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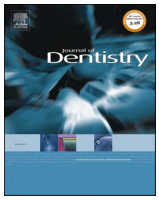

Amalgam alternatives: Susceptibility of novel self-adhesive materials to changes in dentin mineralization

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ABSTRACT

Objectives: This study explores the effects of artificial de- and hypermineralization on the shear bond strength (SBS) of three innovative dental materials: a modern ion – releasing resin–based composite (Cention Forte (CF)), a resin-modified glass ionomer cement (Surefil One (SO)), and a high-viscosity glass ionomer cement (Equia Forte (EF)), all tested on human dentin. Methods: A total of 360 human dentin specimens were divided into 18 groups ($n = 20$). Each material was bonded to either healthy, hypermineralized, or demineralized dentin. After aging at 37 °C in distilled water for 1 week and artificial saliva for 6 months, SBS was tested following ISO 29,022. Fracture origins and patterns were analyzed via light microscopy. Results: The SBS values of EF on hyper- and demineralized dentin were lower after 1 week and 6 months when compared to sound dentin, whereas for CF this trend was only seen on demineralized dentin after 1 week. Regardless of aging, the SBS values of SO displayed no significant difference in comparison to the reference. Across all substrates and aging types, the SBS values of CF were the highest. The long-term bond reliability of CF and EF on sound dentin was similar, as was the bond reliability of CF and SO on hypermineralized dentin. Conclusions: CF produces the strongest bond to dentin irrespective of dentin substrate or aging. Demineralization deteriorates the bond strength of both CF and EF, whereas hypermineralization only that of EF. SO remains unaffected by changes in dentin mineralization, enabling it to form stronger bonds to hypermineralized dentin than EF. Clinical Significance: A proper assessment of a material's bond strength to dentin substrates with varying degrees of mineralization is crucial for evaluating its potential as an effective amalgam replacement, capable of forming reliable bonds across a broad range of tooth substrates

1. Introduction

The modern-day clinical landscape of dentistry is a multifaceted one and it confronts practitioners on a daily basis with a variety of challenges. This is particularly noticeable when observing modern filling therapies. Dentists are often required to manage various tooth substrates (enamel, dentin, cementum) with a specific mineralization degree (demineralized, hypermineralized) of a broad patient spectrum (children, adults, elderly) with differing compliance. The importance of proper management of demineralized dentin surface stems for the most part from modern tendencies regarding caries excavation. The desire to avoid overtreatment of the tooth and unnecessary exposure of the pulp has led practitioners to promote minimally invasive techniques [1], which in turn has been seen as the driving force for the shift from complete to partial caries excavation [2]. Patients with low compliance can be considered an additional vehicle for these changes, since complete caries removal becomes impossible due to time constraints, therefore turning the various methods for partial caries removal into a necessity. As a result a partially demineralized dentin surface, also known as caries – affected dentin, is usually left as bonding substrate for filling materials, which has been proven to be a particularly difficult bonding substrate [3]. At the same time, however, chronic caries lesions can lead to hypermineralization of the adjacent dentin surface [4], which presents itself as a different and completely unique bonding challenge. Hypermineralized dentin should not be solely associated with carious lesions as chronic physical and chemical irritations as well as physiological aging processes are known to induce it [4]. These causing factors combined with the observed increase in population age [5] turn the hypermineralized dentin surface into a bonding substrate with an ever-increasing prevalence.

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These heterogeneous challenges confronting dentists on a daily basis underline the increased need for a versatile material that displays equal bond efficiency regardless of the degree of dentin mineralization, but also has a quick application process with reduced technique sensitivity. For decades, amalgam fillings have played an essential role in oral health care in numerous countries [6] leading to their placement on a wide spectrum of tooth substrates. The reason for this is its simple application process, reduced technique sensitivity, affordable cost and durability [7,8]. The mercury release observed with amalgam fillings, as well as the concerns regarding mercury pollution presents some of the major drawbacks of the material [6,9], leading to the global desire to reduce its placement [6,8]. This has resulted in an increased development and improvement of amalgam alternatives. The fruits of this labor are recognizable when observing resin – based composites (RBC) and glass ionomer cements (GIC), which have been two of the more prominent amalgam alternatives so far. Multiple studies have proven that RBCs are materials with good mechanical and aesthetic properties that allow, unlike amalgam, minimally invasive placement thus reducing unnecessary loss of tooth substrate [8,10]. Despite their high long-term survival rate and good clinical performance [11–13] RBCs are materials that have a lengthy and technique-sensitive application process [8]. In contrast to this, GIC application is simple, fast, and does not necessitate pretreatment of the dentin surface or light curing. However, GICs have shortcomings when it comes to aesthetics and mechanical properties such as flexural strength, compressive strength, and wear resistance [14], which in turn makes them inferior to RBCs [15–17]. This has resulted in a shift in the current scientific efforts as the creation of new hybrid restorative materials that encompasses the beneficial qualities of RBCs and GICs has become the goal [18]. As a consequence of these efforts materials such as resin-modified glass ionomer cements (RMGIC), giomers, and compomers were created [19], which despite their variable clinical success [18] have proven to be inferior to modern RBCs [20].

The apparent necessity for improvement of the existing material classes appears to be addressed by the recent development of materials, which can be seen as an evolution of the following material types – GICs, RMGICs, and RBCs [21]. Newly developed GICs, labeled as high-viscosity glass ionomer cements (HVGIC), seem to display an increase in flexural strength and wear resistance due to changes in fillers and polyacrylic acid [18]. Novel RMGIC encompass complex polymerizable acid monomers to improve setting reaction and ability to bond to the tooth structure [22]. In addition alkaline fillers incorporated in the structure of novel RBC enable the release of caries-protective ions such as calcium, phosphate and fluorite [23].

