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ORIGINAL ARTICLE

Shear bond strength and interface analysis between a resin composite and a recent high-viscous glass ionomer cement bonded with various adhesive systems

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Abstract

Objective This study investigated the shear bond strength (SBS) and interface between a resin composite and a new high-viscous glass ionomer cement (HV-GIC), a HV-GIC, a resin-modified glass ionomer cement (RM-GIC), a bulk-fill flowable composite, and a regular flowable composite bonded with various adhesive systems.

Methods and materials A resin composite (Filtek Z350) was bonded to a new HV-GIC (EQUIA Forte Fil) using various adhesive systems, including a universal adhesive in self-etch and etch-and-rinse mode (Scotchbond Universal), a two-step etch-and-rinse adhesive (Scotchbond 1-XT), a one-step self-etch adhesive (Optibond All-in-one) tested also after silane application (Monobond Plus), and a coating material (EQUIA Forte Coat). The resin composite was also bonded to a HV-GIC (Fuji IX GP), a RM-GIC (Fuji II LC), a bulk-fill flowable composite (SDR), and a regular flowable composite (Tetric Evo Flow) with the universal adhesive in self-etch mode (Scotchbond Universal). Two-way ANOVA followed by Dunnett's post hoc test was used to investigate the difference in SBS. Failures were analyzed by chi-square test. Bonding interfaces were examined by environmental scanning electron microscopy (E-SEM).

Results SBS to EQUIA Forte Fil was significantly lower with Scotchbond 1-XT than with all other adhesive systems. By using Scotchbond Universal with the self-etch technique, the SBS to EQUIA Forte Fil was significantly higher than the SBS to Fuji IX GP and significantly lower than the SBS to Fuji II LC, SDR, and Tetric Evo Flow. E-SEM images showed an intimate contact at all interfaces examined.

Conclusion EQUIA Forte Fil showed satisfactory SBS and interfaces with all adhesives tested.

Clinical relevance Bonding between the resin composite and HV-GIC can be achieved using a universal adhesive in self-etch mode, an easy-to-use adhesive system.

Keywords High-viscous glass ionomer cement · Universal adhesive · Sandwich technique · Proximal box elevation · Shear bond strength · Environmental scanning electron microscopy

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Introduction

Laminate restorations have been proposed to restore deep proximal cavities extending beyond the cementoenamel junction and thereby avoid surgical crown lengthening. Two techniques have been described to facilitate direct or indirect restorations [1–3].

The first is the "sandwich technique," during which a resin-modified glass ionomer cement (RM-GIC) is applied at the bottom of the cavity and then covered with a direct resin composite. This technique benefits from fluoride release, moisture tolerance, and the intrinsic adhesion of RM-GIC to dental tissues. These characteristics are particularly advantageous close to the gingival tissue, combined with the esthetics and mechanical properties of resin composites [4, 5].

The second is the "proximal box elevation," also known as "deep/cervical margin elevation/relocation." Here, one or more layers of a base material are applied to raise the cavity outlined and thus facilitate the next steps for indirect restoration (impression-taking and bonding) [6]. Several classes of materials are used as a base [7]. There is no consensus about the best base material in the literature. Resin composites have shown the best mechanical properties in terms of elastic modulus, flexural strength, hardness, and resistance to wear [8], but their sticky texture does not allow for the intimate adaptation to cavity walls, which leads to incomplete marginal sealing. Hence, regular flowable composites have been proposed [9, 10]. Some studies have reported that these composites have a better marginal adaptation and seal than resin composites [11], while other studies have shown no significant improvement [12]. In addition, regular flowable composites have lower mechanical properties, and their low viscosity can result in excess material and increased polymerization shrinkage [13]. Finally, their higher content of resin diluents enhances their cytotoxicity [14], and their highly exothermic setting reaction may cause pulpal damage [15]. Compomers [9] and self-adhesive resin cements [16, 17] have also been considered for this purpose, but studies have shown their poor performance [18].

Recently, new generations of high-viscous glass ionomer cements (HV-GICs) have been introduced. These HV-GICs have the advantages of the glass ionomer family (i.e., fluoride release, hydrophilic nature, and intrinsic adhesion to dental tissues), enhanced mechanical and wear properties [19, 20], and promising clinical results as permanent filling [21]. Moreover, they are resin-free, with optimal biocompatibility and no polymerization shrinkage. These characteristics may be useful when implementing a sandwich technique or proximal box elevation. However, with the absence of resin, copolymerization with resin composite is impossible.

We have no information on the bonding ability of resin composites to new generations of HV-GICs. Several adhesive systems may be used, particularly universal adhesives, also called multi-mode adhesives. They contain functional monomers such as 10-MDP and can be used in self-etch (SE) or etch-and-rinse (ER) mode. They are attractive because of their simple clinical procedures, together with their ability to bond to various substrates, such as tooth structure [22], metal [23], zirconia [24], and, for those containing silane, silica-based ceramics [25, 26].

