**Abstract**

**Purpose:** This study investigated the flexural properties, shear bond strength (SBS) and interface to dentin of three recently developed self-adhesive bulk-fill materials.

**Methods:** Bars of Surefil One (SO), Cention N (CN), Activa BioActive Restorative (AB) and EQUIA Forte HT Fil (EQUIA) were tested for flexural strength and flexural modulus in self-curing and light-curing modes. In addition, SBS to dentin was tested in specimens without pretreatment and after application of universal adhesive (Scotchbond Universal). EQUIA was used as the control material.

**Results:** The flexural properties were significantly better in light-curing mode for all materials except CN. CN had the highest SBS values after universal adhesive application (33.8 MPa), and SO had the highest SBS without pretreatment (20.9 MPa).

**Conclusion:** The mechanical and adhesive properties of these new materials varied widely.

Keywords: flexural strength, scanning electron microscopy, self-adhesive bulk-fill materials, shear bond strength

**Introduction**

Self-adhesive materials are a major development for direct restorative materials because the absence of a specific adhesive protocol makes them easier to use [1]. Bulk-fill techniques simplify procedures by limiting the number of increments needed to fill an entire cavity [2].

The glass ionomer cement (GIC) family is the most widely used self-adhesive bulk-fill material in direct restorations [3]. GICs have other interesting properties that are directly linked to their chemistry, such as moisture tolerance [4] and rechargeable fluoride release from fluorooalumino-silicate (FAS) fillers, which leads to potential dental hard tissue remineralization [5] and cariostatic effects [6]. These materials can be resin-free, such as the GICs and their improved formulation—called high-viscosity glass ionomer cements (HV-GICs)—or may contain additional resinous content (mainly 2-hydroxyethylmethacrylate), such as resin-modified glass ionomer cement (RM-GIC) [3].

Conventional GICs are no longer used for definitive restorations because of their strong tendency to abrasion, fracture and debonding [7]. RM-GICs possess improved adhesion [8] and flexural characteristics but still have low abrasion resistance and must be laminated in accordance with the manufacturer’s instructions [9]. HV-GICs can be successfully used as definitive restorations of occlusal and limited proximal cavities; however, their enamel and dentinal bonding strength values are inferior to those of resin composites, [10,11] and bulk fracture may occur because of low flexural strength [12]. Recently, some new self-adhesive resinous materials with claimed fluoride-releasing and “bulk-fill” properties were introduced as “bioactive materials” or “smart materials”. These materials differ in chemical composition from the GIC family [13] and could be an alternative that exceeds their performance [9].

Many parameters must be considered when restoring a large cavity, including the mechanical properties of the restorative material and dentin bond strength [14]. Few studies have evaluated the mechanical properties and bonding ability of Activa BioActive Restorative (Pulpdent Corp., Watertown, MA, USA, launched in 2013), Cention N (Ivoclar-Vivadent AG, Schaan, Liechtenstein, launched in 2016) and Surefil One (Dentsply-Sirona, Konstanz, Germany, launched in 2019), which are putative competitors to HV-GICs and have extended indications. In contrast to HV-GICs, these materials are described as being able to restore all types of cavities when the tooth does not require cuspid coverage.

The aim of this study was, first, to study the flexural properties of these materials, with different curing protocols, and, second, to evaluate their dentin bond strength and the bonded interface. The null hypotheses tested were (i) that flexural properties would not differ in relation to material or curing mode, (ii) that there would be no differences in dentin bond strength between materials, with or without the use of an adhesive system and (iii) that there would be no differences in the interface pattern between tested groups.

**Materials and Methods**

**Materials used and experimental procedures**

Surefil One, Cention N and Activa BioActive Restorative were compared to HV-GICs. The materials, manufacturers, batch numbers, composition [9,13] and characteristics [15,16] are presented in Table 1. These materials were tested for flexural strength (FS), flexural modulus (E) and dentin bond strength and were compared to a recent HV-GIC (EQUIA Forte HT, GC Corp., Tokyo, Japan), used as a control group.

