Bonding of self-etching adhesive/flowable composite combinations to enamel and dentin: a microtensile bond strength evaluation.

ELISA MAGNI[1], IVANA RADOVIC[2], IVANOVIC CONIGLIO[1], FEDERICA PAPACCHINI[3], CLAUDIA MAZZITELLI[1]

ABSTRACT

Objectives: The purpose of this investigation was to evaluate through the microtensile non-trimming technique the bond strengths of different self-etching adhesive/flowable composite combinations to ground enamel and coronal dentin.

Methods and Materials: Twelve sound extracted human molars were collected and divided into two groups (n=6) according to the bonding substrate (enamel or dentin). Three self-etching adhesive/flowable composite combinations were used: (1) Adper Prompt L-Pop/Filtek Supreme XT Flow (3M ESPE); (2) Futurabond NR/Grandio Flow (Voco); (3) Xeno III/X-Flow (Dentsply). In all groups composite build-ups were made using Tetric EvoCeram (Ivoclar-Vivadent). Bond strength was assessed with the microtensile non-trimming technique. Data were statistically analyzed using the Two-Way ANOVA with dental substrate and self-etching adhesive/flowable composite combination as factors. The Tukey test was used for post-hoc comparisons.

Results: The type of dental substrate was a significant factor affecting bond strength, with bond strengths achieved to dentin higher than to enamel ($p<0.001$). The type of self-etching adhesive/flowable composite combination ($p<0.001$) and the interaction between the factors ($p<0.05$) were also significant. Xeno III/X-Flow and Futurabond NR/Grandio Flow on dentin exhibited significantly higher bond strengths compared to all the other experimental groups.

Conclusions: The self-etching adhesive/flowable composite combinations investigated bonded better to dentin than to enamel. The bond strengths on enamel were comparable among the materials combinations. Xeno III/X-Flow and Futurabond NR/Grandio Flow achieved significantly higher bond strength on dentin than Adper Prompt L-Pop/Filtek Supreme XT Flow.

Clinical significance: The three self-etching adhesive/flowable composite combinations investigated can be used indifferently on enamel. For bonding to dentin Xeno III/X-Flow or Futurabond NR/Grandio Flow should be preferred.

Key words: self-etching adhesives, flowable composites, microtensile bond strength.

Short title: Microtensile bond strength of self-etching adhesive/flowable composite combinations

Introduction

The interface between a resin composite and a restored tooth is subjected to stresses that can lead to debonding and clinical failure of the restoration. The interfacial stress occurs even before the restored tooth is subjected to functional load, due to polymerization shrinkage of the composite.[1] A thick adhesive layer or a liner may act as an elastic intermediate layer between the tooth and the resin composite. These liners can resist polymerization shrinkage[2] and absorb the shock produced by occlusal loads.[3]

1 PhD student, Department of Dental Materials and Restorative Dentistry, University of Siena, Siena, Italy

2 Clinical Assistant, Clinic for Paediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade; PhD student, Department of Dental Materials and Restorative Dentistry, University of Siena, Siena, Italy

3 Department of Dental Materials and Restorative Dentistry, University of Siena, Siena, Italy

Corresponding author:
Elisa Magni, DDS, MSc, PhD student, Department of Dental Materials and Restorative dentistry, Policlinico "Le Scotte", University of Siena, Viale Bracci, Siena, Italy
Tel +390577233131 Fax +390577233117
E-mail: elisamagni@hotmail.com

Flowable composites have been suggested as cavity liners because of their low viscosity and subsequent good adaptability to cavity walls.[4] In vitro studies showed that a flowable composite layer under Tetric Ceram (Ivoclar-Vivadent) and Charisma (Heraeus Kulzer) is able to increase the flexural strength of these composites[5] and that a flowable liner preserves the integrity of the marginal enamel in class II cavities after fracture of the restoration.[6] The elastic and mechanical properties[7-11] of flowable composites allow these materials to create a stress relieving layer under a conventional composite restoration.[12] On the other hand, flowable composites have been reported to fail as filled adhesive layers when used in combination with self-etching primers, without the application of a hydrophobic adhesive resin due to their inability in hybridizing etched and primed dentin.[13] Therefore, the use of flowable composites in combination with dental adhesives is advisable.

