Bonding Efficacy of a New Self-Adhesive Restorative onto Flat Dentin vs Class-I Cavity-bottom Dentin

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Purpose: This study investigated the bonding efficacy of a new so-called self-adhesive composite hybrid onto flat (FLAT) and high C-factor class-I cavity-bottom (CAVITY) dentin.

Materials and Methods: The immediate and aged (50,000 thermocycles) microtensile bond strength (µTBS) to FLAT and CAVITY dentin of the experimental self-adhesive bulk-fill restorative (K-0180 ASAR pilot [ASAR-pilot], Dentsply Sirona) was compared to that of two universal adhesives applied in self-etch mode and combined with a bulk-fill composite (Prime&Bond Elect/QuiXfil [P&Be/QuiXF], Prime&Bond Active/QuiXfil [P&Ba/QuiXF], both Dentsply Sirona), two pre-conditioned materials (Activa Bioactive-Restorative [Activa], Pulpdent; Fuji II LC Improved [Fuji2LC], GC); and one bulk-fill glass-hybrid restorative (Equia Forte Fil [EquiaF], GC). Statistically significant differences were recorded using Welch's ANOVA with Games-Howell contrast (p < 0.05).

Results: No significant difference in immediate µTBS was recorded when the restorative materials were applied onto FLAT dentin, except for Activa_FLAT and EquiaF_FLAT. When bonded to CAVITY dentin, the significantly highest µTBS was recorded for Fuji2LC_CAVITY (layer filled), and was not significantly different only from P&Ba/QuiXF_CAVITY. Upon aging, the highest µTBS to flat dentin was achieved by ASAR-pilot_FLAT, which was not significantly different from P&Be/QuiXF_FLAT and Fuji2LC_FLAT. No significant difference between immediate and aged µTBS was recorded for ASAR-pilot when bonded onto FLAT or CAVITY dentin; the latter, however, was associated with low bond strength.

Conclusion: Favorable bonding performance was found for the new self-adhesive bulk-fill composite hybrid ASAR-pilot when bonded to flat dentin. However, much lower bond strength was recorded when ASAR-pilot was bonded to high C-factor cavity-bottom dentin.

Keywords: bond strength, bulk-fill, composite, dentin, durability.

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Resin-based composite has widely been applied in dental practice since it was introduced into dentistry in the 1950s.[19] When combined with a modern dental adhesive, adhesively restoring teeth in a reliable, predictable, and durable way can now be considered a reality. However, research and development in dental adhesive technology remains challenging, for instance, to fabricate adhesives that are also effective in conditions of suboptimal field control or complex cavity configurations, or to develop true amalgam alternatives that are economic, easy-to-place, and preferably self-adhesive. With the goal of clinically shorter application times and lower technique sensitivity, the development of self-adhesive tooth-colored restorative materials that no longer need pre-treatment with a separate adhesive is ongoing.[7,38,41]

Self-adhesive restorative composites can be considered the logical successor of self-adhesive composite cements, which have been commercially available for quite some time as easy-to-use "trade-off" cements to lute semi-direct/indirect ceramic and composite CAD/CAM restorations. Regarding bonding effectiveness, these self-adhesive luting composites have generally been found to be less effective than their etch-and-rinse and self-etch adhesive-assisted counterparts, which require a separate etch-and-rinse or self-etch adhesive, respectively, as luting pre-treatment.[13,50] With a consistency and composition quite similar to that of luting composites, flowable self-adhesive restorative materials were developed first, of which some were commercialized with mixed success.[38,49,51] Self-evidently, self-adhesiveness is easier to achieve with a flowable than a more viscous paste-like product due to the better surface-wetting potential of less viscous composites.

Self-adhesive composite which contains adhesive in the restorative material must be combined with bulk-fillability to become an amalgam-like dental restorative material. Bulk-filling cavities in posterior teeth enables clinicians to fill 4- to 5-mm deep boxes in one bulk increment.[17,43] Nevertheless, previous research demonstrated that bulk-filling does not always provide favorable results; for instance, some bulk-fill composites were shown to develop higher polymerization shrinkage and larger interfacial gaps than incrementally layered composites.[2,21]

Recently, a so-called self-adhesive bulk-fill restorative composite hybrid (coded as K-0180 ASAR pilot [ASAP-pilot], Dentsply Sirona; Konstanz, Germany) was developed and later commercially introduced as Surefil One (Dentsply Sirona). To our knowledge, few in vitro studies have addressed the performance of self-adhesive bulk-fill restoratives. This study investigated the immediate and aged bonding efficacy of the self-adhesive bulk-fill restorative hybrid when applied to flat vs class-I cavity-bottom dentin. The latter serves as a worst-case condition due to the high C-factor involved. Representative adhesive-composite combinations as well as conventional or resin-modified glass-ionomer cements (GICs) were likewise applied, serving as controls. The null hypotheses tested were: 1. the immediate bonding efficacy to flat dentin or cavity-bottom dentin of ASAR-pilot did not significantly differ from that obtained with the other restorative materials bonded to flat (1a) or high C-factor class-I cavity-bottom dentin (1b); 2. the bonding efficacy did not decrease upon artificial aging when again applied under the two substrate conditions (2a,b).

MATERIALS AND METHODS

Tooth Preparation
Ninety-six non-carious human third molars were collected following informed consent approved by the Commission for Medical Ethics of KU Leuven (file number S57622), stored
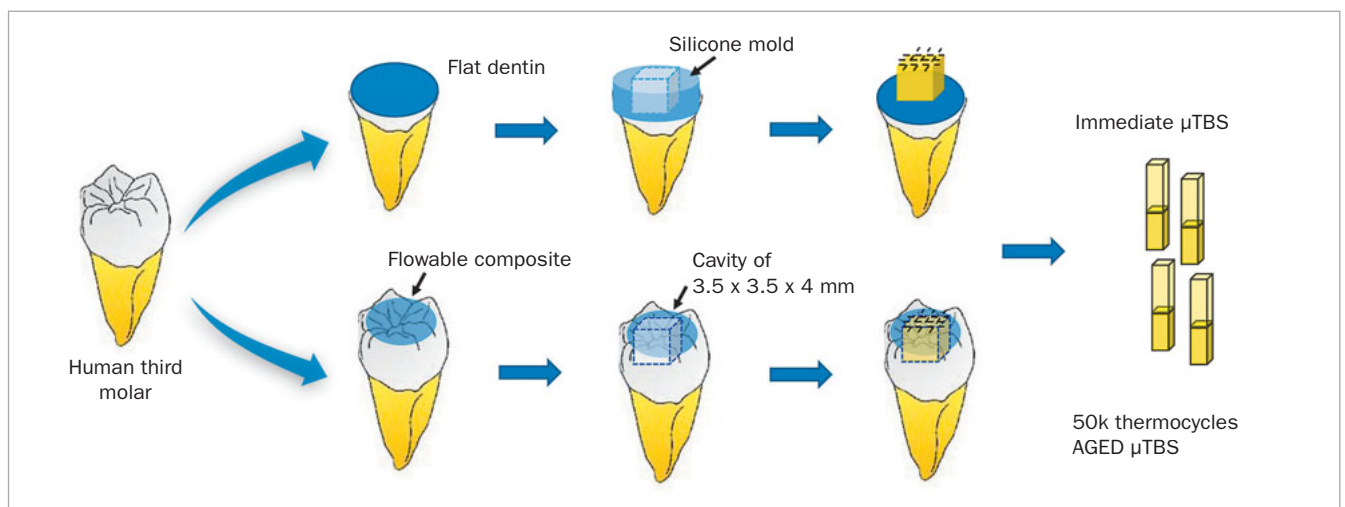
**Fig 1** Schematic illustrating the specimen preparation protocol for microtensile bond strength (µTBS) testing.