Due to these improvements and the ability of all three material categories to be placed quickly and in bulk the question arises whether they may serve as an amalgam alternative. Their ability to create a sufficient and reliable bond to various tooth substrates and conditions is an essential part of the answer to this question. Therefore the goal of this study was to quantify the bond strength of the novel materials, the reliability of the bond as well as the fracture pattern when placed on sound, artificially hyper-, or demineralized dentin. The necessary tests and observations were conducted after 1 week and 6 months in order to assess the influence of aging. The null hypotheses developed were that all materials display similar bond strength and bond reliability to dentin, while neither of them is susceptible to the degree of dentin mineralization or aging.

2. Materials and methods

2.1. Materials and their handling

The composition, manufacturer and batch number of each material used in this study is listed in Table 1. Equia Forte (EF) and Cention Forte (CF) were both bonded to the dentin after prior conditioning of the bonding area, while Surefil One was administered directly. For EF, a dentin conditioner (Cavity Conditioner, GC Corp., Tokyo, Japan), was applied to the dentin surface for 10 s and rinsed thoroughly, after which the dentin surface was air dried. In the case of CF, a primer application of 10 s was followed by removal of excess primer by air drying until a thin, immobile layer of primer covered the specimen area. Bonding to the dentin surface occurred with the help of a clamp and a mold insert (Ultradent, South Jordan, Utah, USA). The materials were pressed against the dentin surface using a hand instrument through the central opening of the mold insert, resulting in cylindrical build-ups approximatively 3 mm high and an average diameter of 2.5 mm. The exact diameter was measured for each sample using an optical microscope. The top layer of the EF build-ups was covered with a coating material (EF Forte Coat, GC Corp., Tokyo, Japan). CF, SO and the EF coating were light cured for 20 s by placing a light curing unit (violet-blue LED, Bluephase® Style, Ivoclar Vivadent, Schaan, Liechtenstein) with an irradiance of 1544 ± 207 mW/cm^2 and a light emitting window of 10 mm diameter above the central opening of the mold insert.

Table 1
Chemical compositions of all used materials as provided by the manufacturer.

Name (Abbreviation)	Components	LOT
Equia Forte HT (EF)	Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: polybasic carboxylic acid, water	220119A
Equia Forte Coat	Methyl methacrylate, colloidal silica, camphorquinone, urethane methacrylate, phosphoric ester monomer	2202,241
GC Cavity Conditioner	Polyacrylic acid, aluminium chloride hexahydrate	2110,271
Surefil One (SO)	Powder: silanated aluminium-phosphor-strontiumsodium-fluoro-silicate glass, dispersed silicon dioxide, ytterbium fluoride, pigments Liquid: acrylic acid, polycarboxylic acid, bifunctional acrylate, self-cure initiator, camphorquinone, stabilizer	2207,000,201
Cention Forte (CF)	Calcium-fluorosilicate glass, barium-aluminium silicate glass, copolymer, calcium-barium-aluminium fluorosilicate glass, UDMA, ytterbium trifluorid, aromatic aliphatic UDMA, PEG-400 DMA	ZL08SV
Cention Forte Primer	HEMA, MDP, Bis-GMA, D3MA, ethanol, methacrylate-modified polyacrylic acid, silicon dioxide, potassium hydroxide, campherquinone	Z03F0P

Abbreviations: UDMA – Urethane dimethacrylate; PEG-400 DMA – polyethylene glycol 400 dimethacrylate; HEMA – hydroxyl ethyl methacrylate; MDP - Methacryloyloxydecyl dihydrogen phosphate; Bis – GMA – bisphenol A-Glycidyl methacrylate; D3MA - 1,10-decanediol dimethacrylate.

2.2. Preparation of dentin specimens

The teeth used in this study were collected anonymously, therefore no patient data was acquired. Regarding this research project, the ethical committee issued a statement under project number 22–0472 KB confirming that no consultation was required. A total of 75 sound third molars were stored in a 0.2 % sodium azide solution at room temperature for no longer than three months. The teeth were subsequently sawn with a slow-speed diamond saw (IsoMet, Buehler, Lake Bluff, IL, USA). Each tooth received two horizontal cuts in a bucco-lingual direction: one at the height of the tooth equator and a second one at the cementoenamel junction. The two tooth discs with exposed dentin surface received further vertical cuts which resulted in the final dentin specimens. The number of vertical cuts was adjusted according to the size of each tooth, yielding a minimum of 4 specimens per tooth and in few situations 6 or 8 specimens. In total, 360 dentin specimens were obtained and randomized into eighteen groups (n = 20), with group composition controlled to prevent inclusion of multiple specimens from the same tooth. The specimens were then embedded in methacrylate

resin (Technovit 4004, Kulzer, Hanau, Germany) using stainless-steel cylinders with a diameter of 16 mm. In order to create a standardized smear layer, all of the dentin specimens were ground on a grinding machine (Leco SS-200, Leco, St.Joseph, MI, USA) with a p1200 silicon carbide paper (Leco, St. Joseph, MI, USA). For dentin specimens that required to be artificially de- or hypermineralized the corresponding protocol was conducted prior to bonding with the material. After bonding all specimens were placed in a humidity bath at 37 °C for 1 hour, after which the clamp and the mold insert were carefully removed. The bonded specimens were then stored at 37 °C in either distilled water for 1 week or artificial saliva for 6 months.