Nowadays self-etching adhesives and in particular the so called 'all-in-one' adhesives are widely used in the dental practice. These products seem to combine the attractive simplicity of a single clinical step and reduced post-operative sensitivity[14] even though there is still no agreement among the authors about the latter.[15-19] These adhesives contain acidic monomers that enable etching of the dental hard tissues and promote the penetration of the adhesive resin into enamel and dentin.[20] SEM observations and micro-Raman spectroscopy demonstrated the ability of these adhesives of interacting with the dentinal substrate and forming resin tags and hybrid layers of minimal thickness.[21,22] Due to their hydrophilic nature these adhesives have been reported to present entrapment of water extending from the hybrid layer/adhesive and enamel/adhesive interfaces into the adhesive, forming, when observed under a TEM, characteristic features known as 'water trees'.[23,24] These water-filled channels within the adhesive layer may act as initiation sites for hydrolytic degradation of the bond over time[25] and may account for the water permeation through the polymerized adhesive which represents one of the mechanisms involved in the incompatibility between self-etching adhesives and chemical-cured composites.[26-29] An in vitro investigation suggests that the application of an intermediate layer of a light-cured hydrophobic resin between a self-etching primer and a chemical-cured cement could reduce the permeability to water of the adhesive enhancing the coupling of the materials.[30]

Little information is currently available on the bonding characteristics of intermediate flowable composites layers combined with self-etching adhesives under a conventional composite restoration. Thus, the aim of this investigation was to evaluate microtensile bond strengths of different self-etching adhesive/flowable composite combinations to ground enamel and coronal dentin. The null hypothesis tested is that neither the dental substrate, nor the materials combination influences the bond strength values.

Materials and Methods

Study design

Twelve sound human third molars extracted due to orthodontic reasons were collected and kept in 37°C saline solution (0.9% sodium chloride in water) for no longer than one month before being used in the experiment. The teeth were randomly divided into two groups (n=6) according to the bonding substrate: enamel (E) and dentin (D). Within every group three subgroups (n=2) were randomly formed, and each was randomly assigned to one of the tested self-etching adhesive/flowable composite combinations. The experimental subgroups were as follows:

- E(1): Adper Prompt L-Pop + Filtek Supreme XT Flow (3M ESPE, Seefeld, Germany) on enamel
- E(2): Futurabond NR + Grandio Flow (Voco, Cuxhaven, Germany) on enamel
- E(3): Xeno III + X-Flow (Dentsply Caulk, Milford, DE, USA) on enamel
- D(1): Adper Prompt L-Pop + Filtek Supreme XT Flow (3M ESPE) on dentin
- D(2): Futurabond NR + Grandio Flow (Voco) on dentin
- D(3): Xeno III + X-Flow (Dentsply) on dentin

Table 1 reports the chemical compositions, batch numbers and application modes of all the materials used.

Microtensile bond strength testing

In order to assess bond strength on ground enamel, the buccal aspect of the tooth was flattened using abrasive paper without dentin exposure, under continuous water cooling. For evaluating bond strength on dentin, mid-coronal dentin substrate was exposed by cutting off the overlying tooth substance with a water-cooled diamond blade (Isomet, Buehler, Lake Bluff, IL, USA). A clinically relevant smear layer was created both on enamel and dentin by grinding the substrate with 180-grit silicon carbide paper under water cooling.[31]

The self-etching adhesive/flowable composites combinations were applied on the substrate of interest as recommended by the manufacturers (Table 1). A 5x5x5 mm composite block (Tetric EvoCeram, Ivoclar-Vivadent, Schaan, Liechtenstein) was then built on the substrate following the incremental technique. Each 1 mm-thick composite layer was individually cured for 40 seconds. In order to ensure the adequate polymerization of each added layer, the light tip was positioned as close as possible to the composite surface. The same halogen curing light (600 mW/cm^2 output; VIP, Bisco, Schaumburg, IL, USA) was used to cure all the materials used in the study.