**Flexural strength and flexural modulus testing**

One hundred forty bars were made by using a 2 × 2 × 25 mm silicon mold (EXA’lence, GC Corp.) according to ISO 4049 [17]. Seven groups (n = 20) were evaluated: Surefil One in self-cure mode (SO-SC), Surefil One in light-curing mode (SO-LC), Cention N in self-cure mode (CN-SC), Cention N in light-curing mode (CN-LC), Activa BioActive Restorative in self-cure mode (AB-SC), Activa BioActive Restorative in light-curing mode (AB-LC) and EQUIA Forte HT Fil in self-cure mode followed by an application of coating material (EF-SC). Photopolymerization was performed with a polymethyl curing light at a minimum output of 950 mW/cm² (Valo Grand Cordless, Ultradent Products, South Jordan, UT, USA).

The abbreviations, groups and detailed light-curing and self-curing protocols are presented in Table 2. Material bars were then stored in water for 2 weeks at 37°C before performing the mechanical tests, to ensure that the maturation process, which requires several hours for the GIC family, was mostly completed. For each group, the flexural strength and flexural modulus of samples were tested by a three-point bending test on a universal testing machine (Shimadzu AGS-X, Shimadzu Corp., Kyoto, Japan). Each specimen was placed at the center of a universal tester between two
crossheads with a width of 20 mm, and the maximum load was measured by applying a vertical load to the center of the specimen at a crosshead speed of 0.5 mm/min until fracture.

Maximum load (fracture load) was recorded in newtons, and flexural strength (FS) was calculated in megapascals, as follows: $FS = (3Pl)/\left(4wb^3d\right)$, where $P$ is the applied load (in N), $l$ is the test interval (in mm), $w$ is the width of the specimen (in mm) and $b$ is the thickness of the specimen (in mm).

The flexural modulus ($E$) was calculated from the three-point bending test and expressed in gigapascals, as follows: $E = (Pl^3)/(4wb^3d)$, where $d$ is the depth of elastic deformation (in mm).

### Table 1: Abbreviations, manufacturers, batch numbers and composition of the materials used

| Materials | Abbreviation | Shade | Manufacturer | Batch number | Composition / characteristics | Properties and use |
|-----------|--------------|-------|--------------|--------------|-------------------------------|--------------------|
| New resins fluoride-releasing materials | Surefil One | SO | Dentply-Sirona, Konstanz, Germany | 1907000895 | Powder: silanated aluminum-phosphate-strontium-sodium-fluoro-silicate glass, dispersed silicon dioxide, ytterbium fluoride, pigments | Self-adhesive bulk-fill resinous restorative material with ionic release after polymerization. Does not require a bonding agent, regardless of the cavity. |
| | CN | U | Ivoclar-Vivadent, Schaan, Liechtenstein | X54163 | Powder: barium aluminum silicate glass, ytterbium trifluoro, isofiller, calcium barium aluminum fluorosilicate glass, calcium fluoro silicate glass | Non-adhesive bulk-fill resinous restorative material with ionic release after polymerization under acidic challenge. Bonding agent not required for retentive cavities but required for a non-retainive cavity. |
| | Activa Bioactive Restorative | AB | Puldent, Watertown, MA, USA | 190307 | Powder: silanated bioactive glass and calcium, silanated silica, sodium fluoride | Self-adhesive bulk-fill resinous restorative material with ionic release. No bonding agent required, regardless of cavity. |
| Former non-resinous fluoride-releasing material (HV-GIC) | EQUIA Forte HT Fil, with self-curing material | EF | GC Corp., Tokyo, Japan | 192051 | Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide | Self-adhesive bulk-fill resinous restorative material with ionic release. |
| Dental adhesive System | Scotchbond Universal | SBU | 3M ESPE, St Paul, MN, USA | 5271401 | HEMA, Bis-GMA, decamethylenedimethacrylate, ethane, silane treated silica, water, MDP, copolymer of acrylic and itaconic acid, dimethylaminoethyl methacrylate, camphorquinone, dimethanolmethacrylate, 2,4-di-t-butyl-p-cresol, photoinitiators, iron oxide. | - |
| Etching product | Scotchbond Universal | SE | 3M ESPE | 465332 | Orthophosphoric acid (37%), water, synthetic amorphous silica, polyethylene glycol, aluminum oxide | - |
| Cavity cleaning product | Dentin Conditioner | DC | GC Corp. | 1805111 | Distilled water, polyacrylic acid | - |