The aim of this study was to investigate the shear bond strength (SBS) and interface between a nanohybrid resin composite and a new HV-GIC, HV-GIC, RM-GIC, bulk-fill flowable composite, and regular flowable composite bonded with different adhesive systems.

Methods and materials

Materials used

Five materials were investigated: a new HV-GIC (EQUIA Forte Fil, GC Corp., Tokyo, Japan), a HV-GIC (Fuji IX GP Fast, GC Corp., Tokyo, Japan), a RM-GIC (Fuji II LC, GC Corp., Tokyo, Japan), a bulk-fill flowable composite (SDR, Dentsply DeTrey, Konstanz, Germany), and a regular flowable composite (Tetric Evo Flow, Ivoclar Vivadent, AG, Schaan, Liechtenstein).

Various adhesive systems were tested, including a universal adhesive (Scotchbond Universal, 3 M ESPE, St Paul, MN, USA), a two-step etch-and-rinse adhesive (Scotchbond 1XT, 3M ESPE, St Paul, MN, USA), a one-step self-etch adhesive (Optibond All-in-one, Kerr Dental, Orange, CA, USA), and a coating material (EQUIA Forte Coat, GC Corp., Tokyo, Japan).

A universal primer for conditioning all types of restoration surfaces (Monobond Plus, Ivoclar Vivadent, AG, Schaan, Liechtenstein) was also used in an experimental group before the application of the one-step self-etch adhesive (Optibond All-in-one, Kerr Dental, Orange, CA, USA).

Materials, abbreviations, manufacturers, batch numbers, and compositions are described in Table 1.

Sample preparation and bonding procedure

In total, 286 samples of self-cure acrylic resin (Plexcil-Escil, Chassieu, France) were produced (diameter 25 mm; height 15 mm), and a standardized cylinder (diameter 7 mm; height 3 mm) was drilled in each. The samples were randomly assigned to 13 groups ($n = 22$).

Group 1 (1a, 1b, 1c, 1d, and 1e, $n = 110$): for each sample, a capsule of EQUIA Forte Fil was mechanically mixed (Rotomix, 3M ESPE, St Paul, MN, USA) and then injected into the standardized cylinder of the resin sample. A Mylar matrix band was placed on the EQUIA Forte Fil, covered with a glass slab, and a 1 kg-weight was applied for 5 min. A cylindrical Teflon mold was placed on each sample to build

Table 1 The abbreviations, the manufacturers, the batch numbers, and the compositions of the restorative materials used

Materials		Classification	Manufacturer	Batch number	Composition
Restorative materials	EQUIA Forte Fil	HV-GIC	GC Corporation, Tokyo, Japan	160608A	Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: polybasic carboxylic acid, water
	Fuji IX GP	HV-GIC	GC Corporation, Tokyo, Japan	160613A	Powder: aluminosilicate glass, polyacrylic acid Liquid: polyacrylic acid, water
	Fuji II LC	RM-GIC	GC Corporation, Tokyo, Japan	160720A	Powder: alumino-fluoro-silicate glass Liquid: polyacrylic acid, HEMA, 2,2,4, trimethyl hexamethylene dicarbonate, TEGDMA
	Tetric Evo Flow	Flowable composite	Ivoclar Vivadent, Schaan, Liechtenstein	V14117	BIS-GMA, UDMA, ytterbium trifluoride, 1,10-decandiol dimethacrylate, barium glass, Ba-Al-fluorosilicate glass, dispersed silica, mixed oxides, copolymer, photoinitiators
	SDR	Bulk-fill flowable composite	Dentsply DeTrey, Konstanz, Germany	1701000793	Polymerizable dimethacrylate resins, polymerizable UDMA, barium boron fluoro alumino silicate glass, silicon dioxide (amorphous), strontium aluminosilicate glass, titanium dioxide, synthetic inorganic iron oxides, photoinitiators
Adhesive systems	Scotchbond Universal	Universal Adhesive	3M ESPE, St Paul, MN, USA	641306	HEMA, Bis-GMA, decamethylene dimethacrylate, ethanol, silane treated silica, water, MDP, copolymer of acrylic and itaconic acid, dimethylamino)ethyl methacrylate, camphorquinone, dimethylaminobenzoat, 2,6-di-tert-butyl-p-cresol, photoinitiators
	Scotchbond 1XT	Two-step etch-and-rinse adhesive	3M ESPE, St Paul, MN, USA	802502	Dimethacrylate, HEMA, polyalkenoic acid polymer, silanized silicium, alcohol, water, photoinitiators
	Optibond All-In-One	One-step self-etch adhesive	Kerr Dental, Orange, CA, USA	5701229	GPDMA, GDMA, HEMA, Bis-GMA, nanosilicates, ytterbium fluoride, camphoroquinone, water, acetone, ethanol
	Equia Coat	Coating Material	GC Corporation, Tokyo, Japan	150205C	Methylmethacrylate, multifunctional methacrylate, camphorquinone
	Monobond Plus	Universal Primer	Ivoclar Vivadent, Schaan, Liechtenstein	U09068	Ethanol, 3-(trimethoxysilyl)propyl methacrylate, methacrylated phosphoric acid ester
Etching system	Scotchbond Universal Etchant	Etchant	Kerr Dental, Orange, CA, USA	N148329	Orthophophoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide
Resin composite	Filtek Z350 XT	Nanohybrid resin composite	3M ESPE, St Paul, MN, USA	N797777	Bis-GMA, UDMA, TEGDMA, Bis-EMA, silica and zirconia filler, photoinitiators