After the specimens had been stored in 37°C saline solution for 24 hours each tooth was secured with sticky wax on an acrylic resin cylinder that was mounted on a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Using a water-cooled diamond blade (Isomet, Buehler, Lake Bluff, IL, USA), a series of 0.9 mm-thick sections was made perpendicular to the adhesive interface. These sections were further sectioned to produce sticks with adhesive area of approximately 0.8 mm^2, according to the non-trimming version of microtensile technique.[32] Each stick consisted of tooth substrate in half of its length and of composite build-up in the other half. The combination of self-etching adhesive and flowable composite of interest formed the adhesive interface between the two halves.

The exact dimensions of each stick's adhesive area were measured using a digital caliper (Orteam s.r.l., Milano, Italy) to the nearest 0.01mm. The specimens were glued (Super Attak Gel, Henkel Loctite Adesivi S.r.l., Milano, Italy) to the two free sliding components of a jig, which was mounted on a universal testing machine (Triax 50, Controls S.P.A., Milano, Italy) and loaded in tension at a crosshead speed of 0.5 mm/min until

Table 1: Composition, batch numbers and application mode of the materials used in the study.

Material	Composition	Application
Adper Prompt L-Pop (3M ESPE) Batch no. 237474	Liquid 1: methacrylated phosphoric esters, Bis-GMA, CQ-based initiators, stabilizers Liquid 2: water, HEMA, polyalkenoic acid, stabilizers	Mix Liquid 1 and Liquid 2. Brush mixture onto dental substrate for 15 s. Gently air dry. Apply a second coat and gently air dry. Light cure for 10 s.
Filtek Supreme XT Flow (3M ESPE) Batch no. 20050817	Matrix: Bis-GMA, TEGDMA, Bis-EMA. Filler: aggregated zirconia/silica fillers (5-20nm; 55vol%)	Apply a thin layer. Light-cure for 20 s.
Futurabond NR (Voco) Batch no. 611518	Liquid A: Polyfunctional adhesive monomers (Methacroyl-Phosphorus-Acid-Ester, Methacroyl-Carbon-Acid-Ester), Dimethacrylates, Functionalized SiO2-nano-particles, Initiators. Liquid B: Ethanol, Water, Hydrophilic adhesive monomers, Fluorides	Mix Liquid A and Liquid B for 5 s. Apply mixture to tooth and massage for 20 s. Air dry for 5 s. Light cure for 10 s.
Grandio Flow (Voco) Batch no. 621020	Matrix: Bis-GMA, TEGDMA, HEDMA Fillers (65.6 vol%)	Apply a thin layer. Light cure for 20 sec.
Xeno III (Dentsply) Batch no. 0409000979	Liquid A: HEMA, purified water, ethanol, BHT, highly dispersed silicon dioxide Liquid B: Pyro-EMA, PEM-F, UDMA, BHT, CQ, ethyl-4-dimethylaminobenzoate	Mix Liquid A and Liquid B (1:1) for 5 sec. Apply for 20 sec.gently air dry for at least 2 sec. Light cure for 10 sec.
X-Flow (Dentsply) Batch no. 0512000880	Strontium alumino sodium fluoro phosphor silicate glass, di- and multifunctional acrylate and dimethacrylate (DGDMA), highly dispersed silicon dioxide, UV stabiliser, ethyl-4-dimethylaminobenzoate, CQ, BHT, iron pigments, titanium dioxide.	Dispense a thin layer. Light cure for 20 sec.
Tetric EvoCeram (Ivoclar-Vivadent) Batch no. H34327	Matrix: dimethacrylates (17-18% weight) Fillers: barium glass, ytterbium trifluoride, mixed oxide and prepolymer (82-83% weight), Additives, catalysts, stabilizers and pigments (<1% weight)	Apply in layers of max.2 mm. Light cure each increment for 40 sec.