2.3. Artificial de- and hypermineralization

Artificial demineralization was achieved through a fourteen-day-long pH-cycling protocol [24]. Every day the specimens were placed at room temperature for eight hours in a demineralizing and for sixteen hours in a remineralizing solution, both of which were renewed daily. The specimens were always rinsed with deionized water before placing them in the respective solution. The demineralizing solution consisted of 1.5 mM $CaCl_2$, 0.9 mM KH_2PO_4 and was adjusted to a pH of 4.8 with a 50 mM solution of 100 % pure acetic acid. The remineralizing solution was mixed with 1.5 mM $CaCl_2$, 0.9 mM NaH_2PO_4, 0.13 M KCl and was adjusted to a pH of 7.0 with a 20 mM solution of a HEPES buffer. Prior to putting the specimens into the solution, each surface of the acrylic resin embedment as well as the enamel portion of the tooth specimen was covered with nail varnish.

Preceding the artificial hypermineralization, the dentin surface of every specimen was etched for 5 s with 35 % phosphoric acid, therefore removing the smear layer. Subsequently, the dentin specimens were kept for fourteen days in a mineralizing solution (pH = 7) consisting of 1.5 mM $CaCl_2{\cdot}2H_2O$, 0.9 mM K_2PO_4 and 0.15 M KCl [25]. The solution was also changed daily. The dentin specimens underwent the same nail varnish procedure as the one conducted for the demineralization groups.

The storage solution was constantly stirred at room temperature by a magnetic stirring machine (Ikamag RCT, IKA, Staufen, Deutschland) with a speed of approximately 200 rpm.

2.4. Shear bond strength (SBS) test

Each bonded specimen was visually inspected for cracks or defects with dental loupes before being placed in a universal testing machine (Z2.5, Zwick/Roell, Ulm, Germany). The SBS measurement adhered to an adapted ISO 29,022 (ISO 29,022:2013) [26] since a straight-edged instead of a notched-edged chisel at a crosshead speed of 0.5 mm/min was used. The SBS values were calculated by dividing the maximum load at fracture by the individual bonding surface area. A light microscope was used to determine the bonding surface area of each specimen.

2.5. Fractographic analysis

After completion of the SBS measurements, every specimen underwent a fractographic analysis via a light microscope (Stemi 508, Carl Zeiss Microscopy GmbH, Göttingen, Germany). Images of the bonding surface of both the cylindrical material buildup and dentin substrate were taken with a camera extension (Axiocam color 305, Carl Zeiss Microscopy GmbH, Göttingen, Germany) and examined with AxioVision computer software. The observed fracture pattern were grouped into three categories: adhesive (the fracture runs in a line between the material and dentin surface on >75 % of the examined surface), cohesive (fracture line goes through either the material or dentin substrate on >75 % of the examined surface), and mixed (fracture line exhibits adhesive and cohesive attributes).

2.6. Scanning electron microscopy (SEM) examination

Two additional teeth were taken in order to conduct a SEM analysis of the effects of the implemented de- and hypermineralization protocols. Each tooth was horizontally cut with a slow-speed diamond saw at the level of the tooth equator and cementoenamel junction but no further cuts were executed. The tooth discs were then subsequently ground on a grinding machine (Leco SS-200, Leco, St.Joseph, MI, USA) with a p1200 silicon carbide paper. While adhering to the corresponding protocol steps one tooth disc was used for the demineralization and the other one for the hypermineralization. After completing the demineralization and hypermineralization processes, a vertical cut was made into the tooth discs to allow for sagittal inspection of their surfaces. A dehydration protocol followed, during which the SEM specimens were placed in five ethanol solutions with ascending concentration for a specific time period: 25 % - 20 min; 50 % - 20 min; 75 % - 20 min; 95 % - 30 min; ≥99.8 % - 60 min. A 25 nm thick gold layer was applied via gold-sputtering before the specimens were inspected with an electron microscope (Zeiss Supra 55 V P, Carl Zeiss AG, Oberkochen, Germany) at a beam voltage of 10 kV.

2.7. Statistical analysis

The data was tested for normal distribution with a Shapiro-Wilk test and a Levene's test for homogeneity of variance. A univariate analysis of variance was applied in order to assess the influence of the main parameters: material (EF, CF, SO), dentin substrate (sound, de- and hypermineralized) and aging (one week, 6 months). A further comparison between the examined parameters was carried out via one-way ANOVA, Tukey's post-hoc test ($\alpha = 0.05$) and a Student́s *t*-test (SPSS 27, IBM SPSS Statistics, Version 27, International Business Machines Corporation, NY, USA). The bond reliability was tested by a Weibull analysis.

3. Results

The results of the SBS measurements and the Weibull analysis are presented in Table 2. Shapiro-Wilk tests confirmed normal data distribution. Levenés test revealed homogeneity of variance for the 1-week CF and EF groups and for the 6-month CF groups. As there were no pretest failures all the groups had equal sample sizes ($n = 20$), therefore further statistical analysis was carried out with the use of parametric tests (ANOVA and Tukey's post hoc test).

The univariate analysis of variance confirmed that all three factors examined (material, dentin substrate, and aging) had a significant impact on the SBS ($p < 0.001$). The material type exhibited the highest impact (partial eta squared $\eta_p^2 = 0.580$, $p < 0.001$) followed by the dentin substrate ($\eta_p^2 = 0.130$, $p < 0.001$), while the effect of aging was significant but very low ($\eta_p^2 = 0.017$, $p = 0.016$). The binary combinations of material × dentin substrate ($\eta_p^2 = 0.154$, $p < 0.001$), material × aging ($\eta_p^2 = 0.024$, $p = 0.015$), dentin substrate × aging ($\eta_p^2 = 0.025$, $p = 0.013$) together with the ternary combination material × dentin substrate × aging ($\eta_p^2 = 0.045$, $p = 0.004$) also displayed a significant but low effect on SBS.