Bis-GMA, bisphenol A diglycidyl dimethacrylate; *Bis-EMA*, ethoxylated bisphénol-A diméthacrylate; *GDMA*, glycerol dimethacrylate; *GPDMA*, glycerol-phosphoric acid dimethacrylate; *HEMA*, 2-hydroxyethyl methacrylate; *MDP*, methacryloyloxydecyl dihydrogen phosphate; *TEGDMA*, triethylene glycol dimethacrylate; *UDMA*, urethane dimethacrylate

a 3-mm-high nanohybrid resin composite cylinder (Filtek 350, 3M ESPE, St Paul, MN, USA) with a flat base of 7 mm^2.

Resin composite cylinders were produced in two increments, which were light-cured for 40 s, with a minimum output of 950 mW/cm^2. After light-curing, the mold was removed and the excess adhesive, if present, was gently removed from around the base of the resin composite cylinder using a scalpel. All samples were stored in water at 37 °C.

Various adhesive systems were applied on the EQUIA Forte Fil to bond the resin composite cylinder. The following sub-groups were investigated:

Group 1a (n = 22): EQUIA Forte Fil + Scotchbond Universal in self-etch mode (U(SE)) + nanohybrid resin composite.

Group 1b (n = 22): EQUIA Forte Fil + Scotchbond Universal in total-etch mode (U(ER)) + nanohybrid resin composite.

Group 1c (n = 22): EQUIA Forte Fil + Scotchbond 1XT (S-1XT) + nanohybrid resin composite.

Group 1d (n = 22): EQUIA Forte Fil + Optibond All-in-one (AIO) + nanohybrid resin composite.

Group 1e (n = 22): EQUIA Forte Fil + Monobond Plus (MP) + AIO + nanohybrid resin composite.

Group 1f ($n = 22$): EQUIA Forte Fil + EQUIA Forte Coat (E-Coat) + nanohybrid resin composite.
Group 2 ($n = 22$): Fuji IX GP + U(SE) + nanohybrid resin composite.

Fuji IX GP was inserted into the cylinder according to the same protocol as the EQUIA Forte Fil. Similarly, a 3-mm-high nanohybrid resin composite cylinder was bonded on the Fuji IX GP with U(SE).

Group 3 ($n = 22$): Fuji II LC (RM-GIC) + U(SE) + nanohybrid resin composite.

Fuji II LC was inserted into the cylinder according to the same protocol as the EQUIA Forte Fil and light-cured for an additional 20 s, with the application of a 1 kg-weight (Valo LED, Ultradent, South Jordan, UT, USA). Similarly, a 3-mm-high nanohybrid resin composite cylinder was bonded on the Fuji II LC with U(SE).

Group 4 ($n = 22$): SDR + U(SE) + nanohybrid resin composite.

SDR was inserted into the cylinder according to the same protocol as the EQUIA Forte Fil and light-cured for an additional 20 s (Valo LED, Ultradent, South Jordan, UT, USA), with the application of a 1 kg-weight. Similarly, a 3-mm-high nanohybrid resin composite cylinder was bonded on the SDR with U(SE).

Group 5 ($n = 22$): Tetric Evo Flow + U(SE) + nanohybrid resin composite.

Tetric Evo Flow was injected into the cylinder by increments of 1.5 mm. Each increment was light-cured for 20 s and the last increment was light-cured through a glass slab, with the application of a 1 kg-weight. Similarly, a 3-mm-high nanohybrid resin composite cylinder was bonded on the SDR with U(SE).

The application details are described in Table 2.

For each group ($n = 22$), 20 specimens were tested for SBS after 48 h of storage, and 2 specimens were used for microscopy analysis.