Abbreviations - BHT: butylated hydroxyl toluene; Bis-EMA: ethoxylated bisphenol A-dimethacrylate; Bis-GMA: bisphenol A-diglycidylmethacrylate; CQ: camphorquinone; HEDMA: 2-hydroxyethyl dimethacrylate; HEMA: 2-hydroxyethyl methacrylate PEM-F: mono fluoro phosphazene modified methacrylate; Pyro-EMA: phosphoric acid modified methacrylate; TEGDMA: triethylene glycol dimethacrylate;UDMA: urethane dimethacrylate.

failure occurred. The load at failure was recorded in Newtons (N). Bond strength was calculated in MegaPascals (MPa) by dividing the load at failure by the cross-sectional area of each stick expressed in square millimeters (mm^2). The failures that occurred during the cutting or gluing procedures were recorded as pre-testing failures. For these sticks, bond strengths of 0 MPa were recorded and included in the statistical analysis. All the fractured specimens were sputtered-coated with gold (Polaron Range SC7620; Quorum Technology, Newhaven, UK) and observed under a scanning electron microscope (JSM 6060 LV, JEOL, Tokyo, Japan) in order to asses the failure mode. Failures were classified as cohesive (within the dental substrate, the adhesive layer, the flowable composite or the restorative composite), adhesive (at the dental substrate/adhesive interface, adhesive/flowable composite interface, flowable composite/restorative composite interface) and mixed (a combination of the two modes in the same interface).

Statistical analysis of the microtensile bond strength data

Having checked that the bond strength was not affected by the tooth of origin (regression analysis), the normality of data distribution (Kolmogorov-Smirnov test) and the homogeneity of the group variances (Levene's test) were verified. The Two-Way Analysis of Variance was applied with bond strength as the dependent variable and dental substrate and type of self-

Table 2: Means and standard deviations of bond strength values (MPa) in all the experimental groups. Different superscript letters indicate statistically significant differences.

Substrate	Materials combination	Bond strength [SD]	Bond strength [SD] pre-testing failures excluded
Enamel	Adper Prompt L-Pop + Filtek Supreme XT Flow	$14.82[12.05]^{B}$	20.75[8.69]
	Futurabond NR + Grandio Flow	$16.95[15.56]^{B}$	24.09[12.98]
	Xeno III + X-Flow	$17.68[13.97]^{B}$	22.44[11.78]
Dentin	Adper Prompt L-Pop + Filtek Supreme XT Flow	$17.72[12.72]^{B}$	23.04[9.17]
	Futurabond NR + Grandio Flow	$36.56[17.76]^{A}$	39.45[15.25]
	Xeno III + X-Flow	$31.40[14.70]^{A}$	36.23[8.29]

etching adhesive/flowable composite combination as factors. The Tukey test was applied for post-hoc comparisons where needed. In all the analyses the level of significance was set at $\alpha = 0.05$ and calculations were handled by the SPSS 11.0 software (SPSS Inc., Chicago, IL, USA).

Results

The results of microtensile bond strength testing for all the experimental groups are summarized in Table 2 and Figure 1. Microtensile bond strenghts resulting from the exclusion of pre-testing failures are also reported (Table 2). Failure modes are shown in Table 3. Representative SEM micrographs of fractured specimens are also presented (Figs. 2-4).

The Two-Way Analysis of Variance showed that the microtensile bond strength was significantly affected by the dental substrate ($p<0.001$) with higher bond strengths achieved to dentin than to enamel. The self-etching adhesive/flowable composite combination was also a significant factor ($p<0.001$) with higher bond strengths obtained with Futurabond NR + Grandio Flow and Xeno III + X-Flow than with Adper Prompt L-Pop + Filtek Supreme XT Flow. The interaction between the two factors was significant ($p<0.05$).

No statistically significant differences in bond strength to enamel were detected among the investigated self-etching adhesive/flowable composite combinations. Adper Prompt L-Pop + Filtek Supreme XT Flow achieved a significantly lower bond strength to dentin than the other two materials combinations and this bond strength value did not significantly differ from the bond strengths to enamel achieved by all the materials combinations investigated. The bond strengths to dentin of Futurabond NR + Grandio Flow and Xeno III + X-Flow were significantly higher than those of all the other experimental groups and no significant difference was found between them.