A one-way ANOVA comparison of the materials revealed that CF SBS values are superior to those of SO and EF across all dentin substrates and aging conditions ($p < 0.001$) (Fig 1). Regardless of aging, SO displays higher SBS values than EF ($p < 0.001$) only when bonded to a hypermineralized surface.

When compared to sound dentin, a demineralized dentin surface decreases the SBS values of CF ($p = 0.016$) and EF ($p = 0.032$) after 1 week, whereas SO ($p = 0.467$) bond strength remains unchanged. A hypermineralized surface has no effect on the SO ($p = 0.130$) and CF ($p = 0.076$) bond strength after 1 week but does significantly deteriorate the EF ($p = 0.002$) bond strength values. This trend on the hypermineralized surface persists after 6 months, whereas that on the

Table 2
Shear bond strength (SBS) in MPa (mean ± standard deviation), Weibull modulus (m) with 95 % confidence interval (CI) in brackets and R2 values for both aging conditions. Lowercase or uppercase letters indicate significant differences within one aging condition; Cention Forte (CF); Surefil One (SO); Equia Frote (EF).

1 week									
Groups	CF			SO			EF		
	SBS	m	R^2	SBS	m	R^2	SBS	m	R^2
sound dentin	17.8 ±7.2[a]	2.8 (2.6; 3)	0.98	5 ±2.6[c]	2.2 (1.8; 2.5)	0.9	5 ±2.6[f]	2.1 (2.0; 2.3)	0.97
hypermin. dentin	22.7 ±7.3[a]	3.7 (3.2; 4.2)	0.91	7.2 ±5.0[c,d]	1.4 (1.2; 1.6)	0.94	2.7 ±1.5[h]	1.3 (1.1; 1.6)	0.87
demin. dentin	11.4 ±6.5[b]	2.1 (1.7; 2.4)	0.87	3.7 ±1.9[c,e]	2.3 (2.1; 2.5)	0.97	3.3 ±1.9[h]	2.1 (1.9; 2.4)	0.95
6 months									
Groups	CF			SO			EF		
	SBS	m	R^2	SBS	m	R^2	SBS	m	R^2
sound dentin	17.5 ±5.6[A]	3.6 (3; 4.3)	0.87	9 ±5.0[B]	1.9 (1.7; 2.1)	0.95	10.4 ±2.2[D]	4.9 (4.2: 5.6)	0.92
hypermin. dentin	18.8 ±7.2[A]	3 (2.8; 3.2)	0.98	11.4 ±5.8[B]	2.5 (2.0; 2.9)	0.85	0.5 ±1.1[E]	0.7 (0.4; 1.0)	0.51
demin. dentin	13.9 ±7.4[A]	1.9 (1.7; 2.2)	0.94	4.8 ±3.0[C]	1.5 (1.2; 1.7)	0.89	3.8 ±2.9[F]	0.6 (0.5; 0.8)	0.89

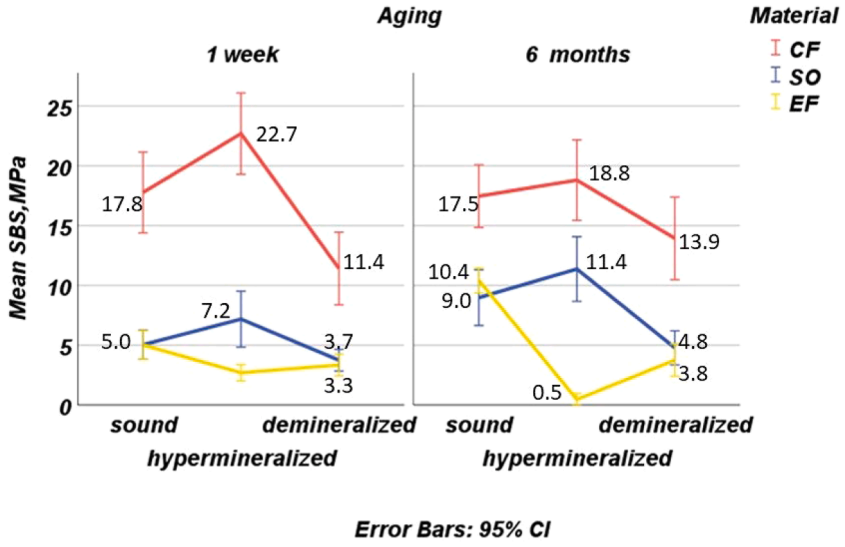

Fig. 1. Shear bond strength (SBS) in MPa of Cention Forte (CF), Surefil One (SO), Equia Forte (EF) after 1 week and 6 months of aging.

demineralized surface changes. In comparison to sound dentin, CF SBS values on demineralized dentin are similar ($p = 0.234$), while those of SO ($p = 0.019$) and EF ($p < 0.001$) are inferior.

During aging, SBS values were preserved across all substrates for CF (sound, hyper- and demineralized dentin; $p = 0.882$; 0.097; 0.258; Student's *t*-test), whereas for SO the values increased on sound ($p = 0.003$) and hypermineralized ($p = 0.019$), yet persisted on demineralized ($p = 0.209$) dentin. For EF, SBS increased with aging only on sound dentin ($p < 0.001$), was preserved on demineralized ($p = 0.593$), but decreased on hypermineralized dentin ($p < 0.001$).