Table 1 List of materials investigated in this study

Materials	Code	Composition[1]	Filler loading	Application procedure	Batch No.
K-0180 ASAR pilot (Dentsply Sirona)	ASAR-pilot	Aluminum-phoshor-strontium-sodium-fluoro-silicate glass, highly dispersed silicon dioxide, ytterbium fluoride, polycarboxylic acid, bifunctional acrylate, acrylic acid, iron oxide pigments, water, titanium dioxide pigments, camphorquinone, stabilizer, self-cure initiator	Not specified	Upon application in 4-mm bulk, light cure for 20 s with an output of 1200 mW/cm^2.	Not specified
QuiXfil (Dentsply Sirona)	QuiXF	UDMA, TEG-DMA, di- and trimethacrylate resins, carboxylic acid modified dimethacrylate resin, BHT, UV stabilizer, camphorquinone, phosphate silicate glass, ethyl-4-dimethylaminobenzoate, silanated strontium aluminum sodium fluoride	66 vol% 86 wt%	1. Application of Prime&Bond Elect (P&Be; PENTA, urethane dimethacrylate monomer, 2-hydroxy-3-acryloyloxypropyl methacrylate, HEMA, trimethylolpropane trimethacrylate, acetone) or Prime&Bond Active (P&Ba; MDP, PENTA, bisacrylamide 1, bisacrylamide 2, propan-2-ol, 4-(dimethylamino)benzonitrile) in self-etch mode (both adhesives from Dentsply Sirona) 2. Upon application in 4-mm bulk, light cure for 10 s with an output of 1200 mW/cm^2	1710000818
Activa Bioactive-Restorative (Pulpdent)	Activa	Blend of diurethane and other methacrylates with modified polyacrylic acid, silica, amorphous, sodium fluoride	56 wt%	1. Etch prepared dentin for 5 s with DeTrey Conditioner 36 (Dentsply Sirona; 25–50% phosphoric acid), rinse well and lightly dry 2. Upon application in 4-mm bulk, light cure for 20 s with an output of 1200 mW/cm^2	171102
Fuji II LC Improved (GC)	Fuji2LC	HEMA, polybasic carboxylic acid, UDMA, dimethacrylate, others	76 wt%	1. Apply Dentin Conditioner (GC; 20% polyacrylic acid, 3% aluminum chloride, distilled water) for 20 s, rinse thoroughly with water and dry gently 2. Upon successive application in layers of max. 1.8-mm thickness (3 layers), light cure each layer for 20 s with an output of 1200 mW/cm^2	170713A
Equia Forte Fil (GC)	EquiaF	Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: polybasic carboxylic acid, water	Not specified	No pre-treatment; self-cure, wait for 2.5 min prior to further specimen processing	170807A

[1]According to information provided by the respective manufacturer. Abbreviations: BHT: butylated hydroxy toluene; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyl-oxydecyl-dihydrogenphosphate; PENTA: dipentaerythritol pentaacrylate phosphate; TEG-DMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.

in 0.5% aqueous chloramine-T at 4°C, and used within 1 month of extraction. All teeth were randomly subdivided in 12 experimental groups (n = 8 per group). For the six FLAT groups, the crown was cut 4 mm below the cusp tips, ending with a surface in mid-coronal dentin. For the six CAVITY groups, the teeth were first built up using the flowable composite G-ænial Universal Flo (GC; Tokyo, Japan) after etching enamel with phosphoric acid (DeTrey Conditioner 36, Dentsply Sirona) and subsequent application of the univer-

sal adhesive Prime&Bond Active (Dentsply Sirona). A flat surface was made at the level of the cusp tips, upon which standard box-type class-I cavities (3.5 x 3.5 x 4 mm) were prepared with the cavity bottom ending in mid-coronal dentin, as for the FLAT dentin surfaces. In this way, the effect of regional variability on microtensile bond strength (µTBS) was minimized. All preparations were made using the MicroSpecimen Former (University of Iowa; Iowa City, IA, USA), equipped with a high-speed medium-grit (107 µm) diamond
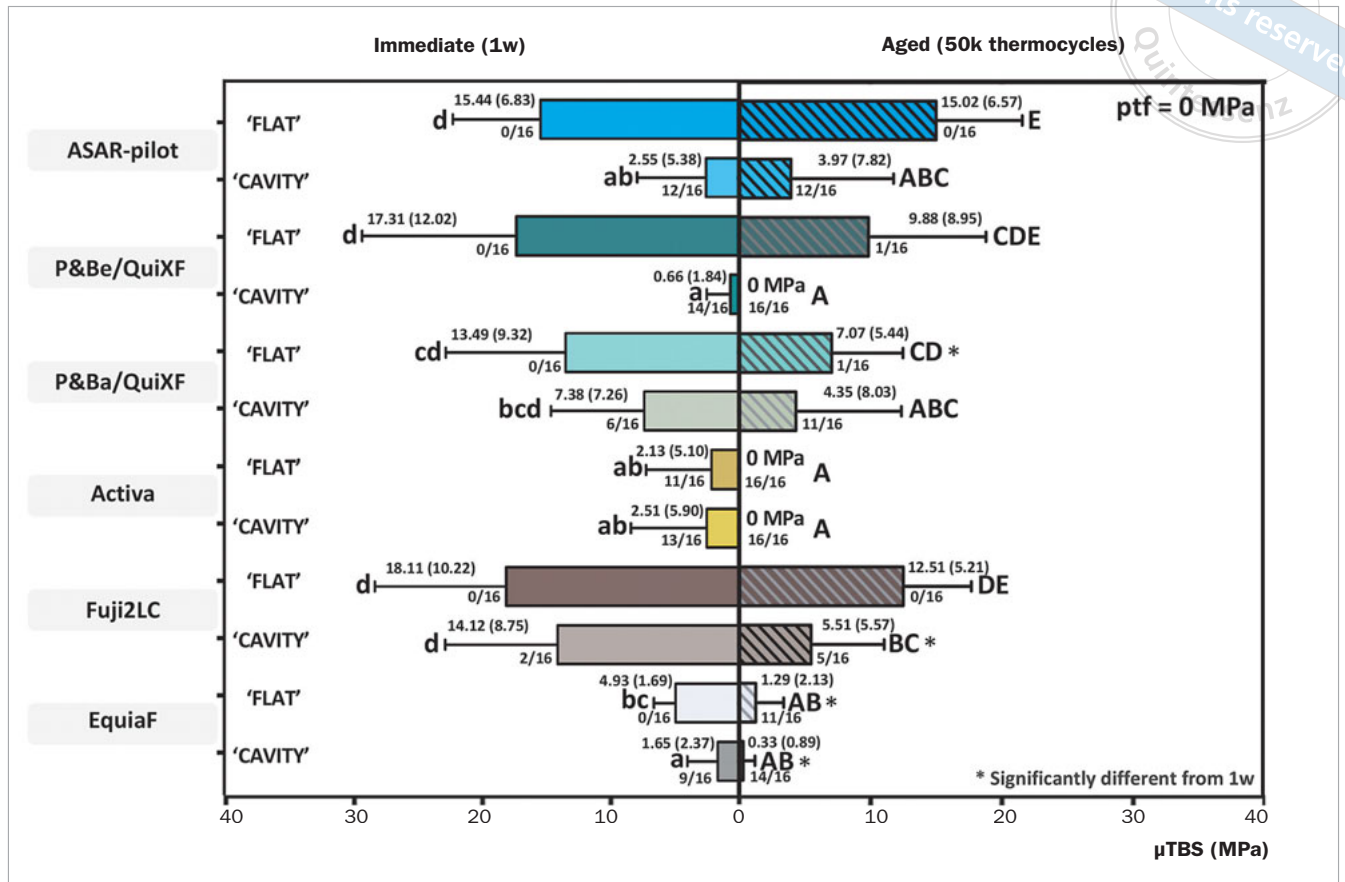
Fig 2 Immediate and aged µTBS of the six restorative systems to flat (FLAT) and class-I cavity-bottom dentin (CAVITY). The mean µTBS (SD) and the number of ptfs/total number of micro(µ)-specimens are given for each bar. Groups with the same small (immediate µTBS) or capital (aged µTBS) letter are not statistically significantly different (p > 0.05). Asterisk indicates a significant difference between the immediate and aged µTBS (p < 0.05).

bur (882, Komet; Lemgo, Germany). A 3.5 x 3.5 x 4-mm buildup (the same dimensions as those of the class-I cavities) was made on the flat dentin surfaces by applying the respective restorative material in bulk/layers in an addition-silicone mold (Aquasil medium body, Dentsply Sirona), while the class-I cavities were likewise bulk/layer-filled with the restorative materials. The experimental tooth preparation protocol is schematically presented in Fig 1.

