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Pre-cure contact time and dentin surface roughness in modulating bonding and impregnation of self-adhesive composites to dentin

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Abstract

Background Self-adhesive flowable resin composites (SAFRCs) offer simplified, single-step placement and are intended to reduce technique sensitivity, but their bonding remains largely inconsistent. Therefore, this study investigated whether pre-cure contact time and dentin surface finish affect immediate bond strength and interfacial morphology of two commercial SAFRCs.

Materials and methods Sound human molars were allocated to 12 groups: two SAFRCs (Vertise™ Flow; Constic) × two finishes (fine vs. coarse diamond) × three pre-cure contact times (immediate/0 min, 1 min, 5 min). For positive controls a universal adhesive/conventional composite were applied. Three 2 × 2-mm cylinders/group were bonded to mid-coronal dentin discs and were light-cured (20 s; 950 mW/cm^2) and tested in shear bond strength setup (SBS; 24 h at 1 mm/min). One fractured disc/group was examined by SEM and ~ 1-mm interface slices underwent Masson's trichrome and were imaged at 100× with calibrated scaling. The 12 SAFRC groups were analyzed by three-way ANOVA with Tukey's ($\alpha = 0.05$); comparisons with the matched controls were performed separately using one-way ANOVA followed by Dunnett's test.

Results Material ($p = 0.02$) and application time ($p < 0.0001$) significantly affected SBS; surface finish did not ($p = 0.97$). Five-minute pre-cure contact yielded the highest SBS for both SAFRCs and, in several groups, values not significantly different from the matched controls. SEM showed an application time-dependent improvement of the interface quality: with 0 min application showing no continuous interfacial resin/tubule tags; from 1 to 5 min, progressively continuous resin fronts with tags, most evident at 5 min, irrespective of finish. Masson's trichrome revealed a more conspicuous dark-pink collagen band at the interface of immediate cure samples and attenuated/discontinuous after 1–5 min. Surface finish mainly altered interfacial contour without qualitative improvement.

Conclusion Prolonged pre-cure contact enhances SAFRC bonding; the tested roughness range was not determinative. Trichrome staining corroborates shallow demineralization with partially exposed collagen, indicating limited monomer infiltration and explaining the material-dependent, low bond efficacy of current SAFRCs.

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Keywords FE-SEM, shear bond strength, self-adhesive, self-adhesive composite

Introduction

Adhesive dentistry transformed restorative care by enabling minimally invasive cavity preparations and micromechanical/chemical bonding of resin composites to enamel and dentin. From Buonocore's 1955 acid-etch concept for enamel to contemporary multi-mode adhesives and self-adhesive flowable resin composites (SAFRCs), the field has been marking a pursuit for simplified adhesion with fewer clinical steps and substantially lower technique sensitivity [1, 2]. These advances matter since adhesive failure remains a major driver of restoration replacement, with associated biological and economic costs for patients and health systems [3]. In fact, multiple laboratory and clinical studies indicate that current SAFRCs generally produce lower bond strengths to dentin [4], more adhesive failures, and less interfacial infiltration and adaptation than conventional systems used with a separate adhesive step [4, 5]. Transmission electron microscopy shows limited interaction of SAFRCs with smear-covered dentin and aprismatic enamel [6, 7]; mechanically, the relatively high viscosity and lack of volatile solvent further restricts monomer diffusion into the poorly-conditioned substrate [7, 8].

Two substrate- and technique-related variables seem particularly consequential for self-adhesive technology, when bonding to dentin is considered: (i) the nature/roughness of the prepared surface (and the smear layer it produces) [1, 9] and (ii) the way the material is applied (time, brushing/agitation) [10–12]. Smear layer thickness and roughness are shaped by abrasive grit or bur type and this alters dentin permeability, surface free energy, and the ease with which acidic monomers condition and infiltrate the substrate; for self-etch/"self-adhering" strategies, thicker, compact smear layers typically impair interaction and durability [6, 9, 13–15]. In parallel, there is consistent evidence that *how long* and *how actively* an adhesive/cement is applied, affects chemical interaction, etching efficacy and bond development. In fact, these findings are repeatedly demonstrated for universal/self-etch adhesives on both enamel and dentin [10]. Functional monomers, following the adhesion-decalcification theory, rely on time and surface conditions to effectively bond to the substrate, particularly in limited concentrations [1, 16, 17].

But while the literature on universal/self-etch adhesives clearly shows that surface roughness/smear layer and application time/agitation can meaningfully shift performance, analogous evidence for SAFRCs is sparse. Several in vitro studies comparing commercials such as Vertise™ Flow or Constic versus conventional systems report significantly lower dentin bond strengths for SAFRCs, with limited or inconsistent data isolating the effects of dentin roughness or manipulation time for SAFRCs per se [18–20]. For example, Rangappa et al. (2018) found that bur type influenced the shear bond strength of Dyad Flow and a conventional flowable but not Constic; application time was however, fixed [19]. Mine et al. demonstrated poor interfacial hybridization of a SAFRC on smear-covered dentin by TEM, but did not vary the duration or specifics of SAFRC application [7]. Clinical trials and short-term evaluations similarly suggest inferior retention or mixed outcomes for SAFRCs in small Class I or sealant-like indications [21, 22], yet they seldom report or control the substrate roughness and do not test extended/active manipulation times tailored to SAFRC chemistry. Grit-stratified datasets do not yet exist for self-adhesive materials. Furthermore, monomer-dependent responses (e.g., GPDM- vs. MDP-based) are also absent [23]. For universal adhesives, prolonging application time and/or applying actively (scrubbing) improves dentin interaction and bond strength in many systems [11, 24, 25]. Whether analogous time/activation effects can compensate for the lack of a dedicated primer/adhesive in SAFRCs, and whether such effects depend on dentin roughness, has not been systematically tested.

Therefore, this study evaluates commercial SAFRCs applied to dentin surfaces prepared with clinically relevant abrasives that generate distinct smear-layer morphologies, under controlled application times. Shear bond strength metrics and characterization of the resin–dentin interface by light and scanning electron microscopy, allowing structure–property inferences, was undertaken. The following hypotheses were tested: (i) increasing SAFRC application time and/or actively scrubbing the material improves dentin bond strength and interfacial infiltration compared with the default handling recommended by manufacturers; (ii) dentin prepared with fine diamond burs (thinner, less compact smear layers) yields higher bond strength and more favorable interfacial morphology than dentin prepared with coarser diamond burs; and (iii) there is a significant interaction between application time and surface roughness such that extended application partially offsets the effects of a coarse bur. These aims directly address the identified gaps.

Materials and methods

Study design, factors and materials

This laboratory study used a full-factorial design to test how dentin surface roughness (2 levels), application time for a self-adhering flowable composite (SAFRC; 3 levels), and type of SAFRC, affect resin–dentin bonding

and interfacial characteristics. The primary outcome was shear bond strength at 24 h; As qualitative, complementary endpoints to contextualize mechanical findings, secondary outcomes were fracture interface morphology observation and histochemical collagen exposure staining via Masson's Trichrome, thereby providing mechanistic plausibility for time-dependent effects.