Weibull analysis of the SBS data showed that, compared to sound dentin, hypermineralization enhances CF bond reliability, while demineralization diminishes it after 1 week (Fig. 2). As a result of this CF bond reliability is the highest on hypermineralized dentin, while on demineralized dentin it equals that of EF and SO (Table 2). These trends persist on demineralized dentin after 6 months, whereas those on sound and hypermineralized dentin change. The bond reliability of CF and EF on sound dentin is similar, as is the bond reliability of CF and SO on hypermineralized dentin. Aging deteriorates EF and SO bond reliability on demineralized dentin, whereas that of CF remains unaffected across all substrates. Improvements of the bond reliability due to aging can be seen with SO on hypermineralized dentin and with EF on sound dentin.

Fig. 2 shows as an example the Weibull distribution of the SBS as a function of dentin condition and aging for the material CF.

Fractographic analysis revealed that adhesive fracture was the predominant fracture mode for all materials, regardless of aging. After 1 week the highest amount of adhesive fractures was recorded for EF specimens (96 %), followed by SO (92 %) and CF (74 %). This trend was largely maintained after aging (96 % (EF), 97 % (SO), and 93 % (CF)).

The SEM images in Fig. 3 show that the implemented protocol for artificial dentin hypermineralization creates a thin hypermineralized layer that is loosely bonded to the underlying dentin surface. The pH – cycling model used leads to homogenous surface demineralization, as evidenced by the uniformly exposed dentin tubules.

4. Discussion

The growing demand for amalgam alternatives in dentistry highlights the need for materials with simplified application, strong adhesion, and bulk-fill capabilities. An ideal substitute must also bond effectively to various tooth substrates, particularly dentin, which presents challenges due to its structural peculiarities and varying degrees of mineralization (hyper- and demineralization). This study therefore focuses on how different materials address these bonding challenges

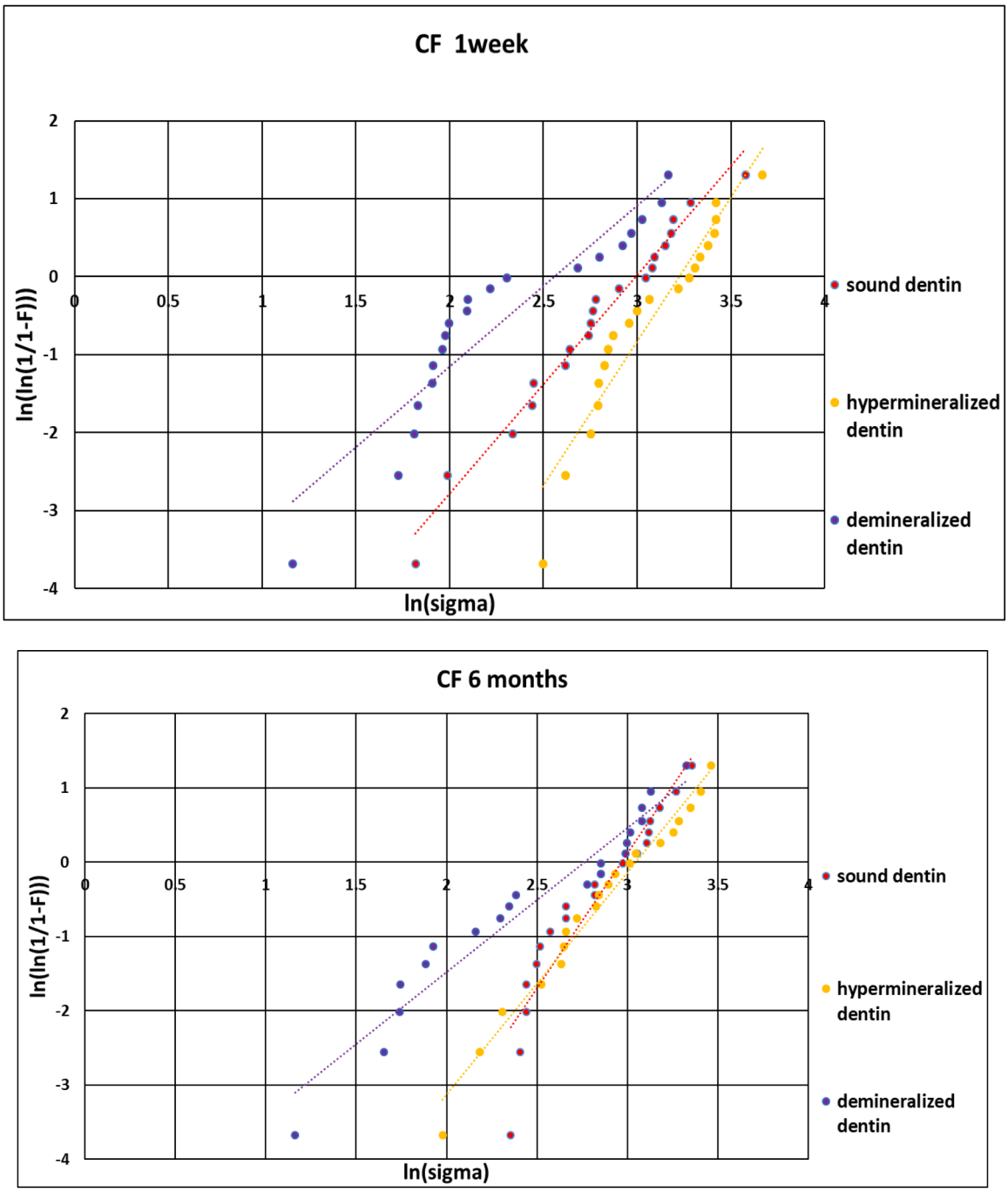

Fig. 2. Weibull analysis of the shear bond strength values exemplified for the material CF values after 1 week and 6 months of aging.

through their specific adhesive strategies.