All restorative materials were applied strictly following the respective manufacturers instructions (Table 1). Bonding efficacy of the experimental self-adhesive bulk-fill composite hybrid ASAR-pilot (group 1) was compared with the adhesive/composite combinations consisting of:

- the universal adhesives Prime&Bond Elect or Prime&Bond Active (both Dentsply Sirona) applied in self-etch mode prior to the application of the paste-like full-body bulk-fill composite QuiXFil (Dentsply Sirona) (P&Be/QuiXF and P&Ba/QuiXF, groups 2 and 3, respectively)
- the so-called bioactive ionic resin with reactive glass filler (Activa Bioactive-Restorative, Pulpdent; Watertown, MA, USA) following phosphoric acid-etching using DeTrey Conditioner 36 (Dentsply Sirona) (Activa, group 4)

- the resin-modified glass-ionomer restorative Fuji II LC Improved (GC; Tokyo, Japan) applied in three layers following conditioning with the polyalkenoic glass-ionomer conditioner Dentin Conditioner (GC) or the bulk-fill glass-hybrid restorative Equia Forte Fil (GC) (Fuji2LC and EquiaF, groups 5 and 6, respectively).

The composite buildups/restorations were light cured using an LED curing light (Bluephase 20i, Ivoclar Vivadent; Schaan, Liechtenstein) with an output of 1200 mW/cm^2 when used in high mode, confirmed regularly during the experiment using a Marc Resin Calibrator (BlueLight Analytics; Halifax, Canada). The light output was measured each time before the start and at the end of a test.

Specimen Preparation for µTBS Testing

Once the bonded macrospecimens were prepared, they were kept for 1 h at 100% humidity prior to being immersed and stored for 1 week in distilled water at 37°C. After 1-week water storage, all specimens were sectioned perpendicular to the interface using a water-cooled diamond saw (Accutom-50, Struers; Ballerup, Denmark) to obtain
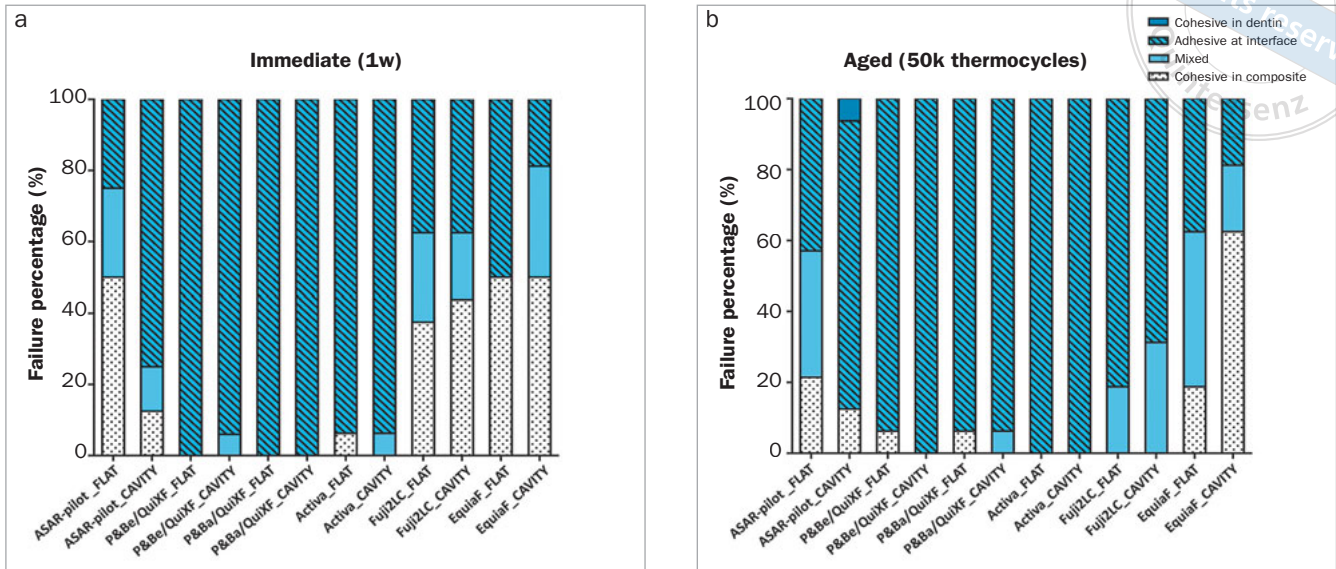
Fig 3 Light microscopy failure analysis of all µ-specimens of the different experimental restorative systems investigated regarding the immediate µTBS in (a) and the aged µTBS in (b).

rectangular sticks (4 central micro(µ)-specimens per tooth: 1 x 1 mm wide; 8-9 mm long). During µ-specimen preparation, alginate was used to support the thin slabs after the first cut in an attempt to prevent pre-test failures. For each experimental group, 16 non-trimmed µ-specimens (2 µ-specimens per tooth) were immediately tested to determine the immediate µTBS. Another 16 µ-specimens were aged for 50,000 thermocycles between two water baths at 5°C and 55°C using a THE-1200 thermocycler (SD Mechatronik; Munich, Germany) prior to testing to determine the aged µTBS (Fig 1). For the µTBS test, the specimens were fixed to a BIOMAT jig with cyanoacrylate-based glue (Model Repair II Blue, Dentsply Sirona Sankin; Tochigiken, Japan) and stressed at a crosshead speed of 1 mm/min until failure in an LRX testing device (LRX, Lloyd; Hampshire, UK) using a load cell of 100N. When specimens failed before actual testing, they were recorded as pre-test failures (ptfs) and included as 0 MPa in calculating the mean µTBS. The whole test protocol followed the Academy of Dental Materials guidelines for µTBS testing.[3]

Fractographic Analysis by Light Microscopy and SEM

After the µTBS test, all fractured specimen halves (both the dentin and restoration sides) were observed under 50X magnification using a stereomicroscope (Stemi 2000-CS, Zeiss; Oberkochen, Germany) to classify the mode of failure as either cohesive in dentin, cohesive in composite, adhesive (interfacial), or mixed.

Representative fractured surfaces exhibiting the most frequently recorded failure mode and a µTBS close to the mean, as well as pre-test failures, were further processed for high-magnification SEM examination (JSM-6610LV, Jeol; Tokyo, Japan). These SEM specimens were fixed using 2.5%

glutaraldehyde, gradually dehydrated in increasing concentrations of ethanol, and chemically dried using hexamethyldisilazane (HMDS, Acros Organics, Thermo Fisher Scientific; Geel, Belgium).[35] Finally, the specimens were thinly gold-coated using a gold-sputter machine (JFC-1300, Jeol). SEM photomicrographs were taken at 85-95X, 2000X, and 9000X original magnification under an accelerating voltage of 5 kV.

Statistical Analyses

Statistical analysis was carried out using SPSS 23 (IBM; Chicago, IL, USA) with statistical significance set at $\alpha = 0.05$. As some µTBS data of the experimental groups revealed unequal variance, Welch's ANOVA with Games-Howell contrast was used to check for statistically significant differences ($p < 0.05$).