SBS testing and failure mode determination

SBS was determined using a universal testing machine (LRX, Lloyd Instruments, Fareham, UK). The shear force was applied at the junction between the resin composite cylinder and the tested material included in the self-cured resin sample using a chisel-shaped blade parallel to the resin surface. A cross-head speed of 0.5 mm/min was selected.

The debonded specimens were observed under a binocular microscope (BZH10 Olympus, Hamburg, Germany) at × 30 magnification and failures were classified as the following three types:

Type CF: cohesive failure within the resin composite or the tested material (i.e., EQUIA Forte Fil, Fuji IX GP, Fuji II LC, SDR and Tetric Evo Flow);
Type AF: adhesive failure at the interface between the resin composite and the tested material (i.e., EQUIA Forte Fil, Fuji IX GP, Fuji II LC, SDR and Tetric Evo Flow); and
Type MF: mixed failure (adhesive and cohesive failure).

Environmental scanning electron microscopy examination

Two specimens from each group were sectioned perpendicularly to the bonded interface using a low-speed diamond observed (Isomet, Buehler, Coventry, UK), with water cooling, as near as possible to the center of the cylinder. The sections obtained were polished with abrasive discs of decreasing grit size (400, 800, 1200, 2400, and 4000 SiC) and then with diamond particles of 3.1 and 0.25 μm. The specimens were cleaned by ultrasonication after each polishing step and then examined using environmental scanning electron microscopy (E-SEM) (Kanta, FEI, Hillsboro, OR, USA).

Statistical analysis

The normal distribution of data was confirmed by the Shapiro-Walk test, and the equality of variances was assessed by the Levene test, before the tests were performed. SBS data are expressed as the mean and standard deviations. Two-way ANOVA followed by Dunnett's post hoc test was used to investigate the difference in SBS between the tested materials. Failures were analyzed by chi-square test. In all tests, the significance level was $p < 0.05$. Statistical calculations used XLSTAT software (Addinsoft, Paris, France).

Results

SBS and failure mode analysis

The SBS for all experimental groups are summarized in Table 3. The two-way ANOVA revealed that SBS was significantly influenced by the adhesive and material tested.

The SBS significantly differed between the various adhesive systems for the groups of the EQUIA Forte Fil. From the Dunnett's post hoc test, with the universal adhesive in self-

Table 2 Bonding protocol for each group tested

Group tested	Restorative material used	Adhesive system used (abbreviation)	Adhesive protocol used	Bonding protocol
1a	EQUIA Forte Fil	Scotchbond Universal U(SE)	One-step self-etch	Scotchbond Universal was applied on EQUIA Forte Fil (after set-up), rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20 s.
1b	EQUIA Forte Fil	Scotchbond Universal U(ER)	Two-step etch-and--rinse	Scotchbond Universal Etchant was applied on EQUIA Forte Fil (after set-up) for 15 s and then rinsed thoroughly for 10 s. Excess water was blotted with a cotton pellet. Then, Scotchbond Universal was applied, rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20s.
1c	EQUIA Forte Fil	Scotchbond 1XT (S-1XT)	Two-step etch-and--rinse	Scotchbond Universal Etchant was applied on EQUIA Forte Fil (after set-up) for 15 s and then rinsed thoroughly for 10 s. Excess water was blotted with a cotton pellet. S-1XT was applied and rubbed for 15 s. A second application was performed for 15 s, gently air-dried for 5 s, and light-cured for 20 s.
1d	EQUIA Forte Fil	Optibond All-In-One (AIO)	One-step self-etch	A first layer of AIO was applied on EQUIA Forte Fil (after set-up) and then rubbed for 20 s. Next, a second layer of AIO was immediately applied with brushing motions for 20 s, air-dried for 5 s, and light-cured for 20 s.
1e	EQUIA Forte Fil	Monobond Plus + Optibond All-in-One (MP + AIO)	Silanization + one-step self-etch protocol	MP was applied on EQUIA Forte Fil (after set-up), rubbed for 60 s, and gently air-dried for 5 s. Then, the first layer of AIO was applied on the prepared surface and rubbed for 20 s. Next, a second layer of AIO was immediately applied with brushing motions for 20 s, gently air-dried for 5 s, and light-cured for 20 s.
1f	EQUIA Forte Fil	EQUIA Forte Coat (E-Coat)	One-step self-etch	After its set-up, EQUIA Forte Fil was dried but not desiccated by the gentle blowing of oil-free air. E-Coat was applied, rubbed for 20 s, not air-dried, and light-cured for 20 s.
2	Fuji IX GP	Scotchbond Universal U(SE)	One-step self-etch	Scotchbond Universal was applied on Fuji IX GP (after set-up), rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20 s.
3	Fuji II LC	Scotchbond Universal U(SE)	One-step self-etch	Scotchbond Universal was applied on Fuji II LC (after set-up), was rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20 s.
4	SDR	Scotchbond Universal U(SE)	One-step self-etch	Scotchbond Universal was applied on SDR (after set-up), rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20 s.
5	Tetric Evo Flow	Scotchbond Universal U(SE)	One-step self-etch	Scotchbond Universal was applied on Tetric Evo Flow (after set-up), rubbed for 20 s, gently air-dried for 5 s (until there was no movement, i.e., complete solvent evaporation), and light-cured for 20 s.