In the case of EF, ultrafine, highly reactive glass particles [27] as well as a high molecular weight polyacrylic acid [14] have been incorporated into the material structure. As a consequence of these changes, the material exhibits fewer cohesive fractures and increased flexural strength [18], a faster setting reaction that alleviates early moisture sensitivity [28], and has higher shear bond strength to dentin compared with former GICs [29]. The use of dentin conditioner is advised, as the polyacrylic acid plays an essential role in GIC adhesion [30].

SO is a dual-cure material, common in RMGICs, so its setting reaction consists of two simultaneous processes – an acid-base reaction and a radical polymerization of methacrylate monomers by light [21]. As these coexisting processes compete with each other [18], they limit each other [31], which in turn causes RMGICs to exhibit increased sensitivity to hydrolysis [32]. The reason for this increased hydrolysis risk lies within the greater water absorption caused by the hydrophilicity of unreacted HEMA monomers [18]. These well-known drawbacks of RMGIC were mitigated in SO by adding polymerizable acid monomers such as t-butyl acrylate and amnio-propylvinylether to the polyacrylic acid [22]. Due to these modifications, the polyacrylic acid of SO can not only hybridize the smear layer but also create ionic bonds between its carboxyl groups and the calcium in dentin [33]. This means that SO can build good bonds to dentin even without the use of an adhesive [33].

CF is a self-adhesive polymer-based composite in whose structure alkaline fillers (SiO_2–CaO–CaF_2–Na_2O glass; 24.6 wt%) are incorporated within, which is why it is usually referred to as alkasite. In comparison to its predecessor, Cention N, a primer is applied to the tooth surface prior to the restorative material application. This additional step aims to

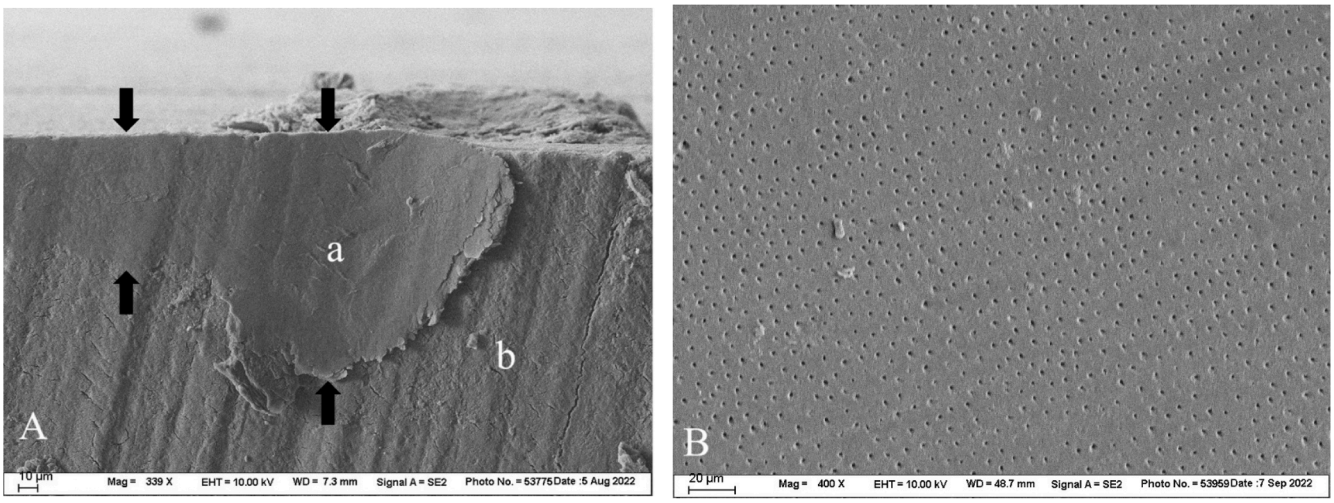

Fig. 3. SEM images of a hypermineralized (A) and demineralized (B) dentin surface. Markings *a* = hypermineralized layer; *b* = sound dentin surface; the space between the arrows indicates the width of the hypermineralized layer.

address some of the observed drawbacks of Cention N – such as poor marginal adaptation and low bond strength values [34,33]. The mechanical properties of CF not only resemble those of regular nano- and micro-hybrid RBCs, but also show a lack of susceptibility to acidic or neutral storage conditions for up to three months [35]. CF is capable of releasing acid-neutralizing ions from its alkaline fillers, allowing it to prevent tooth demineralization at the restoration margin, increase pH, and form fluorapatite in phosphate-containing media [23,36–39]. Bulk application is possible with CF and the material exhibits constant mechanical properties regardless of whether it is light cured or not when applied in increments of up to 4 mm [35].

The results of the current study revealed that all three materials differed in their bond strength to dentin, which rejected our first null hypothesis. CF consistently displayed the strongest bond to dentin, regardless of dentin substrate condition or aging. There is currently a lack of comparable data in the literature. For the previous material, Cention N, Francois et al [33] tested its SBS using an experimental design similar to that of the present study, though aging data were not included. The SBS values of Cention N (3.0 ± 1.0 MPa) were not only lower than those of CF in this study (17.8 ± 7.2 MPa), but the same authors also note a large increase in SBS (33.8; ±5.7 MPa) when an adhesive is used [33]. These findings clearly justify the need of the primer in the current, improved material version.