Resin composites and adhesives used are shown in Table 1 and the factors/levels are described below:

- Surface preparation (dentin roughness):
- (R) coarse-grit black diamond bur (≈125–150 μm),
- (F) fine-grit yellow diamond bur (≈25–30 μm),
- SAFRC application time: ~20 s manufacturer default (T0); 1 min (T1) and 5 min prolonged application time (T5).

Specimen collection and allocation

A total of 130 sound human molars without caries, structural defects, and free of restorations were used (as approved by Egas Moniz School of Health & Science Ethics Committee; internal process no. 1342). Teeth were cleaned of soft tissues/debris, disinfected in 1% chloramine-T for one week, then stored in distilled water at 4 °C (changed weekly) until testing. Specimens were then allocated a priori by outcome. For shear bond strength (SBS) testing, the tooth/disc was the experimental unit; 70 teeth/discs were assigned to 14 groups (12 SAFRC conditions plus two positive-control groups), with $n = 5$ teeth/discs per group. After SBS testing, SEM was performed on one representative fractured disc per group (total 14 discs), selected from the SBS specimens. For Masson's trichrome, a separate set of 60 teeth was assigned to the 12 SAFRC groups ($n = 5$ teeth/group). Each tooth was sectioned to obtain two ~ 1-mm slices encompassing the resin–dentin interface (≈ 10 slices/group), and nine slices per group were randomly selected for staining. Remaining slices served as reserves or were excluded if damaged during preparation.

Table 1 Materials and reagents (composition as supplied by manufacturers)

Material	Manufacturer (city, country)	Chemical compositition*[1]	Shade	Lot
Vertise™ Flow (SAFRC)	Kerr Italia S.r.l. (Scafati, Italy)	**Organic Matrix**: HEMA 5–10%, UDMA 5–10%, GPDM 1–5% **Fillers**: Ytterbium fluoride + barium aluminosilicate (≈ 70 vol%)	A2	11,043,458
Constic (SAFRC)	DMG Chem-Pharm. Fabrik GmbH (Hamburg, Germany)	**Organic Matrix**: Bis-GMA 15–35%, TEGDMA < 45%, 10-MDP n/a **Fillers**: Barium aluminosilicate (≈ 66%);	A2	292,495
Charisma® Diamond (nanohybrid composite)	Kulzer GmbH, Hanau, Germany	Fillers: barium aluminosilicate + silica (≈ 64%); matrix: UDMA n/a, TEGDMA n/a	A3	66,044,522
Clearfil™ S'Bond Universal Quick (adhesive)	Kuraray Noritake, Okayama, Japan	10-MDP, Bis-GMA, HEMA (universal adhesive)	—	—

*1 Information derived from safety datasheets and manufacturer supplied product details

Specimen preparation

Crowns were sectioned perpendicular to the long axis under water cooling using a diamond saw (Accutom-50, Struers, Denmark) to obtain mid-coronal dentin surface discs 2 mm thick (first cut ≈ 3 mm below cusp tips; second cut 2 mm apical to the first). Discs were mounted with their long axis parallel to bur axis for standardized surface finishing. Dentin surfaces were standardized using diamond burs of two grit sizes (Edenta, Switzerland): a black-ring X-coarse round-end taper bur (SG850/314 – 018; ≈180 μm) and a yellow-ring X-fine needle bur (C859/314 – 012; ≈20 μm), both in FG shank (314). Burs were replaced every six preparations to minimize the effect of bur wear.

For group allocation (shown in Fig. 1) and restorative procedures, fourteen groups were created (n distributed at the tooth level):

- Experimental (12 groups): two SAFRCs (Vertise™ Flow; Constic) × two surface finishes (fine vs. coarse) × three application times prior to light curing (20 s, referred to as 0 min or immediate, 1–5 min).
- Positive controls (2 groups): Charisma® Diamond applied over the same fine or coarse finish with Clearfil™ S'Bond Universal Quick, was used in self-etch mode (without prior phosphoric acid etching). This mode was chosen to ensure a clinically relevant and methodologically aligned comparison with the SAFRCs. In this case, the whole surface of dentin was prepared with the adhesive and then the composite was bonded using the same mold geometry as reported in 2.3.

Shear bond strength testing (SBS)

Although microtensile bond strength (μTBS) is widely regarded as the gold standard, it was not viable for SAFRCs, which develop relatively low bond strengths; the serial sectioning required to prepare specimens frequently induces pretest failures in weak bonds. For the same reason micro-shear (μSBS) is impractical as

Fig. 1 Schematic of the twelve experimental groups according to the two surface finishes and three application times

sub-millimetric bonded areas are only feasible when an adhesive layer covers the whole substrate and a separate composite rod is bonded onto, thus making micro-cylinder fabrication from SAFRCs would be failure-prone. We therefore adopted a shear bond strength (SBS) setup. A custom CAD/CAM polyacetal mold with three cylindrical wells (internal Ø 1 mm; height 2 mm) was positioned on each dentin disc. For the experimental SAFRC groups, the composite was placed into each well and maintained in contact with dentin for 20 s, 1 min, or 5 min before light curing, according to group allocation. For the positive controls, Clearfil™ S'Bond Universal Quick was applied to dentin in self-etch mode according to the manufacturer's instructions, air-thinned, and light-cured. Charisma® Diamond was then placed into the mold wells and light-cured using the same specimen geometry and irradiation conditions.

Specimens were light-cured in two 20 s exposures (total 40 s) at 0-mm distance, using acetate sheets as stops to stabilize the tip; reported tip irradiance was 950 mW/cm² minimum (420–480 nm), using Elipar Deep-Cure S (3 M ESPE, St. Louis, USA). Molds were removed after curing, and discs were stored in distilled water at 37 °C for 24 h. Each disc (with three composite cylinders) was mounted in an acrylic cylinder that was placed on a universal testing machine (Autograph AG-X, Shimadzu Corporation, Tokyo, Japan). A pre-formed 0.010 orthodontic wire loop was placed flush around each cylinder, aligned parallel to the composite–dentin interface. Load was applied at 1 mm/min until failure; three cylinders per disc were tested and in total five discs per experimental group. Failure patterns were observed under a stereomicroscope (Leica EZ2, Leica Microsystems, Wetzlar, Germany).

Scanning electron microscopy (SEM)

To image the interface, one randomly selected disc with fractured interfaces, per group (after SBS testing), underwent SEM following an adapted protocol described by Perdigão et al. (1996) [26]. Because conventional high-vacuum SEM requires dehydration, the protocol is intended to expose resin infiltration patterns rather than preserve the native hydrated collagen network; thus SEM findings were interpreted qualitatively only as qualitative fracture-plane and interfacial remnant morphology. They were not used to directly measure the native bonded interface, hybrid layer thickness, or true infiltration depth. The following was undertaken: fixation in 2.5% glutaraldehyde at 4 °C for 24 h; rinsing in 0.1 M sodium cacodylate buffer (pH 7.3) with three solution changes; deproteinization/demineralization in 0.1 M HCl (10 min) then 13% NaOCl (2 min); graded ethanol dehydration (70% 20 min; 95% 20 min; 100% 60 min); hexamethyldisilazane (two 10-min baths) and 24 h air drying. Specimens were mounted with the surface up and sputter-coated with ≈ 20 nm Au/Pd, then imaged in a JEOL JSM-5400 at 25 kV, 16 mm working distance, at low/high magnifications (500× and 1500×).