HEMA, 2-hydroxethyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate

### Table 2: Abbreviations, sample preparation and curing protocol for testing of flexural strength and flexural modulus

| Material and curing protocol used | Bonding protocol |
|----------------------------------|-----------------|
| Surefil One, self-curing protocol (SO-SC) | For each sample, a SO capsule was mechanically mixed (Silver Mix 90 Mixer, GC Corp.) for 10 s according to the manufacturer’s instructions, injected in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. After 10 min at room temperature, the sample bar was unmolded and stored in 37°C water for 2 weeks. |
| Surefil One, light-curing protocol (SO-LC) | For each sample, a SO capsule was mechanically mixed (Silver Mix 90 Mixer, GC Corp.) for 10 s according to the manufacturer’s instructions, injected in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. The upper surface of the sample bar was light-cured for 60 s at three points and then unmolded and stored in 37°C water for 2 weeks. |
| Cention N, self-curing protocol (CN-SC) | For each sample, CN was hand-mixed according to the manufacturer’s instructions, inserted in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. After 10 min at room temperature, the sample bar was unmolded and stored in 37°C water for 2 weeks. |
| Cention N, light-curing protocol (CN-LC) | For each sample, CN was hand-mixed according to the manufacturer’s instructions, inserted in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. The upper surface of the sample bar was light-cured for 60 s at three points and then unmolded and stored in 37°C water for 2 weeks. |
| Activa Bioactive Restorative, self-curing protocol (AB-SC) | For each sample, AB was injected in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. After 24 h at room temperature (material was unset if removed earlier), the sample bar was unmolded and stored in 37°C water for 2 weeks. |
| Activa Bioactive Restorative, light-curing protocol (AB-LC) | For each sample, AB was injected in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. The upper surface of the sample bar was light-cured for 60 s at three points and then unmolded and stored in 37°C water for 2 weeks. |
| EQUIA Forte HT Fil, with self-curing protocol followed by application of coating material (EF-SC) | For each sample, an EF capsule was mechanically mixed (Silver Mix 90 Mixer, GC Corp.) for 10 s according to the manufacturer’s instructions, injected in excess inside a silicon mold and covered on the top surface by a Mylar band and a glass slab under finger pressure. After 10 min at room temperature, the coating material (EQUIA Forte Coat, GC Corp.) was applied on the top surface of the sample bar with a microbrush, rubbed for 20 s (not air-dried) and light-cured for 60 s at three points. Then, the bar was unmolded and stored in 37°C water for 2 weeks. |
Dentin shear bond strength tests, failure mode determination and scanning electron microscopy examination

One hundred and fifty-four freshly extracted human permanent molars were collected from adults after extraction, cleaned of soft tissues, stored at 4°C in a solution of 1% chlorine and used within 3 months. Teeth were obtained from the dental departments of AP-HP, France. All experiments were conducted in accordance with the principles articulated in the Declaration of Helsinki. All teeth were collected with the informed oral consent of all patients, in accordance with the ethical guidelines set by French law and with specific authorization by Paris university dental school (n°DC-2009-927, Cellule Bioéthique DGRI/A5, Paris, France).