The polyacrylic acid in the dentin conditioner of EF aims to remove the dentin smear layer, create mechanical retentions in the tooth surface, and enhance the marginal seal of the material [40]. In contrast, the polyacrylic acid in SO, due to the polymerizable acid monomers can implement a hybridization of the smear layer [33]. Our results highlight that the effect of these two different bonding strategies is equal when the corresponding materials are bonded to a sound or demineralized dentin surface, but not to a hypermineralized substrate, where the modified polyacrylic acids in SO cope with this condition better than the dentin conditioner of EF. The simulated aging further support this assumption as hypermineralization of the dentin surface leads to severely reduced SBS values for EF after 6 months (0.5 MPa (±1.1)).

Various studies have shown that bonding to caries-affected dentin leads to lower bond strength compared to sound dentin [41,42]. Common features of caries – affected dentin is a reduced number of apatite crystals due to dissolution [43], an intertubular dentin with increased porosity [44] and the occlusion of dentin tubules by whitlockite crystals [45]. The smear layer of caries – affected dentin also exhibits structural changes, as it has greater thickness, a higher degree of structural irregularities and more organic components such as collagen compared to sound dentin [3,45]. The reduced amount of calcium ions due to the loss of hydroxyapatite crystals impairs the GIC or RMGIC bond to caries – affected dentin as fewer chemical bonds between the carboxyl groups of the polyalkeonic acid and the calcium ions of the dentin hydroxyapatite can be built [41]. Dentin hybridization of caries – affected dentin is also hindered [46,47] as the collagen enriched smear layer together with the acid resistant whitlockite crystals are impermeable to monomers [45]. This in turn causes a degradation of unprotected collagen by host-mediated enzymes such as matrix metalloproteinase thus reducing the overall bond strength of the material to dentin [48,49]. As a result of this, the drawbacks of bonding to caries – affected dentin have been observed for various materials or bonding techniques such as: self – etch adhesive systems, GICs and RMGICs [3,41,45]. The CF and EF results in this study support these findings as both materials displayed lower bond strengths after 1 week when bonded to simulated caries – affected dentin, therefore rejecting our second null hypothesis. In the case of CF, the lack of a statistically significant difference between the SBS values of sound and demineralized dentin after aging could have been caused by a relatively high standard deviation that potentially stems from the individual peculiarities of the dentin substrates. The SO SBS values, however, contradict the previously described observations as the material is not influenced by demineralization of the dentin surface, suggesting that the inclusion of polymerizable acid monomers to the polyacrylic acid of SO enables it to overcome the obstacles presented to it by the demineralized dentin substrate. The statistically significant difference displayed by the SBS values of SO and EF on sound and demineralized dentin after 6 months is not caused by an increased effect of demineralization as aging had no influence on it. The origin of this result lies within the increase of SO and EF SBS values on sound dentin over time due to their maturation processes.

It is important to note that a clear distinction should be made between *in vitro* and *In vivo* demineralized dentin. The used *in vitro* model for demineralization aims to simulate caries-affected dentin [25,50] which differs from its sound counterpart not only by its degree of mineralization, as it represents a complex interconnected system of organic and inorganic factors. The occlusion of dentin tubules with whitlockite crystals, the collagen-rich smear layer as well as collagen with reduced cross – links [3] are aspects of *In vivo* caries – affected dentin that cannot be reproduced by a pH – cycling model as it mainly simulates the chemical changes that occur during a caries lesion [50,51]. The results of the SEM analysis conducted within this study not only confirm this demineralizing effect of the pH-cycling model but they are also identical to those of other studies that have implemented the same pH-cycling model [50,52]. It has, however, been reported that this type of demineralization is shallower when compared to naturally created

caries defects, because vital factors of the oral environment, such as biofilm and saliva, are missing [50]. Despite these drawbacks, the pH-cycling method is still better suited than acidifying gels or microbiological models at representing caries-affected dentin [50]. However, it is important to note that while a pH-cycling model can create a demineralized dentin surface resembling caries-affected dentin, it cannot fully replicate the clinical situation. Therefore, laboratory simulations cannot replace the need for clinical studies on caries-affected dentin. Results from *in vitro* simulations should be regarded as indicative of the expected interactions between corresponding materials and caries-affected dentin in a clinical context.

In vivo hypermineralized dentin is an especially challenging bonding substrate as its characteristic features such as hypermineralized layers of mineral deposits, mineral occlusion of dentin tubules as well as bacterial layers with mineralized bacterial matrices act as a diffusion barrier [53,54]. The crystals found in the mineral accumulations of hypermineralized dentin exhibit an increased acid resistance when compared to hydroxyapatite, which further hinders bonding to hypermineralized dentin [53]. In that sense various authors report on the negative effect of hypermineralized dentin on the bonding potential of different adhesive systems [25,53,54]. The CF and SO results seen in this study, however, contradict these observations, because the bond strengths of neither material is negatively impacted by a hypermineralized surface. A possible explanation for this is that the MDP monomers in the CF primer and the acidic monomers within SO are able to create sufficient dentin bonds by overcoming the obstacles presented by the hypermineralized surface. This assumption is further supported by observations from other authors, who found that the bond strength of adhesives with mild acidity or those containing MDP remains unchanged when applied to a surface that was hypermineralized using the same protocol presented in this study [55,56]. In contrast to this, the EF results in this study imply that the polyacrylic acid in the dentin conditioner of EF appears to lack the strength to overcome the hypermineralized layer as the material displays significantly lower SBS values on hypermineralized dentin when compared to those on sound dentin. As of now, no further works report on the GIC bond to artificially hypermineralized dentin surface, however the authors of a clinical study examining the survival rate of EF when bonded to sclerotic dentin expressed the same theory [57]. Nevertheless, direct correlation between the results of laboratory and clinical studies of hypermineralized dentin should be avoided as the *in vitro* hypermineralization method implemented in the present study is unable to mimic the biological aspects i.e. bacterial layers and mineralized bacterial matrices, of an *In vivo* hypermineralized dentin. The SEM analysis in the presented work (Fig 3) confirms that this model for artificial hypermineralization leads to the creation of a thin layer that appears to be loosely bonded to the dentin surface. This finding adds further explanation to the observed low SBS values on hypermineralized dentin in EF, as the material appears to bond only to the hypermineralized layer. Therefore, it can be assumed that the measured SBS values are a representation of the loose bond between the hypermineralized layer and the underlying dentin structures. This hypermineralized layer appears, however, to lack acid resistant crystals as other authors have observed that the artificially hypermineralized surface exhibited good adhesive penetration and a demineralization similar to that of sound dentin [53]. Despite the discrepancies between this *in vitro* model and the clinical hypermineralized dentin, this method for *in vitro* dentin hypermineralization still creates a layer of precipitated minerals that should be seen as a real bonding obstacle for materials. As such, it provides valuable insight into whether the materials are able to overcome such a barrier.