etch mode as the control (EQUIA Forte Fil + U(SE), 47.4 MPa), the SBS was significantly lower with the two-step etch-and-rinse adhesive (EQUIA Forte Fil + S-1XT, 39.2 MPa) than with the other adhesive systems. The SBSs of the other adhesive systems did not differ (EQUIA Forte Fil + U(ER), 47.4 MPa; EQUIA Forte Fil + AIO, 43.1 MPa;

Table 3 Mean and standard deviations (SD) of SBS for the various groups tested

Groups tested	Materials tested	Adhesives tested	SBS (±SD)	*p* values
1a	EQUIA	U(SE)	47.4 ± 8.1	
1b		U(ER)	47.4 ± 5.8	1.000
1c		S-1XT	39.2 ± 9.2*	0.047
1d		AIO	43.1 ± 9.6	0.459
1e		MP + AIO	45.6 ± 8.4	0.969
1f		E-Coat	41.6 ± 9.3	0.159
2	F-IX	U(SE)	39.4 ± 5.1*	0.014
3	F-II LC	U(SE)	54.3 ± 9.4*	0.043
4	SDR	U(SE)	59.8 ± 10.7*	< 0.0001
5	TEF	U(SE)	62.9 ± 10.0*	< 0.0001

Values with an asterisk are significantly different from the control group (1a) at $p < 0.05$

EQUIA Forte Fil + MP + AIO, 45.6 MPa; and EQUIA Forte Fil + E-Coat, 41.6 MPa).

The SBS significantly differed among the various tested materials bonded with the universal adhesive. The SBS was significantly higher with the EQUIA Forte Fil than with the Fuji IX GP (47.4 vs 39.4 MPa) but was lower with the EQUIA Forte Fil than with the Fuji II LC (54.3 MPa), SDR (59.8 MPa), and Tetric Evo Flow (62.9 MPa).

The failure mode results are shown in Table 4. The various groups did not differ in failures and were predominantly cohesive failures. However, the groups EQUIA Forte Fil + U(SE), EQUIA Forte Fil + U(ER), and Tetric Evo Flow + U(SE) showed cohesive failures only, whereas groups EQUIA Forte Fil + S-1XT and EQUIA Forte Fil + E-Coat showed 20% mixed and adhesive failures. The group EQUIA Forte Fil + S-1XT had the highest number of adhesive failures.

Table 4 Mode of failure for the various groups tested

Group tested		Number of samples	CF	MF	AF
1a	EQUIA + U(SE)	20[a]	20	0	0
1b	EQUIA + U(ER)	20[a]	20	0	0
1c	EQUIA + S-1XT	20[a]	16	1	3
1d	EQUIA + AIO	20[a]	19	0	1
1e	EQUIA + E-Coat	20[a]	16	2	2
1f	EQUIA + MP + AIO	20[a]	18	1	1
2	F-IX + U(SE)	20[a]	19	0	1
3	F-II LC + U(SE)	20[a]	19	1	0
4	SDR + U(SE)	20[a]	18	2	0
5	TEF + U(SE)	20[a]	20	0	0

Values with the same superscript letter are not significantly different at $p < 0.05$. *CF*, cohesive failure; *MF*, mixed failure; *AF*, adhesive failure

E-SEM examination

Figures 1 and 2 show the E-SEM images for all group 1 groups at × 1000 and × 5000 magnifications, respectively. Figure 3 shows the E-SEM images for groups 2 to 5 at × 5000 magnification.

Each group showed intimate contact between the resin composite and tested adhesive system, as well as between the tested adhesive system and the tested material. Among group 1 groups, the EQUIA Forte Fil surface for groups with an etch-and-rinse technique (groups 1a and 1c) was rougher than that for groups with a self-etch technique. The adhesive was interlocked with the EQUIA Forte Fil surface.

Regardless of the glass ionomer cement (GIC) (groups 1, 2, and 3) and adhesive system (groups 1a, 1b, 1c, 1d, 1e, and 1f), the E-SEM image showed a dark layer between the GIC and adhesive on the order of a micron, which was not found for the bulk-fill flowable and regular flowable composite groups (groups 4 and 5).

The adhesive system thickness ranged from 6 to 31 μm, depending on the group.