The criterion for tooth selection was absence of cracks and decay. The greater portion of the roots was removed with sandpaper (80 grit) by using a polishing machine (Planopoli 3, Struers, København, Denmark). The occlusal surface of the crowns was then abraded with water-cooled sandpaper (800 grit) to expose a flat dentin surface (>7 mm² with a roughness corresponding to the finishing obtained with a red diamond bur. The residual crowns were embedded in self-curing acrylic resin (Plexil-ESCL, Chassieu, France) in a plastic cylinder (diameter: 25 mm, depth: 15 mm), with the flat dentin surface exposed. The surfaces were inspected under ×40 magnification to ensure that the enamel had been completely removed and that the dentin was cleared of debris.

These teeth were randomly assigned to seven groups (n = 22): Surefil One (SO-Only); Surefil One after SBU application with a self-etch protocol (SO-SBU); Cention N (CN-Only); Cention N after SBU application with a self-etch protocol (CN-SBU); Activa Bisactive Restorative (AB-Only); Activa Bisactive Restorative after SBU application with a self-etch protocol (AB-SBU); EQUIA Forte HT Fil after Dentin Conditioner application (EF-DC).

For each group, 20 teeth were used for shear bond strength (SBS) testing and two were used for scanning electron microscopy (SEM) interfacial observation. SBS was determined in a universal testing machine (LRX, Lloyd Instruments, Fareham, UK). The shear force was applied at the material cylinder/dentin interface, with a chisel-shaped blade parallel to the dentin surface. A crosshead speed of 0.5 mm/min was used.

The debonded specimens were observed under a binocular microscope (BZH10, Olympus, Hamburg, Germany) at ×30 magnification, and failure mode was classified as follows:

- Type CF-D: cohesive failure within the dentin
- Type AF: adhesive failure at the interface between the material and dentin
- Type MF: mixed failure (adhesive and cohesive failure within the material)

For SEM examination, after the additional inclusion in self-cure acrylic resin of the material cylinder, samples were sectioned perpendicularly to the bonded interface with a low-speed diamond saw (Isomet, Buehler, Coventry, UK) and water cooling, as near as possible to the center of the cylinder. The sections obtained were polished with abrasive disks of decreasing grit size (400, 800, 1,200, 2,400, and 4,000 SiC) and then with diamond particles of 3 μm and 1 μm. Finally, the sections were etched with orthophosphoric acid for 10 s (Scotchbond Universal Etchant, 3M ESPE, St. Paul, MN, USA). The samples were cleaned by ultrasonication after each step and then dehydrated in ethanol and metallized with gold (Sputter Coater, Bio-rad, Marnes-la-Coquette, France) for microscopy examination (JSM-6400, JEOL LTD, Tokyo, Japan).

Statistical analysis

Normal distribution was confirmed by the Shapiro-Wilk test, and equality of variance was assessed using Levene’s test before the tests were performed. SBS data were expressed as means and standard deviations. One-way ANOVA followed by Tukey’s post-hoc test was used to investigate differences in flexural strength, flexural modulus and SBS between groups. Failure mode was analyzed by Fisher’s exact test for single comparisons between groups and in pairwise analysis. In all tests, the chosen significance level was α = 0.05. R software (version 3.6.1; R Foundation for Statistical Computing, Vienna, Austria) was used for all statistical calculations.

Results

Flexural properties

The flexural properties for all experimental groups are summarized in Table 4. The Shapiro-Wilk test confirmed a normal distribution of flexural properties values among all groups (P > 0.05), and Levene’s test showed...
equality of variance ($P > 0.05$). One-way ANOVA followed by Tukey’s post-hoc test revealed significant differences ($P < 0.05$).