Masson's trichrome for resin-dentin interface observation

Two ~ 1-mm-thick slices were obtained per tooth specifically for light microscopy ($n = 9$ slices/group, as explained in 2.2), oriented to include the resin–dentin interface. Slices were fixed to cylindrical mounts with cyanoacrylate and ground/polished on a bench polisher (Labo-Pol-4, Struers) using progressively finer SiC papers (320, 600, 1200, and 4000 grit) to approximately 100 µm thickness and subsequently stored in mineral oil until staining.

Masson's trichrome staining followed a protocol adapted from Inglês et al. (2022) [8]: removal of mineral oil in xylene; graded ethanol dehydration (70%, 90%, 100%; 5 min each); staining in Solution B (1% acid fuchsin in 1% acid; 4 min), brief 1% acetic acid (10 s) and distilled water rinse (15 s); mordant/differentiation in Solution C (1% phosphomolybdic acid; 40 min) followed by 1% acetic acid (10 s) and rinse (15 s); contrast in Solution D (2% light green in 1% acetic acid; 5 min) and final 1% acetic

acid (30 s) with a 60 s distilled water rinse. Stained slices were mounted on glass slides for immediate imaging.

Images were captured at 100× using an optical microscope (CX41RF, Olympus, Tokyo, Japan). Image scale was calibrated at 100× by measuring a reference object, computing pixel size (µm/pixel), and applying a custom scale in ICMeasure. Images were treated using Image J 1.54 [27].

Statistical analysis

Sample-size guidance followed Academy of Dental Materials recommendations for micro-tensile testing (minimum 5 teeth/group), adopted here with the tooth as the experimental unit [28]. Three-way ANOVA evaluated the effects of composite (Vertise™ Flow vs. Constic), surface finish (fine vs. coarse), and application time (T0, T1, T5), with Tukey's post-hoc ($\alpha = 0.05$). Because the positive-control protocol did not vary application time, controls were not included in the factorial ANOVA. Therefore, benchmarking against controls was performed separately using one-way ANOVA within each surface-finish stratum (F and R), followed by Dunnett's post-hoc test comparing each SAFRC condition with its corresponding control. Pre-test failures, when existent, were counted as half of the lowest registered SBS value, of each experimental group. Analyses were performed in Origin 2023b (OriginLab Corporation, Northampton, MA USA).

Results

Shear bond strength

SBS was significantly influenced by material and application time, but not by surface finish. The three-way ANOVA detected a main effect of self-adhering flowable composite (SAFRC: Vertise™ Flow vs. Constic; $F = 5.6$, $p = 0.02$) and a strong main effect of application time (0, 1, 5 min; $F = 43.1$, $p < 0.0001$), whereas the dentin finishing protocol (fine vs. coarse) showed no effect ($p = 0.97$). Increasing the application time doubled to tripled SBS across SAFRCs; Vertise™ Flow yielded higher mean SBS overall. Means and standard errors are shown in Fig. 2

When groups were benchmarked against the matched positive control within each surface-finish stratum (F groups vs. Control_F; G/coarse/rough groups vs. Control_G) using one-way ANOVA within finish followed by Dunnett-adjusted comparisons, the largest deficits were observed at the immediate (0) application-time conditions. In the fine finish, both VF_0_F (2 ± 0.4 MPa) and CT_0_F (2 ± 0.7 MPa) were significantly lower than the corresponding control (5 ± 0.4 MPa) (both $p = 0.001$),

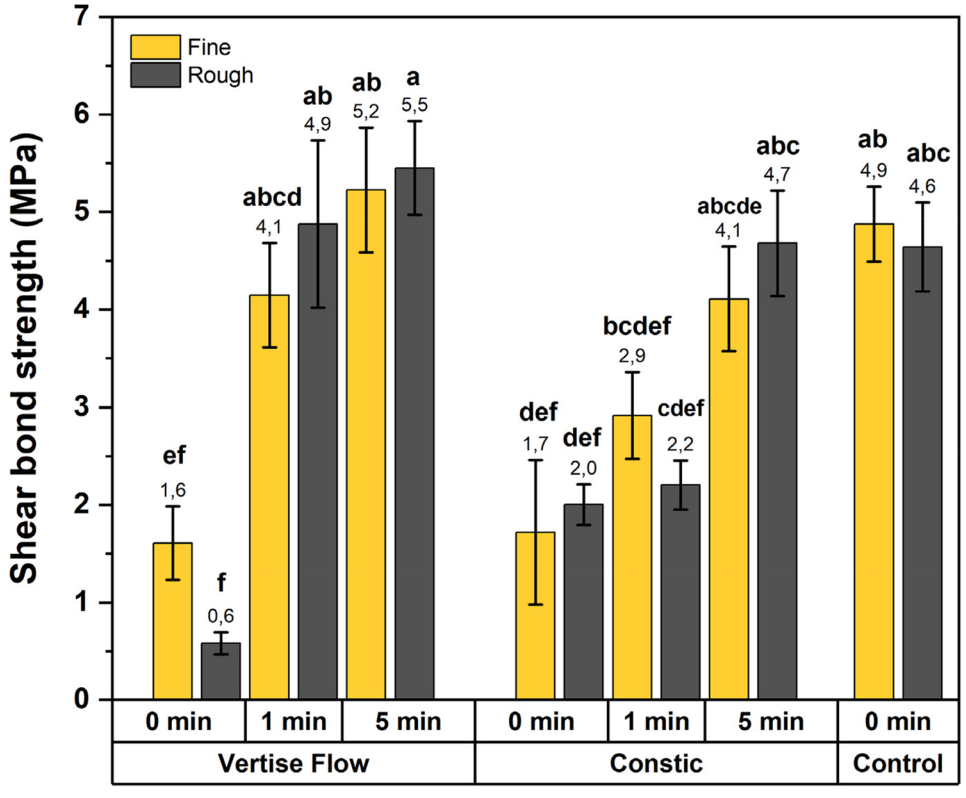

Fig. 2 Mean ± SE SBS (MPa) to dentin for VF and CT at 0 min (20 s application time), 1 min, and 5 min and control groups; colors separate by fine/rough surface preparation. Different letters denote significant differences among groups (Tukey, $\alpha = 0.05$). Extended times (1–5 min) frequently reached values statistically comparable to controls, whereas almost immediate curing (0 min) yielded the lowest performance