RESULTS

µTBS

The bond strengths (including ptf number) are shown in Fig 2. No significant difference in immediate µTBS was recorded when the restorative materials were applied on flat dentin, except for Activa_FLAT and the conventional GIC EquiaF_FLAT, both of which exhibited significantly lower bond strength (for Activa_FLAT mainly due to a high ptf rate). Bond strengths associated with ASAR-pilot were not significantly lower than those of the other restoratives. In addition, when bonded to class-I cavity-bottom dentin, the significantly highest µTBS was recorded for the pre-conditioned resin-modified GIC Fuji2LC_CAVITY (layer-filled), which was statistically different from all other groups but P&Ba/QuiXF_CAVITY. A significantly lower µTBS to cavity-
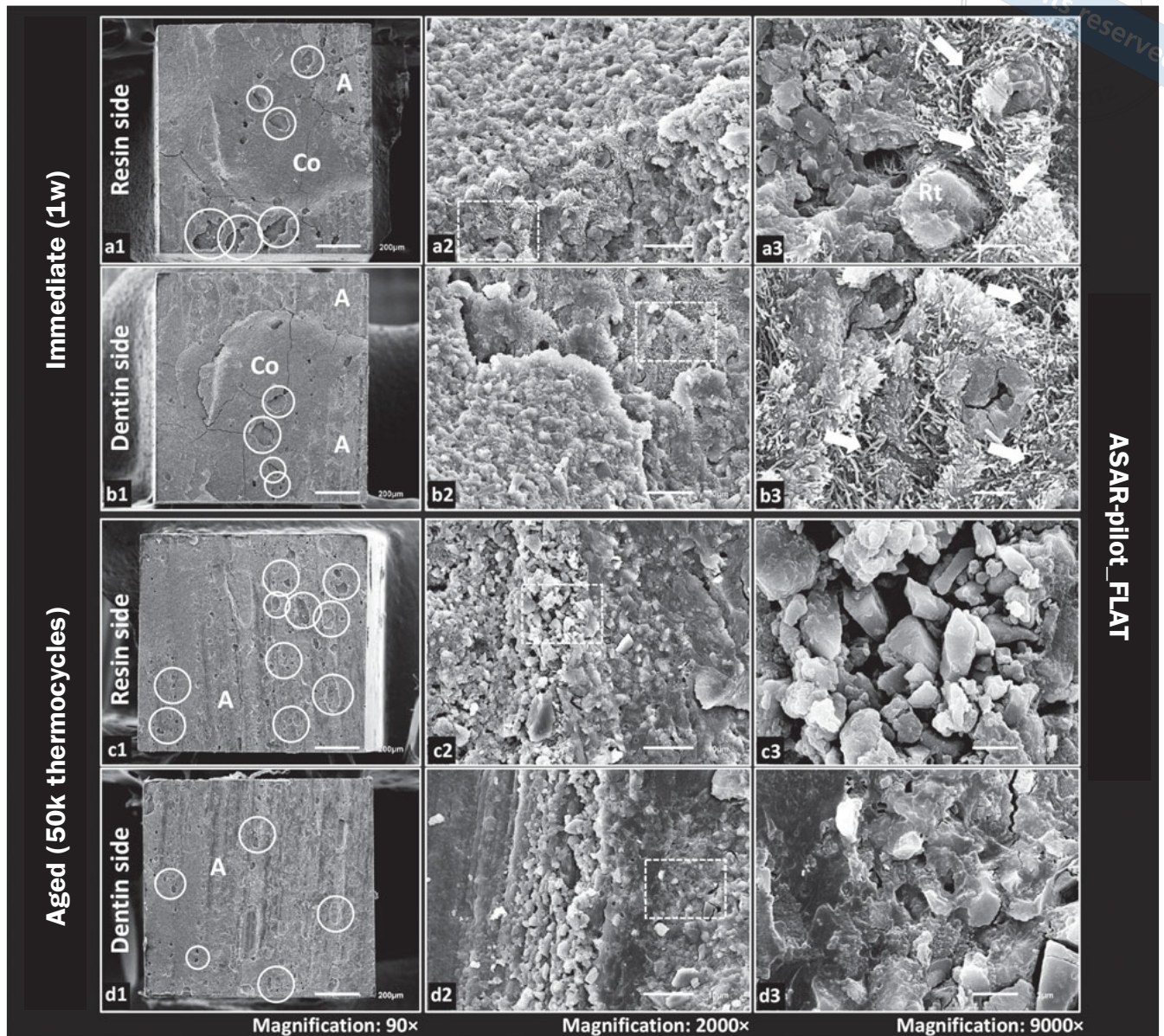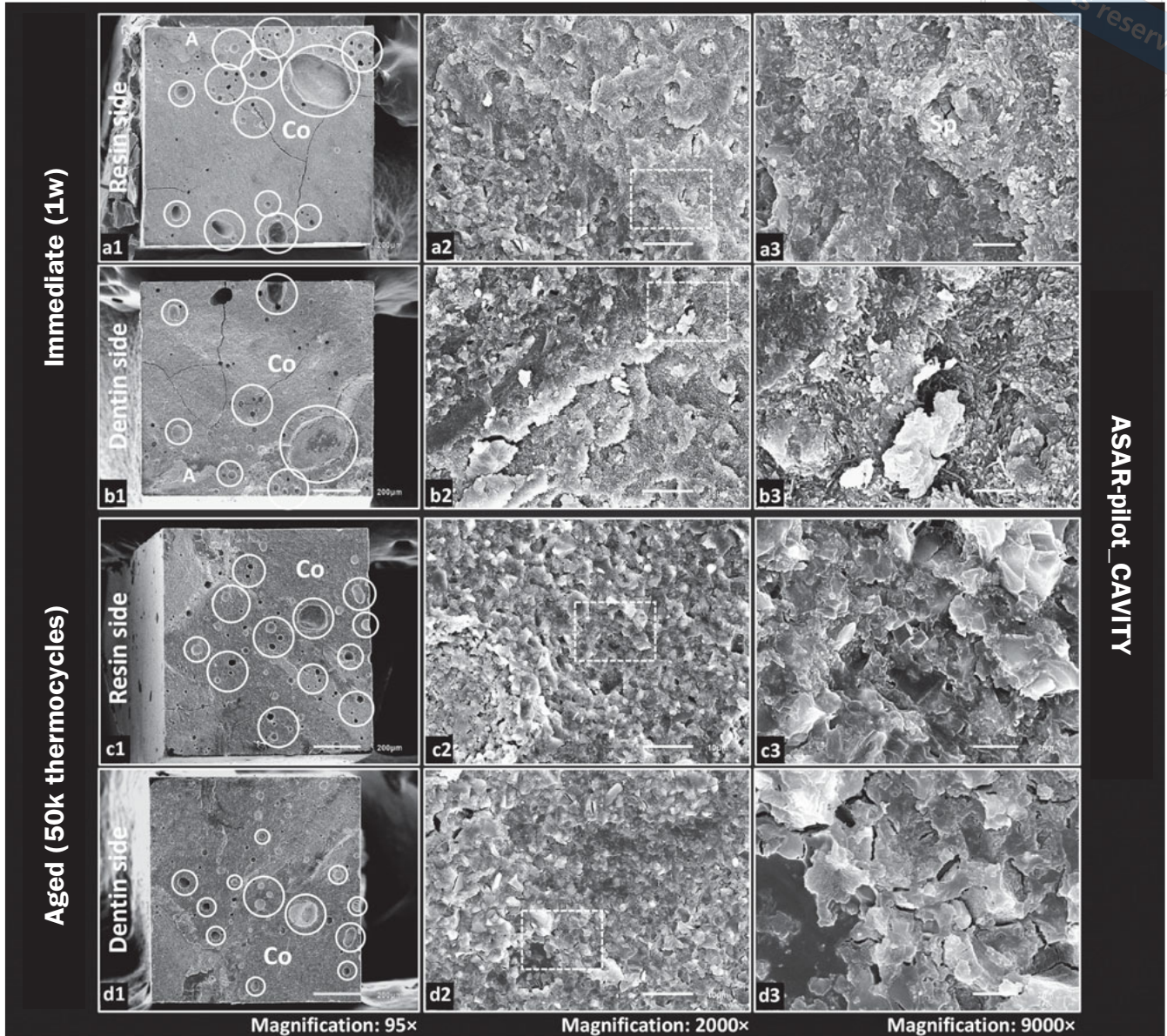
Fig 4 Representative SEM photomicrographs illustrating the fractured sides of ASAR-pilot_FLAT. a1 and b1: Fractured surface (resin and dentin side, respectively) revealing mixed failure mode with ASAR-pilot remnants (Co) attached to the dentin (b1) side, while also some interfacial porosities were found on both the resin (a1) and dentin (b1) side (white circles) (low-magnification image: 90X original magnification). a2 and a3: Higher magnification of the fractured resin side in a1, revealing a structure that may represent a resin tag (Rt) and intertubular collagen fibrils (white arrows). b2 and b3: Higher magnification of the fractured dentin side in b1, revealing intertubular collagen fibers (white arrows). c1 and d1: Fractured surface revealing adhesive (A) interfacial failure mode with diamond-bur scratches clearly observable on both the resin (c1) and dentin (d1) side; also interfacial porosities were exposed, in particular on the resin side in c1 (white circles). c2 and c3: Higher magnification of the fractured resin side in (c1), revealing filler particles in different sizes, ranging from less than 1 µm to around 8 µm. d2 and d3: Higher magnification of the fractured dentin side in d1, revealing bur-cut scratches filled with restorative material. A: adhesive failure; Co: composite; Rt: resin tag.