Discussion

The statistical analysis showed that the bond strength to enamel and dentin of the investigated self-etching adhesive/flowable composite combinations is influenced by

Table 3: Failure distribution in the experimental groups.

Substrate	Materials combination	Failure			
		Adhesive	Cohesive	Mixed	Pre-testing
Enamel	Adper Prompt L-Pop + Filtek Supreme XT Flow	-	3(C), 1(S)	11(F)	6
	Futurabond NR + Grandio Flow	-	6(C), 1(S)	12(F)	8
	Xeno III + X-Flow	-	7(C), 3(S)	16(F)	6
Dentin	Adper Prompt L-Pop + Filtek Supreme XT Flow	-	6(C), 2(S)	12(F)	6
	Futurabond NR + Grandio Flow	-	22(C)	16(F)	3
	Xeno III + X-Flow	-	10(C), 1(S)	15(F)	4

C: cohesive failure within the restorative composite; S: cohesive failure within the dental substrate; F: mixed failure involving the flowable composite layer.

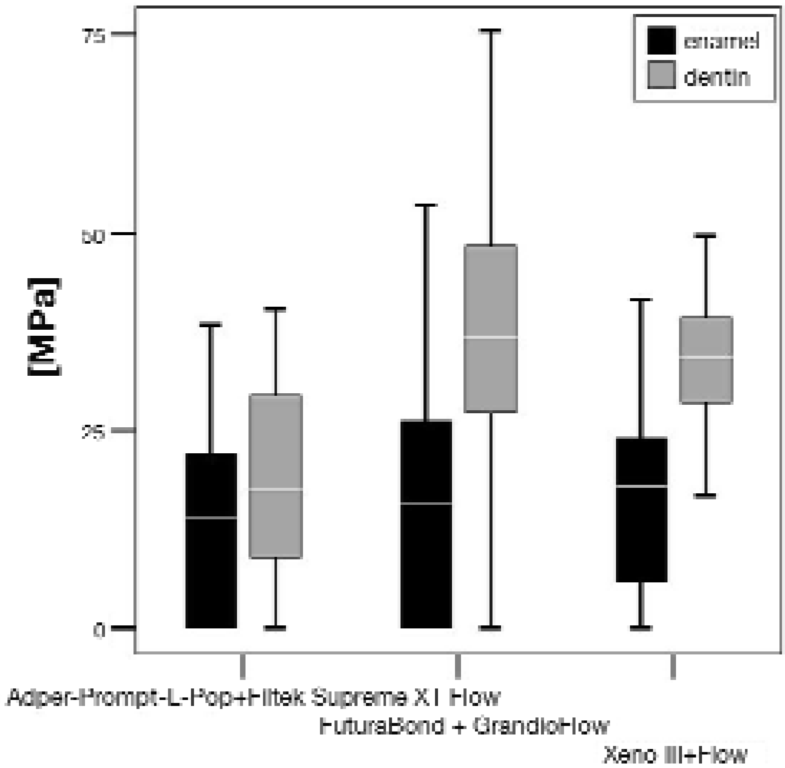

Figure 1: Microtensile bond strengths according to the dental substrate and the self-etching adhesive/flowable composite combination. The length of each box represents the interquartile range of microtensile bond strength data in the experimental groups

both the dental substrates and the material combinations. Thus the null hypothesis tested has to be rejected.

According to the results of the present study the two out of three combinations of an 'all-in-one' self-etching adhesive with a flowable composite of the same manufacturer achieved significantly higher bond strengths to dentin than to enamel. In a recent study Sundfeld et al.[33] evaluated the penetration of a self-etching adhesive into enamel through a light microscope and found the formation of short and non-uniform resin tags.[33] The lack of thick hybrid layer and frank resin tags formation in enamel with 'all-in-one' adhesives has been demonstrated also with SEM and TEM observations.[34] Conversely, the ability of self-etching adhesives to hybridize dentin and form resin tags in dentinal tubules has been showed with SEM and micro-Raman spectroscopy technique[21], even if these bonding agents are not always able to etch and infiltrate to the same extension in dentin.[35] This different behaviour of self-etching adhesives on enamel and dentin could explain the higher bond strengths achieved to dentin in the present investigation, in which 'all-in-one' adhesives were used in combination with flowable composites.