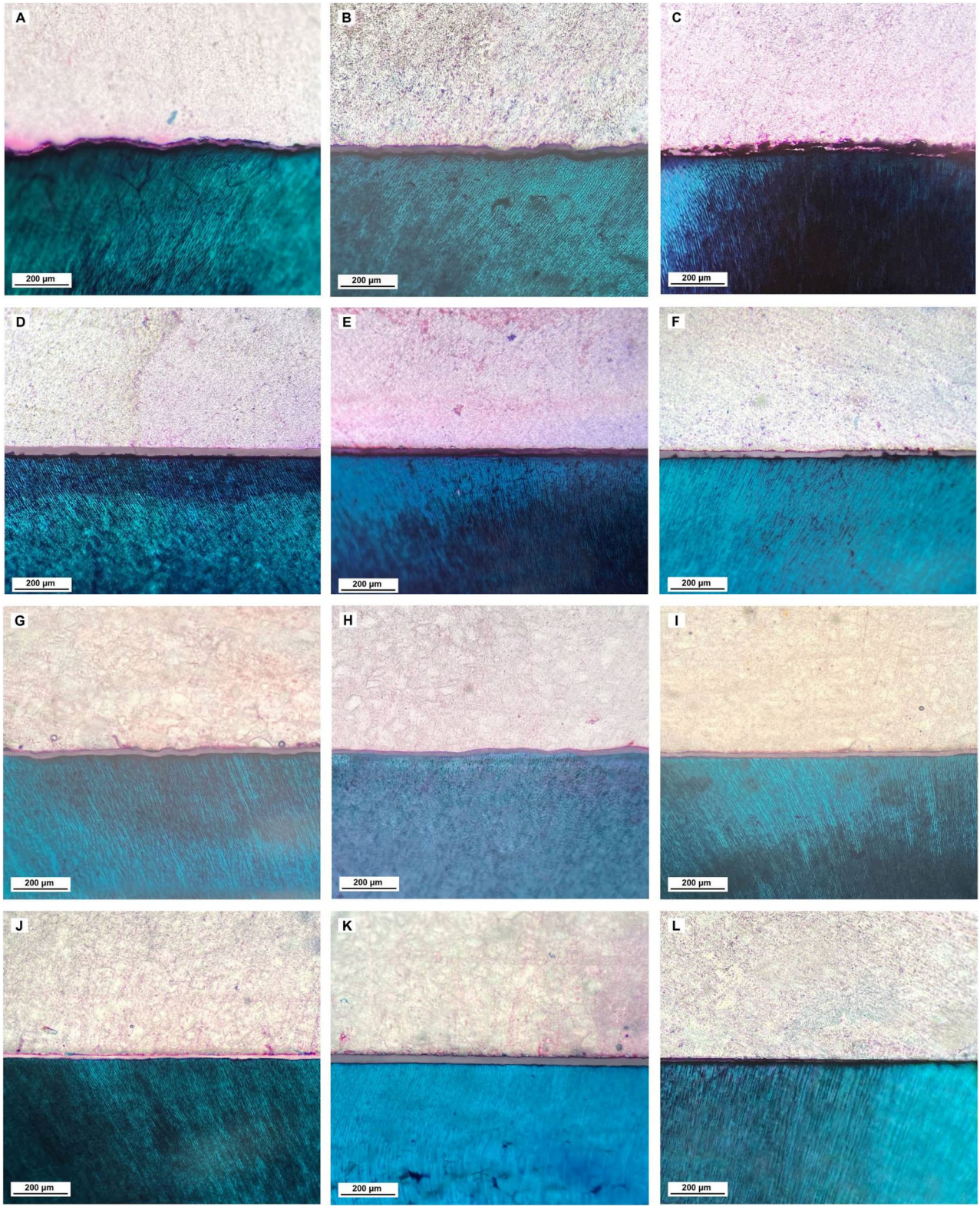

Fig. 3 (See legend on next page.)

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Fig. 3 Masson's trichrome of the resin–dentin interface (100×; representative micrographs). Composite is upper zone; dentin is lower. Mineralized dentin stains blue/blue-green; the self-adhesive composites stain beige/pale pink; a dark-pink line indicates exposed, non-encapsulated collagen. Panels are arranged by material/finish (rows) and pre-cure contact time (columns): A–C: Constic, coarse diamond (R) at time 0 (20 s) / 1 min / 5 min; D–F: Constic, fine diamond (F) at 20 s / 1 min / 5 min; G–I: Vertise™ Flow, coarse (R) at 0 min / 1 min / 5 min; J–L: Vertise™ Flow, fine (F) at 0 min / 1 min / 5 min. A continuous dark-pink interfacial band can be seen, especially in the Constic samples. Coarse finishing produces a more undulating interface than fine, with an apparent qualitative worsening in histochemical appearance. In Vertise™ Flow, longer application times, especially the 1 min, may lead to less collagen exposure

whereas all prolonged application times (1 min and 5 min) were not significantly different from the control comparison. Similarly, in the coarse/rough finish, VF_0_G (0.6 ± 0.1 MPa), CT_0_G (2 ± 0.2 MPa), and CT_1_G (2 ± 0.3 MPa) were significantly lower than the control ($p < 0.001$; $p = 0.003$, and $p = 0.006$, respectively), while the remaining 1 min and all the 5 min application groups did not differ significantly from the control. It is important to note that all failures resulting from the SBS test were of the adhesive type (100%).

Masson's trichrome of the resin-dentin interface

Masson's trichrome clearly differentiated mineralized dentin (blue/blue-green), self-adhesive composites (beige to light-pink), and collagen signal (dark-pink) at the resin–dentin interface. Across groups, a narrow dark-pink band was frequently visible along the resin side of the interface, compatible with superficial demineralization and partial exposure of collagen fibrils. Results are shown in Fig. 3.

The thickness and continuity of the pink/dark-pink band varied between specimens and groups, and in these samples, it was more evident in the groups treated with Constic. This was particularly pronounced in the course/rough group. In Vertise™ Flow, the interfacial dark-pink/red staining band was generally more conspicuous at 20 s and appeared less conspicuous at 1 min (and remained thin in several 5 min sections). For Constic, differences among contact times were subtle and variable, and the staining band did not show a clear monotonic reduction with longer contact time. Surface finishing influenced the macroscopic interface contour in all samples, with coarse/rough preparations often presenting a more undulating junction than the finely polished surfaces, with higher staining in some samples.

SEM of the resin-dentin interface

Representative micrographs from all experimental groups in low and high- magnification are shown in Figs. 4, 5 and 6 and the corresponding controls in Fig. 7. SEM of fractured interfaces revealed a time-dependent evolution in interfacial morphology. At immediate curing (0 min), dentin surfaces displayed a dense tubule field in which many tubule entrances appeared partially occluded by plug-like material, consistent with smear plugs and/or very short intratubular resin remnants; however, a continuous interfacial resin front and organized elongated resin tags were not evident. Large composite remnants on the dentin surface were not found (Fig. 4)

After 1 min of pre-cure contact, discontinuous interfacial resin films/islands were more frequently observed at the fracture plane, often with a transition between resin-covered regions and exposed tubule-rich dentin. Compared with 0 min, these resin-rich remnants are consistent with a shift towards a more mixed fracture pattern as interfacial retention improved; nevertheless, resin tag formation remained sparce. At 1 min, the dentin surface more often exhibited features consistent with greater surface conditioning, including more irregular and enlarged tubule orifices and less sharply defined peritubular outlines, compared with the 0 min specimens.