Flexural strength significantly differed in relation to material and curing protocol. CN-SC and CN-LC had the highest flexural strength ($86.3 \pm 7.8$ MPa and $83.3 \pm 15.0$ MPa, respectively). Flexural strength was higher with a light-curing protocol for SO-LC and AB-LC ($45.3 \pm 11.7$ MPa and $67.7 \pm 11.8$ MPa, respectively) than with a self-curing protocol ($24.5 \pm 7.4$ MPa). Flexural strength was significantly lower for EF-SC ($22.7 \pm 6.9$ MPa) than for the other groups.

Flexural modulus significantly differed in relation to material. EF-SC had the highest flexural modulus ($12.0 \pm 2.1$ GPa), whereas flexural modulus values were lowest for AB-LC and SC-LC, in light-curing ($2.3 \pm 0.3$ GPa) and self-curing ($1.4 \pm 0.4$ GPa) modes, respectively.

### SBS and failure analysis

SBS values for all experimental groups are summarized in Table 5. The Shapiro-Wilk test confirmed a normal distribution of SBS values in all groups ($P > 0.05$), and Levene’s test showed equality of variance ($P > 0.05$). One-way ANOVA followed by Tukey’s post-hoc test revealed significant differences in SBS values ($P < 0.05$).

SBS significantly different in relation to material and dentin treatment. All the materials tested with an adhesive had higher SBS values. SBS was significantly higher for CN-SBU ($33.8 \pm 5.7$ MPa) and AB-SBU ($28.9 \pm 5.2$ MPa) than for SO-SBU ($20.9 \pm 4.1$ MPa). However, in the absence of adhesive, SBS was significantly higher for SO-Only ($14.0 \pm 3.4$ MPa) than for EF-DC ($8.0 \pm 1.8$ MPa), but these values were significantly higher than those for AB-Only ($4.4 \pm 2.5$ MPa) and CN-Only ($3.0 \pm 1.0$ MPa).

Failure modes are listed in Table 6. Fisher’s exact test indicated significant differences between the groups. The most frequent failure for AB-SBU and CN-SBU was cohesive failure in dentin, and the frequency of such failures in these groups significantly differed from those in the other groups.

### SEM examination

Figure 1 shows SEM images of Surefil One, Cention N and Activa BioActive Restorative, with or without application of a universal adhesive, and Fig. 2 shows an SEM image of the control group, EQUIA Forte HT Fil, after application of Dentin Conditioner at ×1,000 magnification.

In the EF-DC group, HV-GIC/dentin appeared to be continuous. No material deposits are present inside dentin tubules. Many gaps are visible between FAS fillers.

In the CN-Only and AB-Only groups, the material/dentin interface appears discontinuous. No material is deposited inside dentin tubules. Gaps are visible between some fillers (especially for CN-Only) and the resin matrix. In the SO-Only, SO-SBU, CN-SBU and AB-SBU groups, the material/dentin interface appears continuous, and tags can be seen inside dentin tubules. Gaps are visible between some fillers (especially for CN-SBU) and the resin matrix.

### Discussion

An ideal restorative material should be biocompatible, mechanically resistant and aesthetically pleasing. It should also yield spontaneous, durable adhesion to dental tissues and have bulk-fill properties and bioactivity.

HV-GICs, the present control material, are the most used self-adhesive bulk-fill material family for definitive restoration [3,12]. However, because of their tendency to debonding and bulk fracture, they have limited indications as definitive materials [12] and lower dentin bond strength than resin composites [18], a limitation that cannot be improved by use of an adhesive system. Moreover, their aesthetic outcomes are worse than those of resin composites, and microleakage at restorative margins has been reported [19].

Resinous self-adhesive, bulk-fill, “hybrid materials” with putative bioactivity and enhanced indications for direct restorations were recently introduced. However, few studies have evaluated their chemical characteristics, clinical behavior and flexural and bonding properties. In this study, all materials underwent three-point bending testing with a self-curing and...
light-curing protocol, to determine how the polymerization reaction affects mechanical performance and the setting reaction of materials. Significant differences were observed in relation to material and curing mode; thus, the first null hypothesis was rejected.