bottom dentin along with a large number of ptfs was recorded for ASAR-pilot_CAVITY.

Upon aging, the highest aged µTBS to flat dentin was achieved by ASAR-pilot_FLAT, which was not significantly different from P&Be/QuiXF_FLAT and Fuji2LC_FLAT. The significantly lowest aged µTBS to flat dentin was again recorded for Activa_FLAT and EquiaF_FLAT (mainly due to a high ptf rate). Bond strength to cavity-bottom dentin was significantly lower upon aging than to flat dentin for all restorative materials, including ASAR-pilot_CAVITY, but not P&Ba/QuiXF_CAVITY. All P&Be/QuiXF_CAVITY and Activa_FLAT/CAVITY specimens failed prior to testing (ptf).

Fig 5 Representative SEM photomicrographs illustrating the fractured sides of ASAR-pilot_CAVITY. a1 and b1: Fractured surface (resin and dentin sides, respectively) mainly revealing cohesive failure mode in composite (Co), interfacial porosities were also exposed on both the resin (a1) and dentin (b1) side (white circles) (low-magnification image: 95X original magnification). a2 and a3: Higher magnification of the fractured resin side in a1, revealing dentin tubules obstructed by a smear plug (Sp). b2 and b3: Higher magnification of the fractured dentin side in b1. c1 and d1: Fractured surface revealing a cohesive failure mode within composite (Co) and interfacial porosities (white circles). c2 and c3: Higher magnification of the fractured resin side in c1, revealing the size of most filler particles in this area being less than 5 μm. d2 and d3: Higher magnification of the fractured dentin side in d1. A: adhesive failure; Co: composite; Sp: smear plug.

Furthermore, no significant difference between immediate and aged μTBS was recorded for ASAR-pilot when bonded to flat or cavity-bottom dentin. However, ASAR-pilot_CAVITY was associated with a high ptf number, while no ptfs were recorded for ASAR-pilot_FLAT. After aging, P&Ba/QuiXF_FLAT, EquiaF_FLAT, Fuji2LC_CAVITY, and EquiaF_CAVITY presented lower μTBS than when immediately tested (p < 0.05).

Failure Mode Analysis

Failure mode analysis in percentage is graphically presented in Fig 3, which clearly shows that most fractured
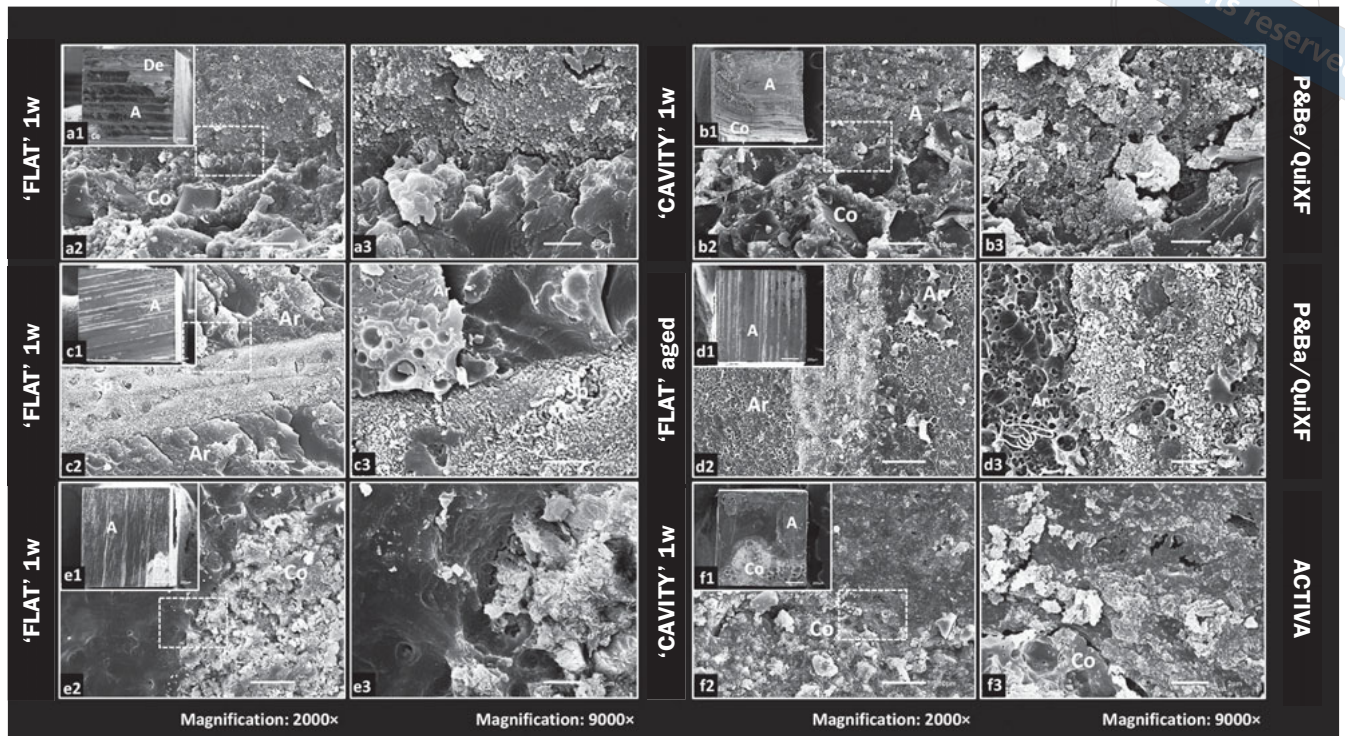
Fig 6 SEM photomicrographs of the fractured dentin side of the experimental restorative systems P&Be/QuiXF, P&Ba/QuiXF and ACTIVA, at low magnification in a-f1 (inserts: 85x original magnification) and at high magnification in a-f2 and a-f3 (2000X and 9000X original magnification, respectively). a/a1: P&Be/QuiXF_FLAT. Immediate fractured surface revealing a predominantly adhesive (A) interfacial failure mode. a2/3: Higher magnification of a1, revealing composite (Co) that remained attached to the dentin surface. b/b1: P&Be/QuiXF_CAVITY. Immediate fractured surface revealing a mixed failure mode which failed partially adhesively (A) at the interface and partially within the composite (Co). b2/3: Higher magnification of b1, revealing the size of most filler particles being around 10 μm. c/c1/c2: P&Ba/QuiXF_FLAT. Immediate fractured surface revealing an adhesive (A) interfacial failure mode. c3: Higher magnification of c1/2, revealing a dentin tubule obstructed by a smear plug (Sp), while also porosities (white circle) within the adhesive resin (Ar) were disclosed. d/d1/d2: P&Ba/QuiXF_FLAT. Aged fractured surface revealing an adhesive (A) interfacial failure mode. d3: Higher magnification of d1/2, revealing porosities (white circles) within the adhesive layer (Ar). e1-3: ACTIVA_FLAT. Immediate fractured surface mainly revealing an adhesive (A) interfacial failure mode with remnants of restorative material (Co). f/f1: ACTIVA_CAVITY. Immediate fractured surface revealing a mixed failure mode. f2/3: Higher magnification of f1, revealing the size of most filler particles being around 3-4 μm. A: adhesive failure. Ar: adhesive resin; Co: composite; De: dentin; Sp: smear plug.

surfaces failed adhesively at the interface. The pre-test failures were predominantly interfacial failures, indicative of very low bond strength. The higher bond strength recorded for ASAR-pilot_FLAT, Fuji2LC_FLAT, and Fuji2LC_CAVITY was associated with a higher percentage of cohesive failures in composite and mixed failures (Fig 3a). Similarly, upon aging, most debonded surfaces represented adhesive interfacial failures, with the exception of ASAR-pilot_FLAT, EquiaF_FLAT, and EquiaF_CAVITY, for which a relatively high percentage of mixed and cohesive failures in composite/GI were also recorded (Fig 3b).