Finally, the prolonged application time of 5 min yielded the most consistent interfacial features for both materials and both finishes, characterized by a more continuous resin presence at the surface and frequent, elongated resin tags within dentinal tubules, indicating deeper infiltration prior to curing. These qualitative improvements paralleled the time-dependent increases in SBS. Regarding surface finish, differences between fine and rough/coarse preparations were mainly expressed as variations in interfacial contour and fracture topography rather than consistent changes in interfacial resin retention or tag formation at matched times.

The control groups showed extensive resin/composite remnants retained on the dentin surface for both finishes. In the coarse/rough control (R), a large resin/composite mass was observed at the fracture plane adjacent to a dense tubule field, with a sharp boundary between composite-covered and tubule-rich dentin regions. In the fine control (F), a broad resin/composite layer remained over dentin, with a continuous interfacial zone separating the composite/resin-rich region from the underlying tubule-rich dentin; tubule orifices were frequently visible beneath this interface and appeared partially covered or locally occluded near the boundary.

Discussion

The literature is consistent in indicating that the bonding effectiveness of SAFRCs is limited by incomplete interfacial interdiffusion and difficulty in hybridizing dentin [8, 29]. Their viscous, highly filled nature and hydrophobic–hydrophilic mismatch to moist dentin can hinder monomer penetration, resulting in poor retention and high failure rates [7, 8, 29]. The present study was therefore

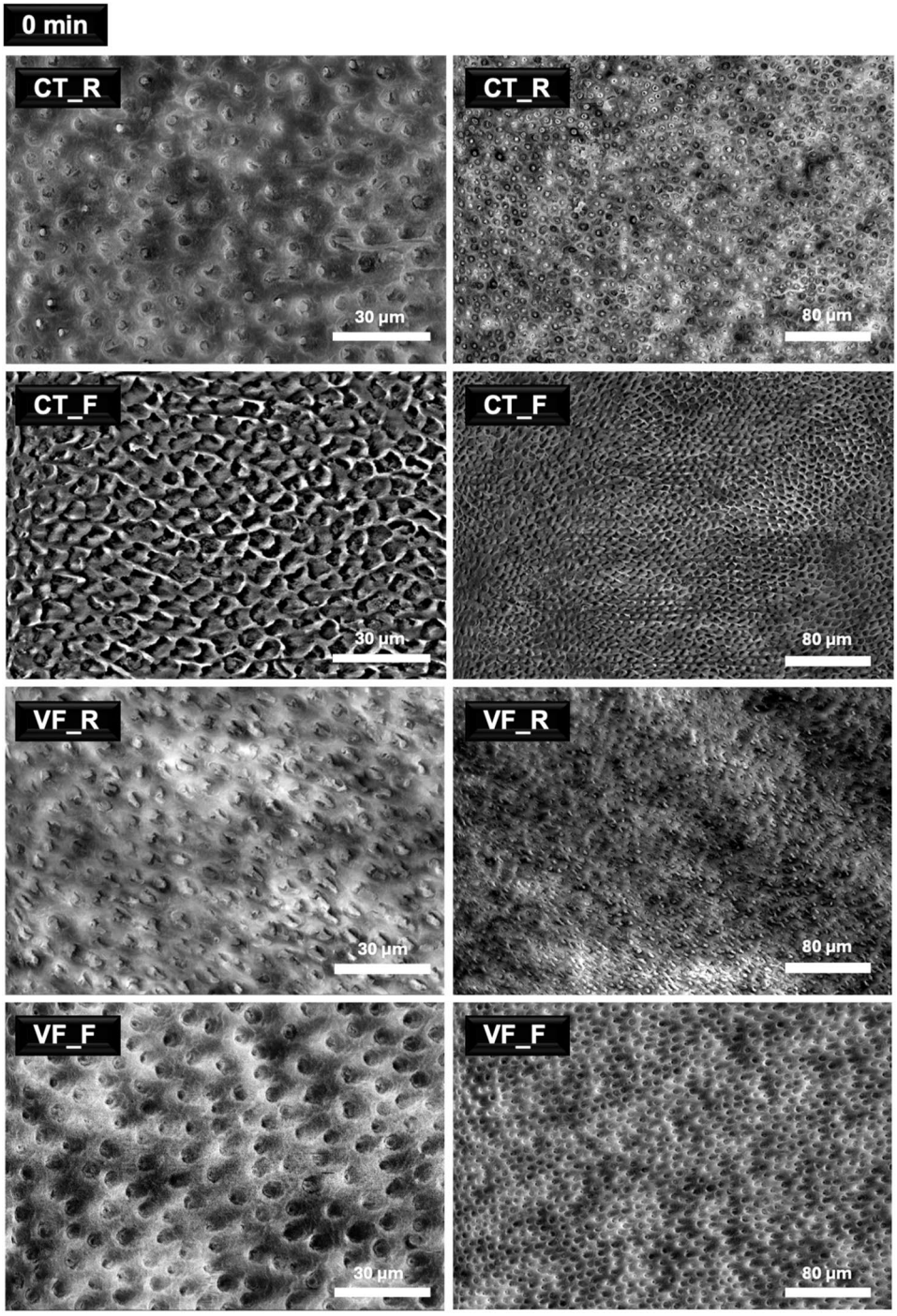

Fig. 4 Scanning electron micrographs of fractured resin–dentin interfaces after immediate light-curing (0 min pre-cure contact) at 1500x and 500x, respectively. Panels show dentin surfaces following microshear testing and interfacial preparation to reveal resin remnants/tags. CT_R: Constic, coarse/rough finish. CT_F: Constic, fine finish. VF_R: Vertise™ Flow, coarse/rough finish. VF_F: Vertise™ Flow, fine finish. Across conditions, tubule orifices are largely patent with minimal interfacial resin remnants and no clear resin tag formation, indicating limited infiltration when polymerization occurs immediately

designed to examine two key factors under the control of the operator: (i) the surface roughness that precedes the restorative protocol and (ii) the pre-cure application time, hypothesizing that these may significantly influence resin–dentin bonding with SAFRCs.