When a definitive restorative material must be placed in a stress-bearing area, such as an occlusal posterior area, ISO 4049 standards recommend a flexural strength of at least 80 MPa [17]. However, many studies have reported that materials that satisfy this standard have increased fracture susceptibility, especially in proximal areas [20]. Flexural strength is therefore an important predictor of the clinical success of a material.

Cention N satisfies the minimum ISO 4049 value. It exhibited the highest flexural strength in self-cured and light-cured reactions, and no significant difference between these modes, which suggests promising mechanical results in stress-bearing areas. Surefil One and Activa BioActive Restorative had values below the ISO 4049 standard. Both had higher flexural strength in the light-curing mode, despite the absence of significant differences in surface microhardness and flexural modulus. These materials, in contrast to Cention N, are based on a double-setting reaction (an acid-base reaction and resinous polymerization) [21]. Competition between these reactions, as previously seen in RM-GICs [22], could explain the mechanical differences between curing modes. A decreased conversion rate in self-cure mode was also suspected in another study of Surefil One [9]. This hypothesis requires confirmation in more-comprehensive studies of polymerization.

As compared with HV-GIC, all the new hybrid materials had better flexural strength values in light-curing mode and some had better values in self-curing mode (Cention N and Activa BioActive Restorative). Thus, they may be stronger alternatives for posterior restorations with simplified protocols, even if the flexural strength needs to be improved to satisfy ISO standards [17]. The low flexural strength values observed for EF-SC could explain why bulk fracture was reported as the main cause of failure for large HV-GIC restorations [12,23].

Analysis of the flexural modulus showed important differences between groups. Many previous studies showed that flexural modulus was strongly associated with material composition [23,24]. The findings of this study suggest that the chemical characteristics of the three recently developed self-adhesive hybrid materials greatly differ from those of resin-free HV-GICs. Further studies of the chemical profiles of these new formulations are thus warranted. SBS testing is preferred to other bonding tests, such as microtensile bond strength testing. In fact, because some materials have lower bond strengths than those of resin composites, other bonding tests would be difficult to perform. SBS is effective in overcoming this limitation [25].

Bonding values and failure patterns significantly differed in relation to material and adhesive procedure; thus, the second null hypothesis was rejected. All the materials tested with an adhesive had higher SBS values. Similarly, dentin bond strength of RM-GICs was reported to be higher when an adhesive was used [26].

SBS values were highest for the CN-SBU and AB-SBU groups. Most failures in these groups were cohesive within dentin, but there was no significant difference in failure pattern. Thus, although SBS was not an exact measure of the interfacial bond, it represented the cohesive strength of dentin. Evidence from several studies supports the hypothesis that cohesive failure in dentin is related to high bond-strength [27], perhaps because of the strong bonding to dentin developed by universal adhesives [28]; the enhanced wettability of adhesives, which allows better micromechanical retention and chemical interaction between the acidic functional monomer contained in SBU (10-methacryloyloxydecyl dihydrogen phosphate) and calcium in dentin [29]; and the high resinous monomer content in CN and AB, which facilitates strong co-curing with the adhesive. The slight but significant decrease in SBS between CN-SBU and AB-SBU, and the absence of a significant difference in failure pattern, may be attributable to their different flexural moduli (7.4 GPa for CN-LC vs 2.3 GPa for AB-LC). Several studies [30,31] reported that, under the same bonding protocol, SBS values were higher for stiffer materials than for more flexible materials.