SEM Fracture Analysis

Representative SEM photomicrographs of fractured μ-specimens of ASAR-pilot_FLAT and ASAR-pilot_CAVITY after 1-week water storage (immediate) and 50,000 TC (aged) are shown in Figs 4 and 5, respectively. Immediate ASAR-pilot_FLAT failed partially at the interface and partially within the composite. Aged ASAR-pilot_FLAT failed at the interface. The scratches produced by the diamond bur were filled with the restorative material, which consisted of filler particles with sizes ranging from less than 1 μm up to ca 8 μm (Figs 4c and 4d). However, immediate ASAR-pilot_CAVITY revealed mainly cohesive failures with partially adhesive interfacial failures. Higher magnification photomicrographs (2000X and 9000X) illustrated that dentin remained covered by the hybrid layer and smear plugs, reflecting less effective bonding performance (Figs 5a and 5b). Furthermore, aged ASAR-pilot_CAVITY failed cohesively within the composite, which was accompanied by air bubbles (Figs 5c1 and 5d1, 95X original magnification).

Representative SEM photomicrographs of the dentin side (except Fig 7d: resin side) of fractured μ-specimens representing the different experimental groups (restorative mater-
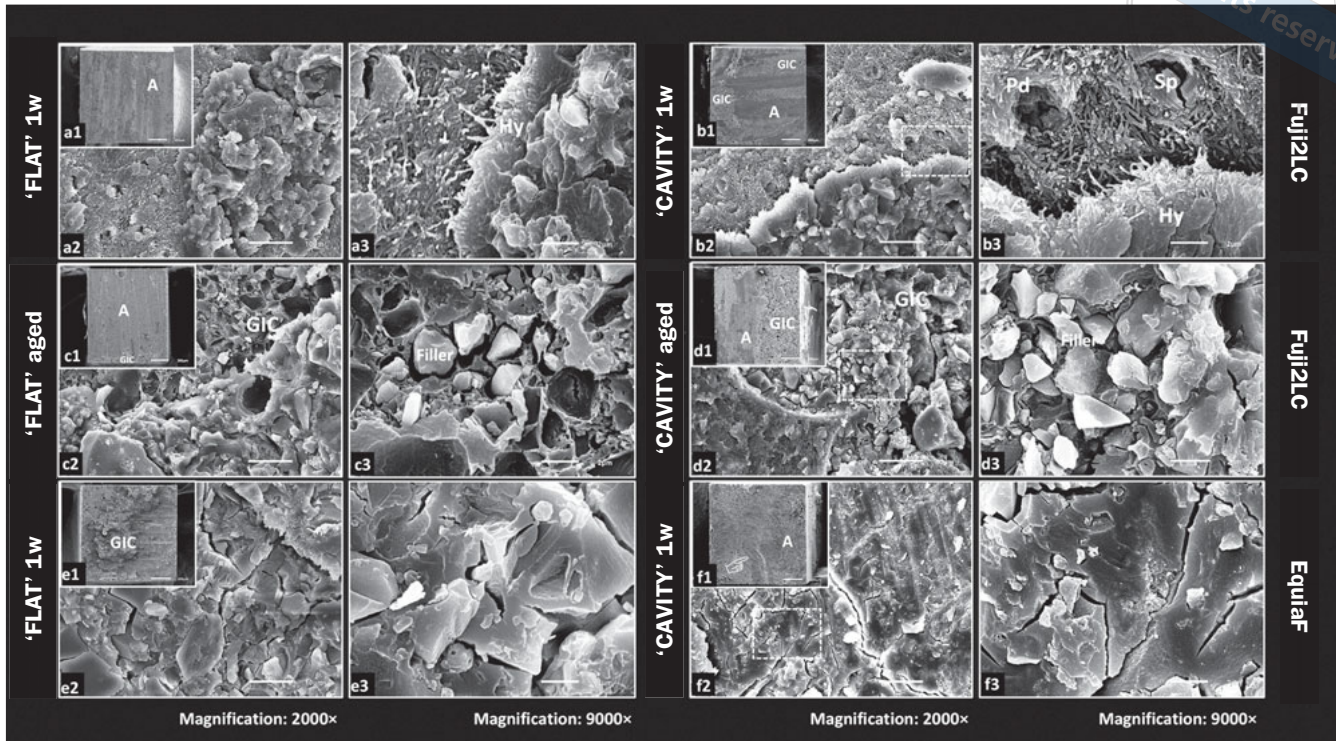
Fig 7 SEM photomicrographs of the fractured dentin side (except for aged Fuji2LC_CAVITY: resin side) of the experimental restorative systems Fuji2LC and EquiaF, at low magnification in a-f1 (inserts: 85X original magnification) and at high magnification in a-f2 and a-f3 (2000X and 9000X original magnification, respectively). a/a1: Fuji2LC_FLAT: Immediate fractured surface revealing an adhesive (A) interfacial failure mode. A typical bur-cut pattern can be observed. a2/3: Higher magnification of a1, most likely revealing the hybrid layer, indicating clear interaction of Fuji2LC with pre-conditioned dentin. b/b1: Fuji2LC_CAVITY. Immediate fractured surface revealing a mixed failure with glass-ionomer cement (GIC) remaining attached onto the dentin surface. b2/3: Higher magnification of b1, revealing smear-plugged (Sp) dentin tubules, peritubular dentin (Pd) and most likely the hybrid layer (Hy). c/c1: Fuji2LC_FLAT. Aged fractured surface revealing an adhesive (A) interfacial failure mode. c2/3: Higher magnification of (c1), revealing filler particles in different sizes, ranging from less than 2 µm to more than 10 µm. d1-3: Fuji2LC_CAVITY: Aged fractured surface (resin side) revealing a mixed failure. e/e1: EquiaF_FLAT: Immediate fractured surface revealing a cohesive failure in GIC. e2/3: Higher magnification of (e1), revealing dehydration artifacts of the water-containing GIC. f1-3: EquiaF_CAVITY: Immediate fractured surface revealing an adhesive (A) interfacial failure mode. Circular scratches represent the bur-cut smear layer produced at the class-I cavity-bottom dentin (f1, 90X original magnification). A: adhesive failure; Hy: hybrid layer; Pd: peritubular dentin; Sp: smear plug.

ials) bonded to flat and cavity-bottom dentin upon 1-week water storage (immediate) or 50,000 TC (aged) are shown in Figs 6 and 7, respectively. Most fractured surfaces (Fig 6) failed at the interface, except for immediate P&Be/QuiXF_CAVITY and ACTIVE_CAVITY (mixed failures). Higher magnification SEM images of aged specimens showed porosities within the adhesive layer for P&Ba/QuiXF_FLAT (Fig 6d3). Interfacial failure was recorded for Fuji2LC when bonded to a polyalkenoic-acid pre-conditioned flat dentin surface (Figs 7a1 and 7c1, 85-90X original magnification). However, a small amount of Fuji2LC remained on the surface, which contained filler particles ranging from less than 2 µm to over 10 µm (Figs 7c2 and 7c3). Also noteworthy are the small cracks present in EquiaF (Figs 7e and 7f, 2000X original magnification), which in the first instance should be regarded as dehydration artifacts of the water-containing GIC. Immediate EquiaF_FLAT failed cohesively within GIC, while

EquiaF_CAVITY failed at the interface. Circular scratches produced during preparation of the standard box-type class-I cavity can be observed (Fig 7f1, 90X original magnification).