Preparing dentin surfaces with different grit diamond burs yields smear layers of different thickness; coarse-grit diamonds produce a thicker (≈2–3 µm) and more compacted smear layer than fine-grit diamonds (≈1 µm) [30]. These debris-rich layers, packed with fragmented

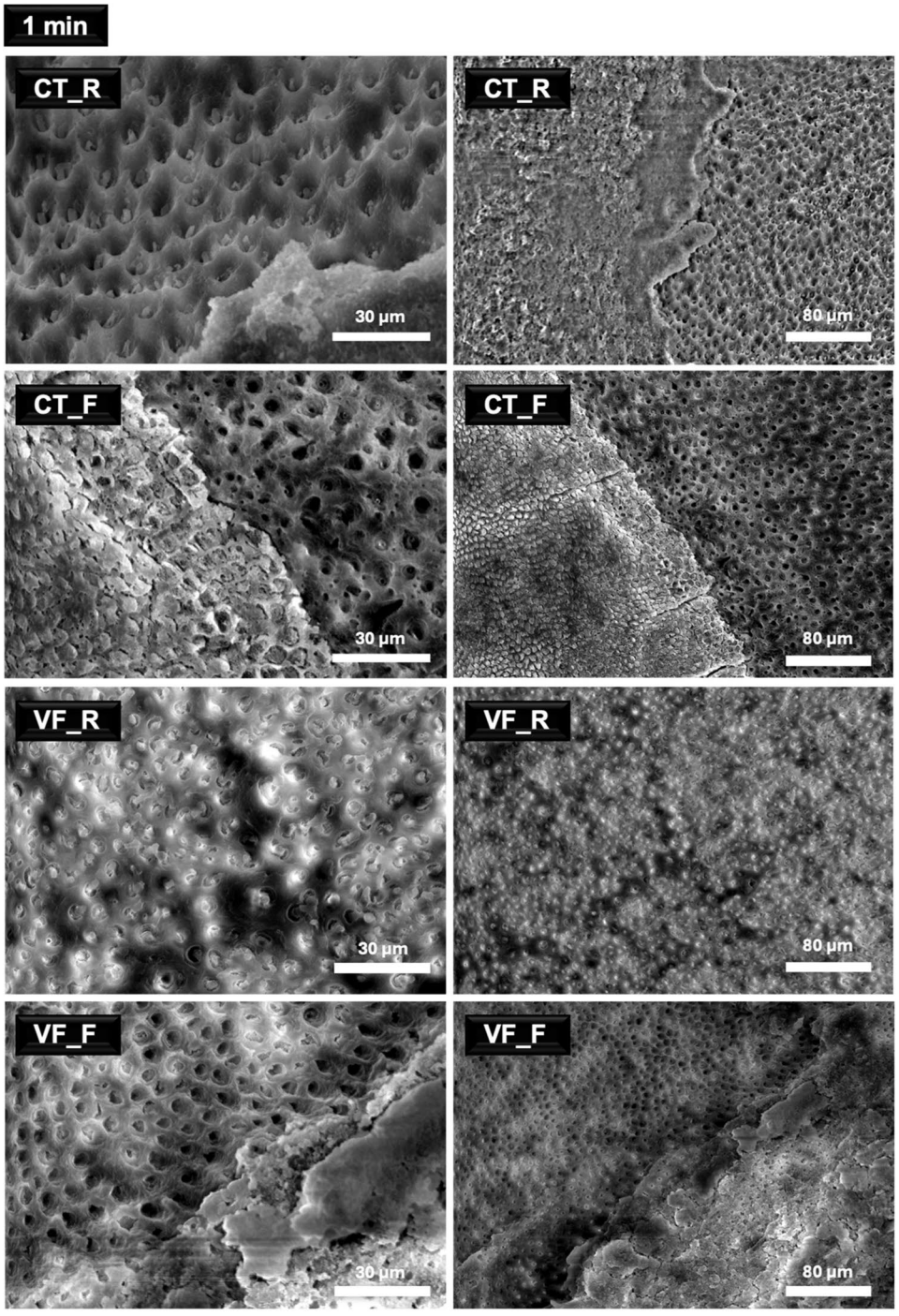

Fig. 5 Scanning electron micrographs of fractured resin–dentin interfaces after 1 min pre-cure contact. CT_R: Constic, coarse/rough finish. CT_F: Constic, fine finish. VF_R: Vertise™ Flow, coarse/rough finish. VF_F: Vertise™ Flow, fine finish. Compared with immediate curing, discontinuous resin remnants/films are more frequently observed at the fracture plane, with localized interfacial coverage but limited resin tag formation

hydroxyapatite and denatured collagen, impede the access of acidic functional monomers. To guarantee effective interaction, such monomers must first partially etch/dissolve the smear and the superficial dentin and then diffuse into the exposed collagen network [31, 32]. Yet, the manufacturer-recommended application time for SAFRCs is typically 20 s of active rubbing/brushing [33, 34], a contact time likely then insufficient for smear layer modification, sufficient substrate demineralization and resin infiltration. Therefore, evaluating whether prolonged pre-cure contact and clinically relevant surface finishes can overcome these barriers is warranted.

