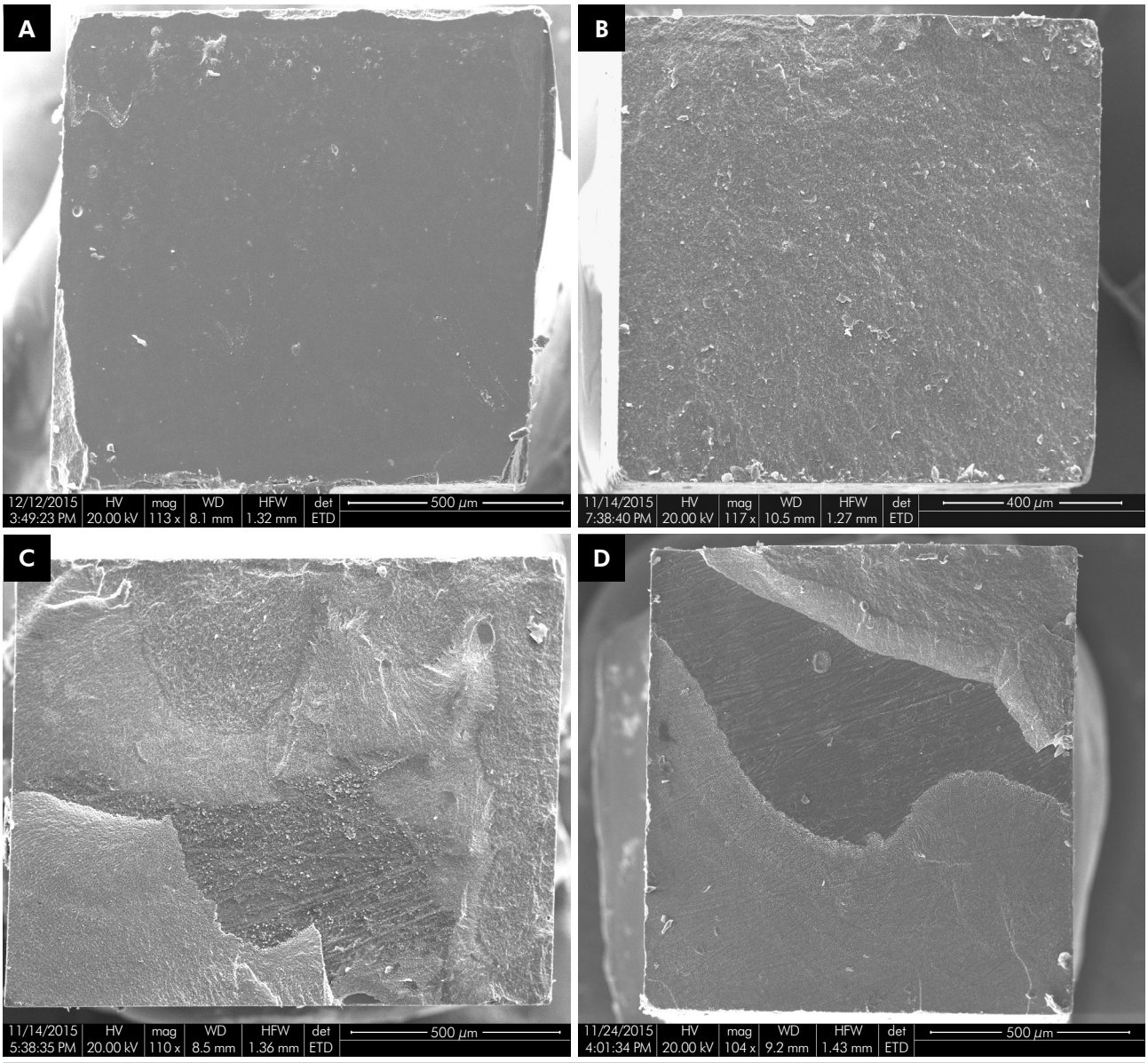

Figure 2. Representative FE-SEM of fractured EMX-dentin sticks. A) Adhesive failure; B) Cohesive failure within ceramic; C) Cohesive failure within dentin; D) mixed failure involving ceramic, adhesive interface and dentin.

while 5% HF performed in between the 1% and 10% HF. The reason for this is that low HF concentrations present lower amounts of ionized HF to react with the glassy matrix,[10,11,12] producing a more superficial etching pattern (Figure 1A). Previous *in vitro* studies have also verified increased removal of the glassy matrix with higher HF concentrations.[10,11,12,26]

The increased removal of the glassy matrix achieved with higher HF concentrations applied to lithium disilicate ceramics is directly related with higher bond strength values.[1,,11,12] However, in the present study, the different HF concentrations only affected the μTBS for the groups luted with resin cement type 1 (BisGMA/TEGDMA), a condition that was not confirmed for resin cement type 2 (BisGMA/TEGDMA/UDMA), with the three HF concentrations (1%, 5% and 10%) demonstrating statistically similar μTBS values. This partially denies our first and second hypothesis. It may be noted that the increased viscosity of the resin cement containing UDMA (BisGMA- molecular weight: 510.6 g/mol, viscosity: 1200 Pa s; TEGDMA- molecular weight: 286.3 g/mol, viscosity: 0.01 Pa s; UDMA- molecular weight: 470 g/mol, viscosity: 23.1 Pa s)[24] hindered the micromechanical interlocking to the etched surface of the lithium disilicate ceramic, thus decreasing/limiting the effect of EMX etching pattern on the bond strength values. On the other hand, a less viscous resin cement (better flowability), such as resin cement type 1, can better infiltrate/interact with the etched EMX surface irregularities, explaining the greater influence of HF on the μTBS results (such as 10% HF, Fig. 1C). Additionally, UDMA not only favors cross-linking[27] but also promotes higher flexural strength, elastic modulus and hardness[28] of composite materials. Even though the cement materials are definitely not composites, they share a lot of commonalities in composition. In fact, the main difference is often only in the filler content. All those combined factors may have led to increased mechanical properties of the resin cement containing UDMA[24,29] and counteracted the lower micromechanical interlocking to etched EMX with lower HF concentrations. Therefore, it may be speculated that the decreased mechanical entanglement of more viscous resin cements into the surface irregularities on EMX is somehow compensated for by the mechanical properties of the resin cement matrix and by the chemical bonding via a silane coupling agent.