The lowest value was obtained with EQUIA Forte Fil + U(SE) (6 μm) and the highest value was for EQUIA Forte Fil + E-Coat (31 μm). The other groups tested showed intermediate values (EQUIA Forte Fil + AIO, 6–7 μm; SDR + U(SE), 8–9 μm; EQUIA Forte Fil + MS + AIO, 10 μm; Fuji IX GP + U(SE), 10 μm; EQUIA Forte Fil + U(ER), 16 μm; EQUIA Forte Fil + S-1XT, 16 μm; and Tetric Evo Flow + U(SE), 16–17 μm).

Discussion

Laminate restorations involve two interfaces: the first is between the tooth structure and the base material, and the second is between the base material and the direct/indirect above the restoration. For the latter interface, a good coaptation is crucial to ensure good stress transmission and long-term success [3]. The present study is the first to investigate the bonding ability of EQUIA Forte Fil to a resin composite with a universal adhesive.

SBS values

Although the EQUIA Forte Fil and Fuji IX GP were resin-free, the resin composite bound to them with all adhesive systems tested. The SBS on EQUIA Forte Fil did not differ between the use of U(SE) (47.4 MPa) and U(ER), AIO, MP + AIO, or E-Coat (47.4, 43.1, 45.6 MPa and 41.6 MPa, respectively). However, the S-1XT SBS was significantly lower than the U(SE) SBS (39.4 MPa).

The equivalent SBS obtained with EQUIA Forte Fil for U(SE) and U(ER) suggests that a chemical bond between the universal adhesive and HV-GIC likely occurs via interactions between the dihydrogenphosphate group of 10-MDP

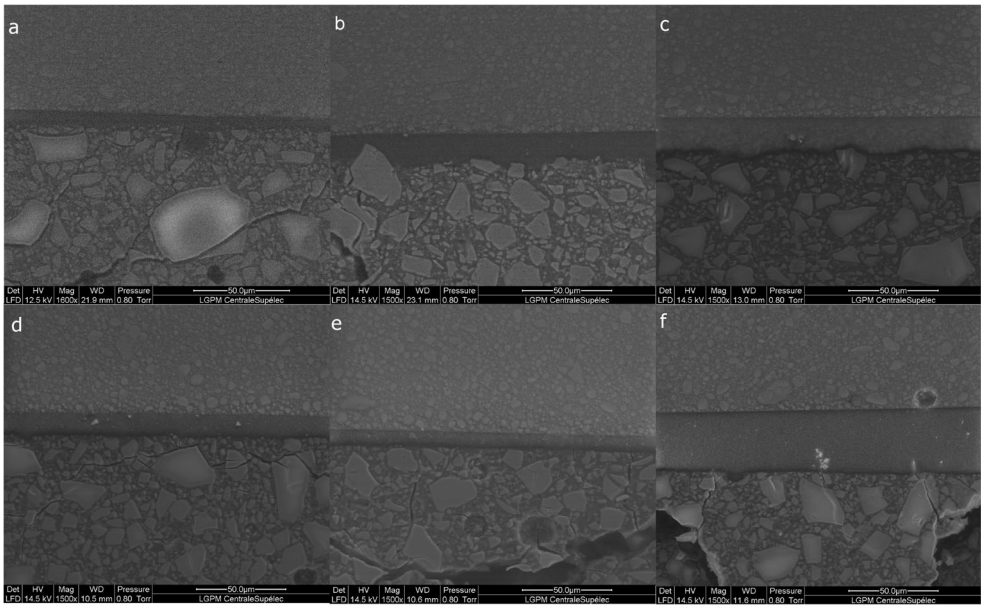

Fig. 1 E-SEM micrograph for groups 1a to 1e, of the interface resin composite/adhesive/EQUIA Forte Fil (× 1500 magnification) with the following adhesives: (a) U(SE), (b) U(ER), (c) S-1XT, (d) AIO, (e) MP + AIO, and (f) E-Coat. The resin composite is at the top and EQUIA Forte Fil is at the bottom

from the universal adhesive and the calcium ions from the GIC matrix [23, 27]. This ionic binding would result in MDP-calcium salts that are stable in an aqueous environment [23]. The hydrophilic nature of dihydrogenphosphate groups would also help them penetrate the hydrophilic matrix. Adhesion was observed between a 10-MDP containing adhesive and calcium-containing material, such as Biodentine (Septodont, St Maur-des-Fossés, France) [28, 29]. Moreover, other interactions are possible between the acrylic and itaconic acid, as well as the silane from the universal adhesive and HV-GIC.

A previous study showed that both etch-and-rinse and self-etch adhesives provide a reliable bond to HV-GIC [30]. Bonding to GIC with an etch-and-rinse adhesive, in the same manner as enamel and dentin, is explained by micromechanical adhesion. Nevertheless, the effect of phosphoric acid on GIC is not well understood, as some studies reported a decreased bond strength (by weakening the cement surface, it is more prone to cohesive failures) [31], whereas others reported increased bond strength, via the formation of a hybrid-like layer [32].