DISCUSSION

This study examined the bonding efficacy of K-0180 ASAR pilot (ASAR-pilot), the experimental precursor of a new self-adhesive bulk-fill restorative material that has recently been commercialized under the brand name "Surefil One" (Dentsply Sirona). According to the manufacturer's information, this so-called self-adhesive composite hybrid claims to offer the dentist an innovative filling concept for posterior teeth.[14] Surefil One is claimed to be a forgiving material that combines the simplicity of a glass-ionomer cement (GIC) with the stability of a conventional resin-based composite (RBC)

without sacrificing aesthetic outcome. Key features of the new posterior restorative material are self-adhesiveness and bulk-filling. Considering the product description, the claims made by the manufacturer, and the new material's key features, a representative but diverse group of restorative materials were included in this study (Table 1). As one of the first bulk-fill composites, although not called "bulk-fill" at the time of its market introduction, the RBC QuiXFil (Dentsply Sirona)[15] was applied in this study in one 4-mm bulk increment, as was ASAR-pilot, following manufacturer's instructions. Not being a self-adhesive composite, QuiXFil was applied after adhesive (pre-)treatment with either the universal adhesive Prime&Bond Elect (P&Be/QuixF) or its successor, the universal adhesive Prime&Bond Active (P&Ba/QuixF), both bonded using self-etch mode. While P&Be contains dipentaerythritol pentacrylate phosphate (PENTA), P&Ba contains 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) as functional monomer. Claimed to be a bioactive restorative material with mineralization potential,[39] Activa Bioactive-Restorative (Pulpdent) is described by the manufacturer as a dual-cure resin-based material with GIC components (modified polyacrylic acid, reactive glass filler). According to the original manufacturer's instructions, in this study, Activa was applied in one 4-mm bulk increment in a semi-self-adhesive mode after phosphoric acid-etching the dentin (at the this paper was written, the manufacturer recommended additionally using a bonding agent of the operator's choice). Fuji II LC Improved (GC; Fuji2LC) – a resin-modified GI – was applied according to its manufacturer's instructions in three 1.8-mm layers following polyalkenoic acid (pre-)conditioning. Finally, representing a conventional GIC, which its manufacturer also terms a bulk-fill glass-hybrid restorative, Equia Forte Fil (GC) was applied in this study both following its manufacturer's instructions in 4-mm bulk and in full self-adhesive mode, as the dentin was not pre-treated.

To measure bonding efficacy, microtensile bond strength (μTBS) testing was selected with the six restorative systems applied to low C-factor flat (mid-coronal, bur-cut) dentin vs high C-factor class-I cavity-bottom (mid-coronal, bur-cut) dentin. The latter bonding condition should be regarded as the worst-case condition, especially for restorative materials that are applied in full self-adhesive and bulk-fill mode (ASAR-pilot, EquiaF). The test involving bonding to class-I cavity-bottom dentin has been applied in previous research[10,11,30,40,46] and appeared especially useful to determine bonding efficacy of bulk-fill composites.[44,45]

Finally, the test design also enabled comparison of immediate with aged bonding efficacy. In this study, long-term aging was performed with 50,000 thermocycles, much more than the 500 thermocycles required by the ISO/DTS 11405 standard. Moreover, thermocycling is the most commonly used method to simulate in vivo aging, which results in contraction-expansion stress and hydrolytic degradation at the bonded interface.[12,48] Challenging the bond strength of specimens in vitro by exposing them to long-term water storage or thermocycling is required to predict clinical bond longevity and measure the bonding efficacy of adhesives, self-adhesive cements, and restorative materials.

While a self-adhesive bulk-fill restorative application procedure is the simplest filling technique, approximating that of amalgam, lower bonding efficacy is logically to be expected when no separate adhesive is applied first to provide with better wetting and more intensive adhesive interaction. However, the findings of this study did not reveal significant differences in immediate μTBS when the restorative materials were applied to flat dentin, except for Activa_FLAT and EquiaF_FLAT, which both exhibited significantly lower bond strengths. Therefore, the null hypothesis 1a, that the immediate bonding efficacy of ASAR-pilot to flat dentin would not significantly differ from that obtained when dentin was bonded with the other restorative materials, was accepted. Additionally, there was no significant difference in immediate μTBS when bonded to class-I cavity-bottom dentin, except for Fuji2LC_CAVITY, which exhibited significantly higher bond strength. Hence, null hypothesis 1b, that the immediate bonding efficacy of ASAR-pilot to cavity-bottom dentin would not significantly differ from that obtained when dentin was bonded using the other restorative systems, was accepted for five of the six restorative systems tested. It failed to be accepted regarding the bonding efficacy to cavity-bottom dentin for ASAR-pilot_CAVITY vs Fuji2LC_CAVITY. Upon long-term aging via thermocycling, the aged μTBS of 8 restorative systems/substrates did not significantly decrease compared to the 1-week immediate μTBS, while a significant decrease in μTBS upon aging was recorded for the four restorative systems/substrates P&Ba/QuiXF_FLAT, Fuji2LC_CAVITY, EquiaF_FLAT and EquiaF_CAVITY. Therefore, the null hypothesis 2a and 2b, that bonding efficacy did not decrease upon substantial artificial aging when applied given the two substrate conditions, were accepted except for the four restorative systems/substrates mentioned above.

Regarding ASAR-pilot, no significant difference in μTBS was recorded compared to the highest recorded μTBS to flat dentin, ie, of the pre-conditioned, layer-filled resin-modified GIC Fuji2LC, confirming ASAR-pilot's quite favorable self-adhesiveness. To achieve self-adhesiveness, the restorative material should contain functional monomers to interact with tooth structure, producing microretention by etching and/or realizing primary chemical interaction.[20,31,37] The latter two main bonding mechanisms can only be effective if the primary requirement for adhesion is met: restorative material must adequately wet the surface. According to the composition given by its manufacturer, ASAR-pilot contains polycarboxylic acid, acrylic acid, and bifunctional acrylate. The acrylate monomers ensure the formation of a crosslinked resin network following polymerization reaction and provide increased mechanical strength.[4,22,26] Besides superficial etching resulting in a submicron hydroxyapatite-rich hybrid layer, ionic bonding potential has been attributed to polycarboxyl acid, which constitutes the main bonding mechanisms of (resin-modified) GICs.[28,52] As disclosed by SEM (Figs 4a and 4b), exposed collagen fibers can be clearly seen, suggesting that ASAR-pilot may have etching potential. Nevertheless, to elucidate whether ASAR-pilot's self-adhesiveness should be

ascribed to both micromechanical interlocking through etching along with primary chemical bonding, interfacial TEM characterization combined with chemical XRD and NMR analysis should be performed in further studies.