When adhesive was not applied, SBS was highest for the SO-Only specimens, most likely because adhesion relies mostly on a functionalized polyacrylic acid of high molecular weight, which is able to facilitate hybridization of the smear layer, and on ionic interactions between calcium contained in dentin and carboxyl groups of MOPOS (for the Modified Polyacid System), as has been reported in RM-GICs [21]. Moreover, even if there is no trace of functional monomer in this material, this hypothesis has been used to explain its good bonding properties [9]. Despite the presence of phosphate dimethacrylates and modified polyacrylic acids in Activa BioActive Restorative [21], which would be expected to provide micromechanical and chemical adhesion, this material had weak self-adhesion to dentin (4.4 MPa) and did not significantly differ from Cention N on untreated dentin (3.0 MPa). These findings accord with those of a previous clinical study reporting dramatic results without prior adhesive application [32]. However, the present results for Cention N are simpler to explain: this material does not contain polyacrylic acid or acidic monomers. These findings support the systematic use of an adhesive system for Activa BioActive Restorative and Cention N.

The present EF-DC (8.0 MPa) group was used to study the self-adhesive properties of an HV-GIC under ideal conditions, since polyacrylic acid was reported to improve dentin bond strength [33]. Interestingly, SBS was better for SO-Only without surface treatment than for EF-DC (even if no significant difference in failure pattern was observed). Indeed, improvement in the adhesion values of adhesive materials was associated with
improved restoration reliability [34]. However, Surefil One is less attractive when used with an adhesive, since Centinon N or Activa BioActive Restorative offer higher SBS.

The groups differed in their interface profiles. CN-SBU, SO-SBU, AB-SBU, SO-Only and EF-DC specimens exhibited close contact between adhesive and dentin or between restorative material and dentin, whereas CN-Only and AB-Only specimens exhibited a gap between dentin and the material. Therefore, the third null hypothesis was rejected. These findings are probably linked to retraction of the acrylic resin in which dentin samples were included: the polymerization stress of this resin should have surpassed the weak bonding properties of these two materials on untreated dentin, as shown in SBS testing.

In CN-SBU, SO-SBU and AB-SBU specimens, some small resinous tags were observed within dentin tubules. These adhesive patterns have been described in many studies that used universal adhesive in self-etch mode and were linked to high bonding strength values [29]. More interestingly, SEM images revealed tags in the SO-Only group, which confirms the SBS values for this group and suggests that the MOPOS contained in these specimens infiltrated the smear layer, thereby partially demineralizing the underlying dentin. This effect could increase the specific surface area and improve microretention by forming a hybrid-like layer, as previously described for RM-GICs [35]. Previous studies have shown that a hybrid-like layer was associated with the higher bond strengths of RM-GICs, as compared with HV-GICs [36]. For technical reasons, and to investigate the internal structure of the tested materials, polished samples were treated with orthophosphoric acid and dehydrated with ethanol before SEM analysis. Acid attacks were previously shown to be sufficient to modify the surface state of samples [37] or even to dissolve the partially reactive FAS filler [38], which explains the irregular structure of EQUA Forte HT Fil. However, this was not the case in studies using environmental SEM, which required no dehydration [39]. These observations are directly linked to aggressive acid and dehydration treatments conducted on resin-free GICs [37] that contain a substantial amount of water. Considerable filler degradation was observed in Centinon N samples, perhaps because of acid attack by highly reactive fillers, namely, calcium fluorosilicate glass fillers, which the manufacturer and some studies [15, 40] claim are activated under acidic conditions. The present results may be clinically relevant. As compared with HV-GIC, all these materials had better flexural strength in light-curing mode (and in self-curing mode, for Centinon N and Activa BioActive Restorative) and thus may be stronger alternatives for posterior restorations with simplified protocols. None of the materials achieved the adhesive values obtained with an adhesive system and had higher SBS values when used with a universal adhesive. However, SBS was better for Surefil One without dentin surface treatment than for EQUA Forte HT Fil. Surefil One might therefore be a promising material for use in difficult clinical situations or for atraumatic restorative treatment. Further in vitro studies are needed to better understand the setting of these materials. In vivo studies of their clinical performance are warranted.

Conflict of interest

The authors declare no conflict of interest.

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