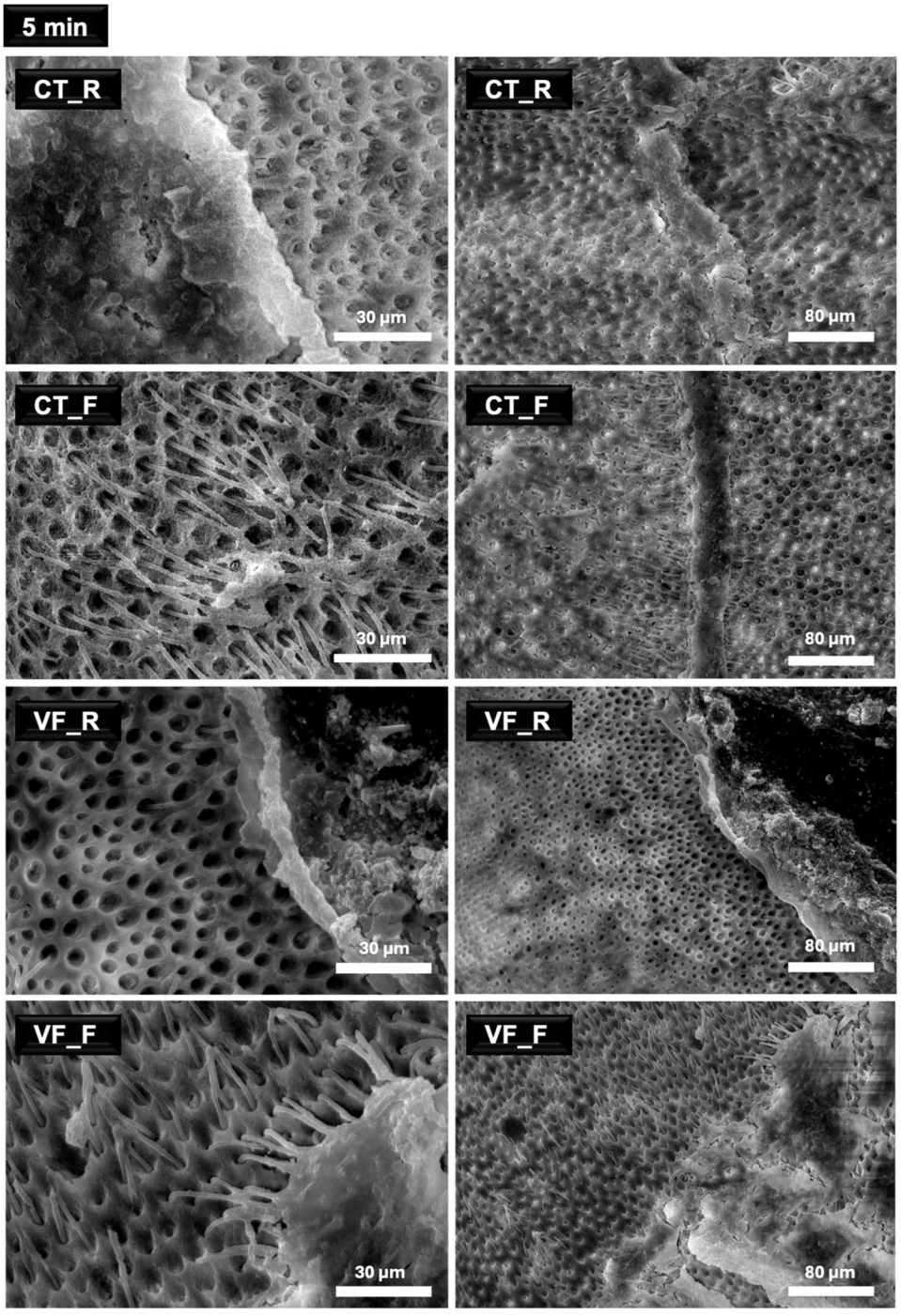

Fig. 6 Scanning electron micrographs of fractured resin–dentin interfaces after 5 min pre-cure contact. **A**–**B**: Constic, coarse/rough finish (low and high magnification). **C**–**D**: Constic, fine finish (low and high magnification). **E**–**F**: Vertise™ Flow, coarse/rough finish (low and high magnification). **G**–**H**: Vertise™ Flow, fine finish (low and high magnification). Prolonged contact yields a more continuous interfacial resin presence and frequent intratubular protrusion-like resin remnants were observed in several specimens, most clearly appreciated on fine-finished surfaces; finish-related differences are mainly topographic

Indeed, to our knowledge, this has not been systematically tested in these materials.

The first null hypothesis, that varying the pre-cure application time would not influence bonding, was rejected. Prolonging the SAFRC application time prior to light-curing significantly increased SBS. Notably, the 20 s application (the default protocol) yielded the lowest bond strengths, whereas 1 min and especially 5 min application times produced higher SBS values. This trend was consistent for both Vertise™ Flow and Constic. The

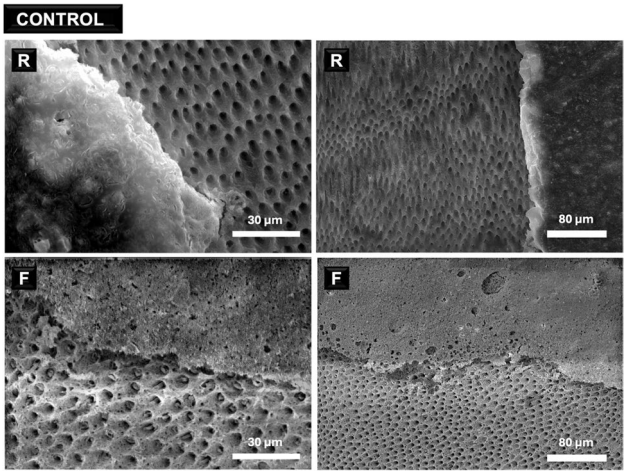

Fig. 7 SEM micrographs of the control groups (universal adhesive + conventional composite) on coarse/rough (R) and fine (F) dentin finishes. In both controls, broad composite/resin-rich remnants remain adherent to dentin, and the fracture plane shows mixed regions comprising resin/composite-covered areas adjacent to tubule-rich dentin

improvement with extended application can be attributed to enhanced monomer diffusion and prolonged acid–base interaction at the interface. Mechanistically, phosphate-ester monomers in these one-step materials (e.g., GPDM, 10-MDP) are ultra-mild self-etchers acting through the adhesion–decalcification balance: they must (i) partially dissolve the smear layer/superficial dentin and (ii) diffuse into the demineralized collagen while (iii) establishing ionic bonding to residual apatite (e.g., formation of MDP–Ca salts and interfacial nano-layering) [1, 35]. All three processes are time-dependent and initially buffer-limited by HAp in the smear and dentin [17, 35]. Longer dwell sustains proton availability and chelation, allowing further smear dissolution, more micro-porosity for uptake, and more extensive monomer adsorption/MDP–Ca salt formation before light-curing induces network formation and crosslinking. This is in line with prior observations that more aggressive or longer application of self-etch adhesives leads to better substrate conditioning and higher bond strengths [36, 37].

In the present study, the fractured-surface SEM analysis also revealed that the 20 s groups had a relatively continuous smear layer covering dentin and only shallow penetration of resin. The default application time of 20 s underperforming is expected. SAFRCs are highly filled, high-viscosity pastes without a volatile solvent, so monomer mobility and water displacement are poor; with a thick/compact smear, the mild acidity is rapidly neutralized, yielding shallow demineralization and incomplete infiltration. Longer contact times (1 and 5 min) were associated with greater amounts of composite remnants remaining on dentin and more frequent intratubular protrusion-like features after interfacial preparation. This was especially true for the longest application time. Masson's trichrome staining technique partially confirmed these findings. With Vertise Flow, in the longer application times, the interface stained predominantly green with little to no red, denoting that collagen was largely encapsulated by resin. The concurrence of these mechanical and microscopic outcomes underscores that extended contact time may in fact allow the SAFRCs to achieve a more effective hybridization. A key limitation is that SEM was performed on fractured specimens obtained after SBS testing. Accordingly, these images primarily reflect fracture-plane morphology and retained resin/composite remnants, which may be influenced by failure mode. They do not provide direct longitudinal visualization of the intact bonded interface.

Clinically, a 5-min dwell is unrealistic; and yet the literature shows that even modest extensions of active application (≈30–60 s of scrubbing) improve conditioning and infiltration for self-etch/universal systems, whereas shortening the recommended time may degrade performance. Meta-analytic and experimental data consistently report higher dentin bond strength with active/prolonged application and lower values when application is abbreviated in adhesives [37]. A consistent pattern observed across the experimental groups was the correspondence between mechanical strength and interfacial morphology. When higher bond strengths were recorded, the qualitative analyses showed more favorable morphology. This is suggestive of a true material effect rather than random scatter, while remaining consistent with broader evidence on the imperfect, but still meaningful, link between interfacial quality and laboratory bond tests [38].

With respect to substrate preparation, the null hypothesis for surface roughness (fine vs. coarse diamond) could not be rejected; the three-way ANOVA showed no main effect of finish on SBS, and SEM did not reveal consistent qualitative differences at matched times. This finding aligns with literature showing that both smear-layer characteristics *and* surface roughness/topography can influence self-etch bonding, but that their impact is adhesive-dependent and often secondary to application/activation time and adhesive aggressiveness [39]. Also, when the smear differences are within a narrower, clinically common range, effects may not attain differences.