The Two-Way Analysis of Variance revealed that the materials combination was a significant factor affecting bond strength. According to the results of this study Adper Prompt L-Pop + Filtek Supreme XT Flow resulted in inferior performance than the other two materials combinations investigated. Moreover, the bond strength to dentin of Adper Prompt L-Pop + Filtek Supreme XT Flow was significantly lower than the dentin bond strengths of Futurabond NR + Grandio Flow and Xeno III + X-Flow. In the study of Sadek et al.[36] the mean microtensile bond strength to dentin of Adper Prompt L-Pop was 30.5 MPa and was comparable to the bond strength achieved by Xeno III and other self-etching adhesives. This finding is not consistent with the results of the present study, in which the bond strength to dentin of Adper Prompt L-Pop + Filtek Supreme XT Flow was only 17.72 MPa. However, in the study of Sadek et al.[36] the pre-testing failures were not included in the statistical analysis as zero bond strength values and this may account for the higher bond strength achieved and for the lack of statistically significant differences with the other self-etching adhesives tested. TEM investigations have demonstrated nanoleakage to occur within Adper Prompt L-Pop and to extend beneath the resin composite layer.[35] It may be speculated that such a weak adhesive layer could account for the low bond strengths achieved with the Adper Prompt L-Pop + Filtek Supreme XT Flow combination. Moreover, the concentration of acidic monomers in Adper Prompt L-Pop, leading to a low pH value, could be responsible for an extensive dissolution of the dentin surface, with a subsequent reduction of hydroxyapatite crystals within the hybrid layer available for a chemical interaction with the phosphate groups of the adhesive's functional monomers.[37] It could be speculated that a milder effect on dentin exerted by Futurabond NR, due to the different chemical composition and the different degree of acidity, left a substantial number of hydroxyapatite crystals, increasing the chemical bond between the adhesive and the dental substrate, and this could have accounted for its higher bond strengths to dentin. The different interaction with dental substrate exerted by self-etching adhesives with different degree of acidity was reported by De Munck et al.[38] as a possible explanation for the absence of differences in terms of microtensile bond strength to ground enamel between self-etching adhesives with different degree