However, the µTBS of ASAR-pilot dropped significantly when bonded to class-I cavity-bottom dentin (ASAR-pilot_CAVITY); a high proportion of pre-test failures (ptf) was observed in that group. This is in agreement with many previous studies which found lower bond strength when bonded to class-I cavity-bottom dentin vs a flat dentin surface.[33,57] The differences can be ascribed to many factors, the most likely being the cavity-configuration factor (C-factor). Polymerization shrinkage stress within cavities is well known to be largely affected by the C-factor.[18] As calculated in a previous study by Van Ende et al,[44] the C-factor of flat dentin with the same dimensions (3.5 x 3.5 x 4 mm) as in this study is 0.18, while that of the class-I cavity (3.5 x 3.5 x 4 mm) is 5.8. This significantly greater C-factor restricted the free flow of the polymerizing and shrinking resin-based material, since more material was constrained by being bonded to the rather stiff cavity walls.[27] Resin-based material can shrink nearly unrestrictedly when bonded to a flat dentin surface.[30] Therefore, more shrinkage stress could have acted on the bond to cavity-bottom dentin, correlating positively with a higher proportion of interfacial gaps[21] and affecting bonding efficacy. Previously, in similar experiments investigating bonding to high C-factor class-I cavity bottom dentin, only the bonding efficacy of the bulk-fill flowable composite SDR (Dentsply Sirona) appeared insensitive to C-factor, while other bulk-fill composites with a paste-like consistency were affected to a much greater extent by high shrinkage stress generated in the high C-factor cavity.[25,45]

Besides the effect of C-factor, another plausible factor that may have contributed to the significant differences between FLAT and CAVITY results for ASAR-pilot may be dentin moisture control. Although containing water, the self-adhesive bulk-fill composite hybrid ASAR-pilot requires some moisture to activate the functional acids it contains.[8] Consequently, according to the manufacturer, the dentin surface should not be desiccated. However, it is much more difficult to control surface moisture in a deep, narrow cavity than on a flat surface, making it difficult to achieve the optimal degree of moisture on the surface.[11] Furthermore, using a relatively high-viscous bulk-fill composite, surface wetting could have been compromised by porosities formed at the bonded interface. Intratubular and inter-collagen infiltration is more difficult for a material with a paste-like consistency compared to the infiltration ability of liquid adhesive solutions.

The first bulk-fill RBC QuixF combined with the self-etch adhesive P&Be resulted in a relatively high µTBS when bonded to flat dentin (QuixF/P&Be_FLAT). No significant reduction in µTBS to flat dentin was recorded upon 50,000 thermocycling. However, when bonded to cavity-bottom dentin (QuixF/P&Be_CAVITY), almost all µ-specimens failed before testing (ptfs), indicating that the polymerization shrinkage stress developed within the high C-factor cavity must have led to interfacial debonding at the cavity bottom. Interestingly, when QuixF was combined with the self-etch univer-

sal adhesive (successor) P&Ba, significantly better bonding efficacy was recorded in the class-I cavities (QuixF/P&Ba_CAVITY). Compared with the PENTA-containing universal adhesive P&B Elect, P&B Active contains the functional monomer 10-MDP. Considering its chemical structure with five methacrylate groups vs one phosphate group, PENTA should be regarded as a crosslinking monomer rather than a monomer with good ionic bonding potential to the Ca of hydroxyapatite. The latter interaction potential of PENTA's phosphate group may even be expected to be sterically hindered by the surrounding methacrylate groups.[1,47] However, much research has demonstrated that the functional monomer 10-MDP is one of the best-performing functional monomers,[54,55] which would explain why most of the newest generation of universal adhesives contain 10-MDP.[32,36] Besides etching capability, which provides surface microretention, and the primary ionic bonding potential to Ca of hydroxyapatite, 10-MDP has been documented to uniquely self-assemble into nanolayers of stable 10-MDP/Ca salts.[53,56] This difference in functional monomer is the most plausible explanation for the superior bonding efficacy of QuixF/P&Ba vs that of QuixF/P&Be recorded in this study. Furthermore, the manufacturer claimed that P&Ba is well balanced in terms of hydrophobic/hydrophilic properties, promoting surface wetting and resin infiltration under various moisture conditions.

Activa's bioactive ionic resin is claimed to facilitate diffusion of calcium, phosphate, and fluoride ions across the restoration-dentin interface,[8,58] hence stimulating hydroxyapatite formation and remineralization at the bonded interface. This claimed interfacial bioactivity, for which no hard (independent) evidence had been published at the time of writing this manuscript, can only work if the restorative material makes direct contact with the dentin substrate, thus requiring self-adhesiveness. Pre-etching with phosphoric acid, as originally recommended by Activa's manufacturer, will not prevent the claimed interfacial bioactivity. However, the newly released application instructions to additionally use a bonding agent will block direct Activa-dentin contact, making any interfacial bioactive interaction very questionable. The adapted instructions for use, now also requiring the (pre-)application of a separate adhesive, were released because of recently documented insufficient self-adhesiveness of Activa. A randomized clinical trial indeed revealed that the use of Activa in class-I/II cavities, applied as instructed by the manufacturer after a short phosphoric-acid pretreatment without adhesive, resulted in an inacceptable, very high failure frequency after one year of clinical service.[42] The authors concluded that further studies involving Activa should be conducted using a bonding agent. The present study confirmed the insufficient self-adhesiveness of Activa by the significantly lowest µTBS along with a very high incidence of pre-test failures (only ptfs upon aging) recorded when Activa was bonded both to pre-etched flat and cavity-bottom dentin. Although alginate was used to support the thin slabs after the first cut during µ-specimen preparation, many Activa µ-specimens failed during the second cutting action perpendicular to the first. Reasons for this

inferior bonding performance of Activa must be multifactorial. The most plausible explanation is Activa's weak self-adhesiveness to dentin, considering the low μTBS and high ptf incidence recorded, even when Activa was bonded to flat dentin (in contrast to all other restorative systems investigated, except for EquiaF). In addition, the initial bond strength of Activa could not withstand the polymerization shrinkage stress developed in the class-I cavity. Hence, apart from developing materials with potential bioactive properties, primary properties such as mechanical strength and bonding potential remain essential.

Overall, in terms of bonding efficacy, the best-performing restorative system in this study was Fuji2LC. When bonded both to flat and class-I cavity-bottom dentin, a high immediate and aged μTBS were recorded for the pre-conditioned resin-modified GIC applied in incremental layers. Our finding is in accordance with previous research which showed that an incremental layer-filling technique reduces contraction stress and improves adhesion to cavities with tight internal adaptation.[2,5,34] As a resin-modified GIC, Fuji2LC's self-adhesiveness should be attributed to combined micromechanical interlocking within a submicron hydroxyapatite-rich hybrid layer and primary chemical bonding of carboxylic groups with calcium in hydroxyapatite.[24,28] Previous research also reported that GIC is more capable of reducing contraction stress during early setting than RBC, increasing the possibility of a durable bond to the cavity walls.[9]

Less favorable bonding efficacy was recorded for the conventional GIC EquiaF, which was applied to non-conditioned dentin. The low μTBS to both flat and class-I cavity-bottom dentin should most logically be attributed to cohesive failure within the GIC rather than to actual bond failure, as this is typical of GICs when subjected to bond strength testing.[6,23] However, failure mode analysis in this study revealed that most μ-specimens failed interfacially and in mixed mode for aged EquiaF_FLAT. Other reasons for the low bond strength of EquiaF are that 1. dentin was not pre-etched, providing less effective micromechanical interlocking along with potential smear-layer interference; 2. the restorative was solely self-cured, potentially having reached lower cohesive strength; 3. the resin-based coating agent Equia Forte Coat (GC) was not used. Equia Forte Coat (GC) is a nanofilled resin coating agent with high hydrophilicity and low viscosity, which not only fills surface cracks and porosities, but also protects early setting against the outer aqueous environment.[29] Additional application of a resin coating has previously been shown to increase abrasion and erosion resistance, provide protection against water sorption, and increase mechanical strength.[16,29,59]

CONCLUSION

In this study, we investigated the bonding efficacy and bond durability of the new self-adhesive bulk-fill composite hybrid K-0180 ASAR pilot (later commercialized as Surefil One, Dentsply Sirona) to flat and class-I cavity-bottom dentin. When applied on flat dentin, favorable immediate μTBS was

recorded for ASAR-pilot; its self-adhesiveness to flat dentin resisted aging by 50,000 thermocycles. However, ASAR pilot suffered from ptfs when bonded in the worst-case scenario to high C-factor class-I cavity-bottom dentin, although upon aging, ASAR pilot did not perform worse than the other restorative systems investigated.

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Clinical relevance: A favorable bonding performance can be achieved when the new self-adhesive bulk-fill composite hybrid ASAR-pilot is bonded to flat dentin. However, its clinical use in deep cavities must be studied further.