Most of the evaluated groups showed decreased μTBS values after water storage, negating our third hypothesis. BisGMA, TEGDMA and UDMA are susceptible to hydrolytic cleavage due the presence of polar groups within their chemical compositions that binds, via hydrogen bonds, to water and plasticizes the polymer (hydroxyl groups (-OH) → BisGMA; urethane linkages (-NH=) → UDMA and ether linkages (-O-) → TEGDMA).[22,30] As water diffuses through the nanometer-sized pores within polymers, more plasticization and degradation can take place (reduction in mechanical properties),[22,30] jeopardizing the bond strength stability over time. Along with the resin matrix plasticization, water molecules tend to degrade the siloxane bonds (bond between silanol groups of silica surface and the silane coupling agent at the filler) via a hydrolysis reaction, causing filler debonding[31] and decreasing the mechanical properties of resin cements. Venz and Dickens[25] demonstrated that the hydrophilicity of monomers follows the descending order: TEGDMA > BisGMA > UDMA. Therefore, water uptake by BisGMA-based resins increases in direct proportion to the concentration of TEGDMA and decreases with the partial substitution of TEGDMA by UDMA.[32] Thus, it might be expected that resin cements with a higher TEGDMA content will present lower physical properties over time, promoting a greater negative influence on bond stability. Nevertheless, an improved and greater resin cement micromechanical interlocking into a more conditioned EMX surface (greater glassy matrix removal and exposure of lithium disilicate crystals) may have countered the resin matrix water degradation, as was seen in the group treated with 10% HF associated with resin cement type 1, at least up to 6 months of water storage.

Along with resin cement matrix plasticization, the dentin-adhesive interfaces are also susceptible to degradation. Two main mechanisms synergistically affect the bond stability to dentin: 1) the collagenolytic activity of host-derived dentin metalloproteinase at exposed collagen fibrils not infiltrated by adhesive resin; and, 2) adhesive polymer plasticization.[33,34,35] Although

the 3-step etch-and-rinse adhesive system (Scotchbond MultiPurpose) is recognized as the gold standard in terms of bonding stability over time,[36] Anchieta et al.[37] found decreased mechanical properties (elastic modulus) of the dentin-hybrid layer when Scotchbond MultiPurpose was evaluated after water storage. Additionally, the hybrid layer is mainly formed by a low molecular weight monomer (HEMA), which presents hydrophilic characteristics and lower mechanical properties.[38,39] All those combined factors play an important role on dentin-adhesive interface degradation, causing lower bond strength values after water aging.

The failure modes from the μTBS analysis showed similar incidences for adhesive, cohesive within ceramic, and mixed failures at the 24-hour period. However, the incidence of mixed failures increased after aging, which may be linked to polymer degradation. Adhesive failure does not always indicate poor bonding, but that the interfacial bond strength has been truly evaluated.[40] However, flaws within the dentin or ceramic (cracks or bubbles) and the association of non-uniform stress distribution during either bond strength testing or the trimming procedures to obtain sticks for μTBS analysis may have triggered the cohesive failures during tension.

It is valuable to note the μTBS results found for the association of HF concentrations and resin cement type 2 (BisGMA/TEGDMA/UDMA). Despite the decreased μTBS after water storage, it is possible to etch the EMX surface prior to luting using 1% HF acid as this group showed similar bond strength performance to 5% and 10% HF at both storage times. This is an interesting outcome considering the hazardous nature of HF because applying low HF concentrations would directly benefit dentists, dental personal, patients and prosthetic technicians. However, clinical studies are necessary to confirm the present study. Also, the present bond strength results contrasts with previous *in vitro* studies,[10,11] which reported lower bond strength results for 1% HF when compared to 5% and 10% HF, but those studies focused on the resin cement—lithium disilicate ceramic interface.

One important observation obtained from the present *in vitro* study is that the bond strength between lithium disilicate glass-ceramic to dentin involves the synergic role between two distinct interfaces: glass ceramic—resin cement and resin cement—dentin. According to previous studies,[6,26] the glass ceramic—resin cement interface appears to be more hydrolithicly stable than the resin materials—dentin interface.[37] These results recognize that the interface of resin materials to dentin is the weak link for the bond strength durability of glass ceramics to dentin. Therefore, dentists must execute state-of-the-art bonding using high quality dental materials in order to contribute to the survival rate of glass ceramic restorations.

Conclusion

If resin cement type 1 formulation is considered for luting glass-ceramics to dentin, 10% HF should be preferred, as it yielded higher and more stable bond strength values after water aging. On the contrary, as the bond strength results demonstrated that the resin cement containing UDMA (type 2) was not affected by different HF concentrations, 1% HF may be indicated over 5% and 10%, considering the hazardous nature of HF.

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