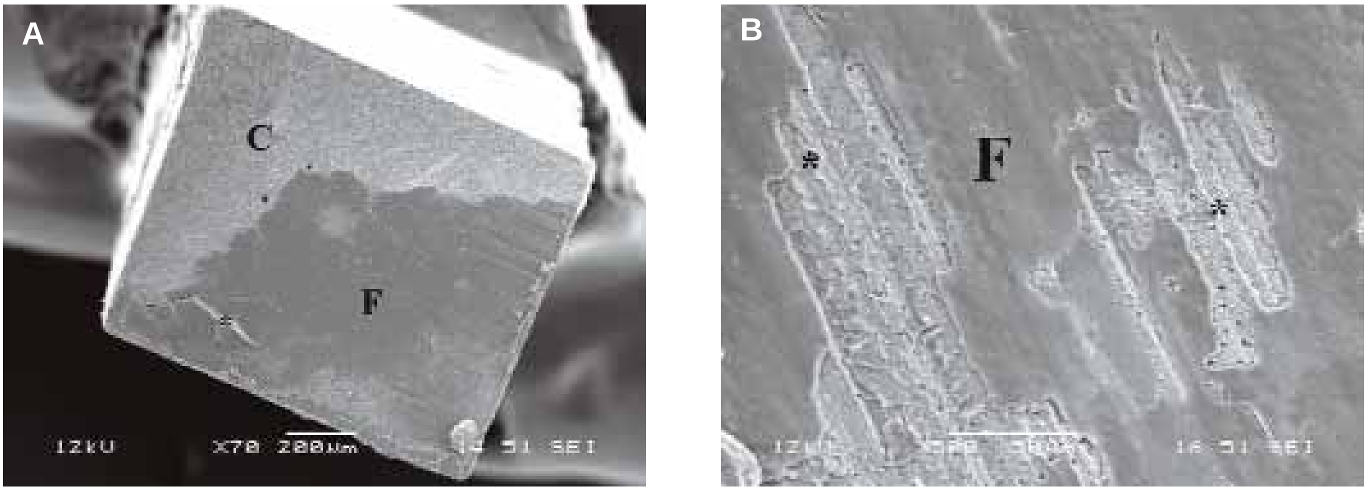

Figure 2: Representative SEM micrographs of fractured specimens of Adper Prompt L-Pop + Filtek Supreme XT Flow on dentin. (A) Mixed failure at the flowable composite [F]/restorative composite [C] interface. The failure site within the flowable composite is indicated by the asterisk (70x, bar = 200 µm). (B) Failure at the adhesive/flowable composite [F] interface. The partially exposed dentin surface appears still covered by the adhesive layer (asterisks) (500x, bar = 50 µm).

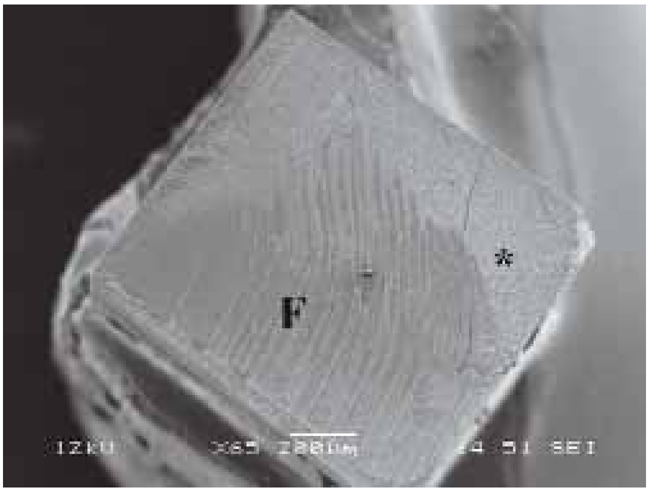

Figure 3: Mixed failure at the flowable composite [F]/restorative composite interface of a microtensile stick of Xeno III + X-Flow on enamel. The restorative composite build-up is indicated by the asterisk (65x, bar = 200 µm).

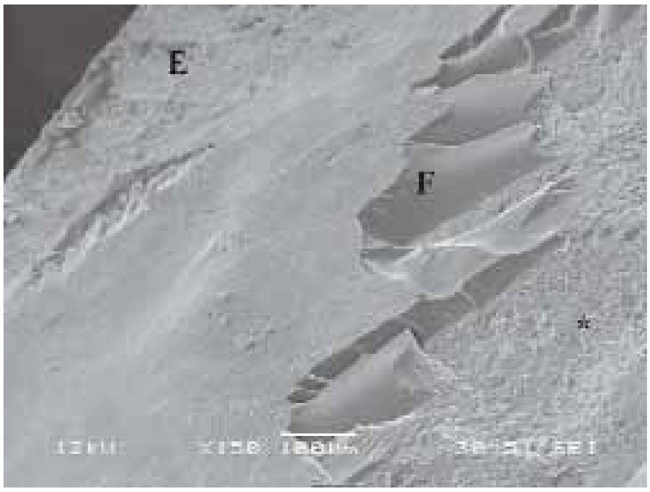

Figure 4: Representative SEM micrographs of a fractured stick of Futurabond NR + Grandio Flow on enamel. The flowable composite [F] did not detach completely from the enamel surface [E], that appears partially covered by the adhesive (asterisk) (150x, bar = 100 µm).

of acidity. The authors speculated that the chemical interaction of milder adhesives with dental substrate compensates their inferior ability of achieving micromechanical interlocking on enamel, resulting in microtensile bond strengths comparable to those of more acidic adhesives.[38] This finding is consistent with the results of the present study, where no significant differences in bond strength to enamel were detected among the three adhesives tested. Despite being a relatively 'strong' adhesive, Xeno III exhibited significantly higher bond strength to dentin compared to Adper Prompt L-Pop, and this could be explained by the presence of the monomers Pyro-EMA and PEM-F in its chemical composition, that could have contributed to the etching and adhesive function, leading to a superior stability of the dentin bond.