Self-etch adhesives contain acidic monomers that are less aggressive than phosphoric acid, with a pH of 2.7 and 2.5 for U(SE) and AIO, respectively, which are compatible with the GIC matrix and provide better SBS [33]. A nanomechanical or a chemical bond is likely; however, the ability of GPDM, a functional monomer contained in AIO, to bind to calcium ions has not been studied. The lower SBS with AIO, even if not statistically significantly different, may be explained by either a lower affinity to the calcium ions of GPDM compared with 10-

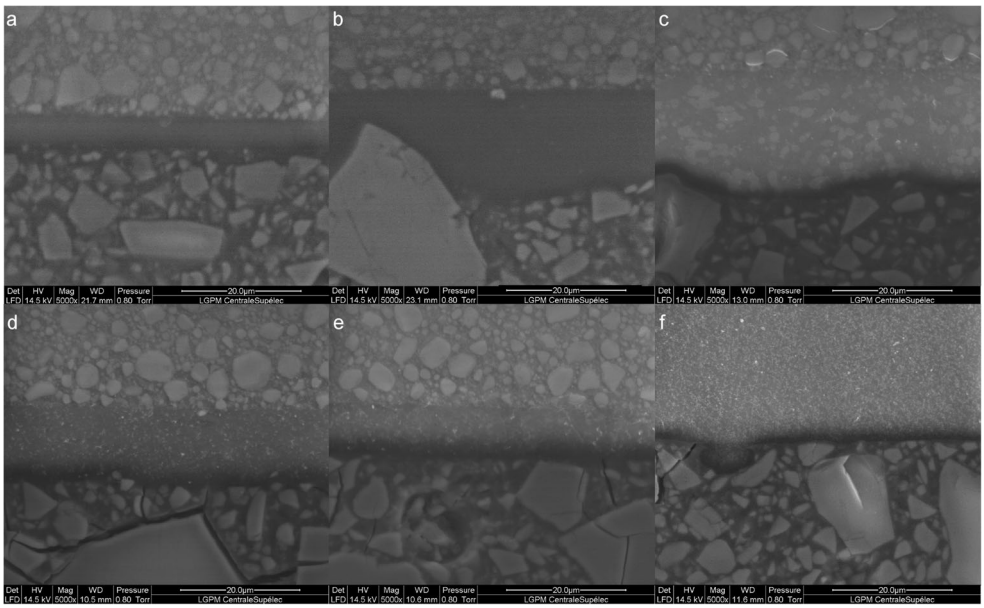

Fig. 2 E-SEM micrograph for groups 1a to 1e, of the interface resin composite/adhesive/EQUIA Forte Fil (× 5000 magnification) with the following adhesives: (a) U(SE), (b) U(ER), (c) S-1XT, (d) AIO, (e) MP + AIO, and (f) E-Coat. The resin composite is at the top and EQUIA Forte Fil is at the bottom (except for 1f because of higher E-Coat thickness)

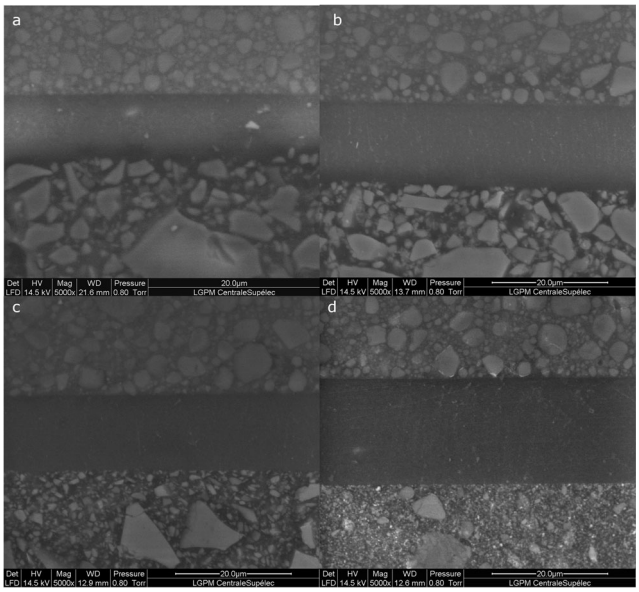

Fig. 3 E-SEM micrograph for groups 2 to 5 of the interface resin composite/U(SE)/tested material (× 5000 magnification) with the following tested materials: (a) Fuji IX GP, (b) Fuji II LC, (c) SDR, and (d) Tetric Evo Flow. The resin composite is a the top and the materials tested are at the bottom

MDP or its higher steric hindrance [34]. Using a silane coupling agent before the AIO did not improve the SBS, whereas MP contains 10-MDP and silane; therefore, its high ethanol content would desiccate EQUIA Forte Fil and weaken its cohesion.