The smear layer, rich in calcium salts, can quickly neutralize acid groups (an acid–base interaction), limiting deeper demineralization. Mild self-etch/SAFRC chemistries rely on the adhesion–decalcification balance: limited demineralization and chemical interaction with residual hydroxyapatite (e.g., MDP–Ca salt formation) and concurrent resin diffusion into a shallow matrix. But a smear layer rich in apatite buffers/neutralizes acidic groups and restricts depth, while the high filler content

and viscosity of SAFRCs (no volatile solvent) constrain wetting and transport, yielding the well-documented shallow infiltration and weak interfaces on smear-covered dentin [40, 41]. Therefore, extending pre-cure contact acts like a slow-etch/slow-infiltration step. This gives more time for proton-driven dissolution of smear debris and for functional monomers to percolate sub-micron pathways before cure, all effects that can outweigh modest differences in finish within normal smear conditions. This interpretation is consistent with classic smear-layer studies (that evaluated thickness/compactness vs. self-etch acidity [14, 15]) and with interfacial microscopic evidence for SAFRCs [6–8, 42].

Two considerations temper over-interpretation. First, while bur grit was standardized (and different), the actual functional difference in smear compactness/permeability achieved under our controlled preparation may have been insufficient to create divergent diffusion kinetics for these ultramild systems at a given time point. Second, the study was powered for main effects in SBS, not to resolve small finish×time interactions; nonetheless, the finish main effect remained negligible even as time exerted a large, consistent influence across materials. In summary, these data support that, within the present smear-layer window, extending SAFRC pre-cure contact time is the critical procedural lever, whereas switching between the two tested diamond finishes had minimal additional impact on the immediate dentin bonding setup that was tested.

Finally, Vertise™ Flow outperformed Constic in μSBS and interface morphological features. This is likely due to a few reasons. Firstly, it is possible that any intrinsic chemical advantage of Constic having 10-MDP, the flagship acidic monomer [43], was offset by its lack of HEMA and higher viscosity; Vertise™ contains HEMA as a co-solvent monomer which is known to improve initial wetting and penetration in dentin, particularly useful in the case of viscous, self-adhesive composites [8, 44]. HEMA, a low–molecular-weight hydrophilic monomer that lowers local viscosity, improves wetting, and promotes initial diffusion into moist dentin; even small HEMA fractions are known to enhance immediate infiltration in adhesive systems [45, 46]. Nevertheless, both materials in this study achieved only moderate bond strengths relative to what is seen with conventional adhesives, and both largely failed in adhesive mode at the smear-modified layer. Under such conditions, the limitations imposed by their high filler content and viscosity likely dominate the outcome [47].

It is important to mention that only 24 h bond strength data was tested. This was chosen since a rapid screening of the variables under study was procured. Future studies should assess long-term durability after aging to confirm if the improved initial bonding translates to clinical longevity. The range of smear layer conditions that were tested (fine vs. coarse diamond) represents two extremes of clinical technique. In practice, clinicians may use a medium-grit bur or a combination of instruments, and they might also use cavity cleaners, which could alter the smear, before applying SAFRCs. Thus, the numeric differences observed here might not directly translate to every clinical situation. Similarly, the contact times of 1 min and 5 min were chosen to exaggerate the effect and understand the trend. It is acknowledged that 5 min of wait time is impractical in routine dentistry. In parallel with the SBS findings showing higher immediate bond strength at longer contact times and the SEM observations of more frequent resin-phase continuity at 5 min, the trichrome images mainly indicated that a collagen-associated interfacial signal could still be detected under almost all conditions, with variability that limited a clear visual separation among application times and between materials at matched finishes. Although improvements can be seen, it is likely that the best application strategy is yet to be determined. The ideal future material would achieve deep dentin hybridization and chemical coupling without additional steps or excessive waiting.

Conclusion

Extending the pre-cure contact of SAFRCs from the manufacturer default to 1–5 min increased immediate shear bond strength for both materials and was accompanied by micromorphological evidence of more continuous interfacial resin features with more frequent tubule tag formation. Masson's trichrome consistently showed a thin dark-pink/red collagen-associated band at the resin–dentin boundary; this band was often less prominent at longer dwell times in Vertise™ Flow (most evident at 1 min) and showed a less consistent trend for Constic. Dentin surface finish produced different interfacial contours yet no main effect on SBS within the smear range tested. Vertise™ Flow outperformed Constic, although both achieved moderate strengths and predominantly adhesive failures, reflecting limitations of these highly filled, solvent-free, formulations. These data identify pre-cure dwell as a consequential operator variable for immediate bonding, while suggesting a ceiling to interfacial quality with current chemistries.

Abbreviations

10-MDP	10–methacryloyloxydecyl dihydrogen phosphate
Au/Pd	Gold/palladium (sputter–coating alloy)
Bis-GMA	Bisphenol A glycidyl methacrylate
CAD/CAM	Computer–aided design / computer–aided manufacturing
CiiEM	Centro de Investigação Interdisciplinar Egas Moniz
CT	Constic (self–adhesive flowable resin composite)
FE	SEM–Field–emission scanning electron microscopy
GPDM	Glycerol phosphate dimethacrylate
HAp	Hydroxyapatite
HCl	Hydrochloric acid

HEMA	2–hydroxyethyl methacrylate
MDP	Methacryloyloxydecyl dihydrogen phosphate
MDP	Ca–Calcium salt of 10–MDP
MPa	Megapascal
NaOCl	Sodium hypochlorite
SAFRC / SAFRCs	Self–adhesive flowable resin composite(s)
SBS	Shear bond strength
SE	Standard error (of the mean)
SEM	Scanning electron microscopy
SPSS	Statistical Package for the Social Sciences (statistical software)
TEGDMA	Triethylene glycol dimethacrylate
TEM	Transmission electron microscopy
UDMA	Urethane dimethacrylate
VF	Vertise™ Flow (self–adhesive flowable resin composite)
μSBS	Micro–shear bond strength
μTBS	Microtensile bond strength

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Authors' contributions
I.M. and T.A.S. performed the investigation, data analysis and drafted the original manuscript. P.P. and A.H.S.D. were involved in the study conception, design, supervision, data curation and manuscript revision. All authors reviewed the manuscript.

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Data availability
full data can be made available upon request to the corresponding author.

Declarations

Ethics approval and consent to participate declaration
This study involved the ex vivo use of extracted human teeth and did not include any direct interaction with human participants. The study protocol was reviewed and approved by the Comissão de Ética Egas Moniz (approval no. 1342). All procedures were conducted in accordance with the Declaration of Helsinki. Extracted teeth that were scheduled for removal as part of routine dental care were obtained from patients attending Clínica Dentária Universitária Egas Moniz. Prior to tooth extraction, all patients received information about the potential research use of their extracted teeth and provided written informed consent for this use

Consent for publication
not applicable

Competing interests
The authors declare no competing interests.

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