The microtensile specimens tested in the present study exhibited mostly mixed failures involving the flowable composites layers. Interestingly, the flowable composites did not tend to detach completely from the dental substrate (Figs. 2-4). This finding is in agreement with a study of Özgünaltay et al.[6] in which the fracture resistance of class II composite restorations was investigated. The authors reported that when a flowable composite was used as cavity liner the fractures did not involve the tooth structure, but some flowable material remained on the axial wall of the preparation after testing.[6] Since it was reported that the tensile strength to dentin correlates with tensile strength, flexural strength and Young's modulus of resin composites[39] but it correlates only with ultimate microtensile strength of self-etching adhesives[40], it may be speculated that the bond strengths measured in the present study could have been affected by the mechanical properties of the tested flowable composites.

It may be questioned that the addition of an intermediate flowable composite layer between a conventional restorative composite and an 'all-in-one' self-etching adhesive could make the clinical procedure more time-consuming, reducing the advantage of using a simplified adhesive, especially if the two materials are cured separately. Since the simultaneous curing of an adhesive and a flowable composite has been shown to increase the microleakage at dentin margins of class V restorations[41], in the present study a separate curing of the two materials was performed and this procedure should be advisable also in the clinical practice.

No total-etch adhesive/flowable composite combination was used as control in the present investigation and this could be considered as a weakness of the study design. However, it was not the objective of this study to assess whether the bond strength to enamel and dentin of self-etching adhesives were comparable to that of total-etch. Conversely the study aimed only to compare the bonding properties of three different self-etching adhesive/flowable composites combinations.

In the present study the microtensile bond strength to enamel and dentin of the three tested adhesives was evaluated after 24 hours storage in saline solution. The presence of water-filled areas within the hybrid layers formed by single-step self-etching adhesives[24,26] may arise some concerns regarding the stability of the bond achieved by these adhesives over time, since sites of incomplete water removal may contribute to hydrolytic degradation of the bonded interfaces.[23] Some in vitro studies evaluated the bond strength of single-step self-etching adhesives to enamel[42,43] and dentin[43-45] after water aging,[44,45] thermocycling[43] or cyclic loading.[42] Most of the studies showed a tendency of single-step self-etching adhesives to perform worse than etch-and-rinse adhesives[42,44,45] after aging. Also the marginal quality of composite restorations bonded with different adhesives systems after thermo-mechanical loading was investigated, showing a better performance of etch-and-rinse systems compared to all-in-one self-etching adhesives.[34] Conversely, a clinical study that compared the effectiveness of all-in-one self-etch and etch-and-rinse adhesive used to bond cervical restorations revealed no significant difference between the two adhesives over an 18-months period, apart a faster but acceptable marginal degradation for the self-etch adhesive.[46] Due to the limitations of in vitro studies and to the lack of long term clinical investigations on the durability of the bond achieved by contemporary all-in-one adhesives, an estimation of the longevity of the adhesive-dentin and adhesive-enamel bonds established by these adhesives is not possible yet. Thus, further investigations should be encouraged in order to address this issue.

Conclusions

Within the limits of the present investigation it may be concluded that both materials combination and dental substrate influence the microtensile bond strength of self-etching adhesive/flowable composite combinations to enamel and dentin. The three materials combinations investigated in this study exhibited comparable bond strengths to enamel. The application of Adper Prompt L-Pop + Filtek Supreme XT Flow resulted in inferior bond strength to dentin than the application of Futurabond NR + Grandio Flow and Xeno III + X-Flow.

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