When using U(SE), the SBS was significantly higher with the resin composite bonded to EQUIA Forte Fil than to Fuji IX GP but was lower than with the resin composite bonded to Fuji II LC, Tetric Evo Flow, and SDR. The resin composite bonded using U(SE) necessarily showed better SBS to regular flowable and bulk-fill flowable composites, but the use of GIC presents some advantages, and the use of HV-GIC presents even more benefits. In fact, regular flowable and bulk-fill flowable composites are hydrophobic materials, and moisture management is difficult in deep cavities, particularly when adjacent cavities must be filled. If saliva contamination occurs, the SBS is greatly reduced for resin composites [35] compared with RM/HV-GIC. HV-GIC can be used without a rubber dam, according to the manufacturer recommendations. In contrast with bulk-fill flowable composites, regular flowable composites also require several layers to reduce stress during polymerization [36] and are time-consuming. Moreover, the biocompatibility of these regular flowable composites is controversial [37–39]. The intrinsic adhesion of HV/RM-GIC to enamel and dentin would secure sealing, which is difficult to control in deep cavities, particularly without the aid of magnification [40, 41]. Finally, HV-GIC can be used in synergy with the immediate dentin sealing technique for indirect restorations to provide high bond strength values and low post-operative sensitivity and avoid bacterial contamination during the temporization phase [42].

The unreacted methacrylate groups of RM-GIC could form a strong chemical covalent bond with U(SE), thereby increasing the SBS [43]. However, previous studies have reported a high water uptake for RM-GIC due to the hydrophilic nature of HEMA [44–46], which causes the hydrolytic degradation of fillers and the resin matrix. The biocompatibility of RM-GICs is also questionable. In addition, the Fuji IX GP is resin-free, but its lower mechanical properties could explain its significantly lower SBS compared with EQUIA Forte Fil.

Failure mode analysis

We found no significant differences in failures among the groups. The most failures were cohesive within the tested material, which is consistent with previous results for GIC [30, 47]. Therefore, the SBS did not exactly measure the interfacial bond but represents the cohesive strength of the material, which is lower than the adhesive bond. In fact, several studies supported that cohesive failures are related to high bond strength [47, 48].

The Tetric Evo Flow + U(SE) group showed only cohesive failures, which suggests the high quality of the bonding interface. The EQUIA Forte Fil + S-1XT and EQUIA Forte Fil + E-Coat groups showed 20% mixed and adhesive fractures, which suggests a lower quality of the bonding interface. The EQUIA Forte Fil + S-1XT group showed the highest number of adhesive failures, which is consistent with the significantly lower SBS values obtained.

E-SEM image analysis

The intimate contact between EQUIA Forte Fil and all adhesive systems, as well as U(SE) and all alternative materials, is consistent with the satisfactory SBS values obtained and the predominance of cohesive failures in all groups. The rougher EQUIA Forte Fil surface for the groups using the etch-and-rinse technique and adhesive interlocking with surface irregularities are also similar to a previous study [49]. For the groups using the self-etch technique, the EQUIA Forte Fil/adhesive interface was plane, and nano-interlocking is possible.

The dark layers (present in the SE and ER protocols) between all GIC groups and the adhesive can be explained by a level difference between the GIC, adhesive, and resin composite, due to their different polishability. However, it may also reflect an ion-exchange process between the GIC (containing calcium ions) and the adhesive (containing monomers and polyalkenoic acid), similar to the gel phase [50, 51] described between RM-GIC and dentin.

Limitations of the study

In vitro investigations cannot simulate all clinical aspects to predict clinical behavior; therefore, further prospective clinical

trials are needed. In addition, further studies that analyze the interfaces between new HV-GIC and enamel/dentin are necessary, particularly in the presence of saliva contamination.

Conclusion

The SBS of resin composite bonded to EQUIA Forte Fil using a universal adhesive in self-etch mode (U(SE)) did not significantly differ from that with the same universal adhesive used in etch-and-rinse mode (U(ER)) or a one-step self-adhesive (AIO) but was higher than that with an etch-and-rinse adhesive (S1-XT).

The SBS of resin composite bonded to EQUIA Forte Fil using a universal adhesive in self-etch mode U(SE) was higher than the SBS of Fuji IX GP but lower than the SBS of Fuji II LC, SDR, and Tetric Evo Flow.

SEM images of bonding interfaces with intimate contacts for each group are consistent with a satisfactory SBS. The cohesive failures obtained in each group also suggest the high strength of the bonding interfaces.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflicts of interest.

Ethical approval Not applicable.

Informed consent Not applicable.

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