Modern dentistry demands adhesive materials capable of forming bonds with tooth surfaces that can endure various physical, chemical, and mechanical stresses in the clinical environment for many years. Water is one of the more notable examples for such a stress as over time it can lead to a degradation of the mechanical qualities of resin – based composites. Repeated water exposure primarily leads to the hydrolysis of ester bonds in methacrylates, as these bonds are most susceptible to water [58,59]. Similarly, though to a lesser extent, carbonate, carbamate, urethane, and amide bonds are also affected by hydrolysis [58]. Additionally a degradation of the silane interface [60] as well as an elution of fillers or unreacted monomers can be observed [61,62]. The 6 months SBS values of CF indicate that the material is not susceptible to water induced degradation as its bond strength exhibits no changes after prolonged exposure to artificial saliva. For GICs water exposure in the initial setting reaction is known to have a detrimental effect on the mechanical properties of the materials [63]. At the same time, GICs improve their mechanical properties during maturation as a series of additional setting reactions, during which an increase in the physical qualities of the material is observed [40]. The maturation process can indeed be recognized in SO and EF when bonded to a sound dentin surface, where their respective SBS values doubled over the course of 6 months. However, a hypermineralized dentin surface appears to be a substrate where GIC maturation does not necessarily lead to higher SBS values. While SBS values increase after aging in SO, those of EF experience a severe drop. In this case, the key factors seem to be the material's ability to penetrate the hypermineralized layer in order to reach the underlying dentin surface and the strength of the bond between the hypermineralized layer and the adjacent dentin structures. As a result, SO can display a growth in SBS after 6 months just as it does on sound dentin. Due to EF binding primarily to the hypermineralized layer poorly bonded to the dentin surface the material's maturation can have no influence on its bond strength. In addition, the bond between the hypermineralized layer and the adjacent dentin appears to be susceptible to the degradation effect of artificial saliva as can be seen by the drop of the SBS values after 6 months. It is important to note that the SO and EF bond on demineralized dentin shows no susceptibility to the degradation effect of artificial saliva as their respective SBS values display no significant changes after 6 months.

Macro SBS tests are one of the most commonly implemented techniques for examining the bond strength of dental materials to the tooth surface [64]. Especially when assessing materials with generally lower bond strengths, such as GICs, the use of macro SBS tests has proven to be more beneficial than other tests [65]. At the same time, however, this test type does exhibit some drawbacks as a variety of factors such as the tooth or material characteristics, the storage conditions, and the applied crosshead speed can influence it [64]. Materials with a high E-modulus tend to exhibit higher bond strengths [66,67] which often leads to a cohesive failure [64]. The fractography results of our study seem to support these observations as CF exhibited more cohesive fractures than SO and EF. Despite this, all three materials predominantly experienced an adhesive failure. Even though it has been observed that shear stress is unequally distributed and not -solely applied to the adhesive interface [64], adhesive failures give a more accurate representation of the shear bond strength of the adhesive interface. The information gained from cohesive failures tends to be more misleading because it represents the structural strength of either the material or tooth substrate rather than the shear bond strength of the adhesive interface.

When evaluating the bonding of a material to dentin, it is crucial to consider not only bond strength but also bond reliability. To assess bond reliability, a Weibull analysis is required, where the Weibull modulus (m) serves as an indicator: the higher the modulus, the greater the bond reliability of the material [68]. Through a Weibull analysis smaller differences between the tested materials can be detected that otherwise would have remained unrecognized in a macro SBS test. In that sense, the SBS results on hypermineralized dentin for CF and SO would imply that the sole advantage of CF over SO is its higher bond strength. CF does, however, exhibit after 1 week a higher Weibull modulus than SO on hypermineralized dentin suggesting that the MDP in the CF primer overcomes the hypermineralized layer more consistently than the acid monomers of SO. In addition to that, the Weibull modulus measured after 1 week on hypermineralized dentin in CF implies that the material not only overcomes the hyperminerlized layer but also builds surface

bonds that are more stable and consistent than those on sound dentin. The demineralization, on the other hand, weakens after 1 week not only the SBS but also the bond reliability of CF to such an extent that it equals the bond reliability of EF and SO. According to the SBS results, aging has no influence on bond strength when EF and SO are bonded to demineralized dentin. The Weibull analysis refined these findings, as the decreased Weibull moduli during aging can be indeed interpreted as a sign of degradation. Moreover, the beneficial effects of material maturation is reflected also in the reliability, as the bond reliability on sound dentin increases in EF after maturation to the point where it matches that of CF on the same substrate. In contrast, the maturation in SO manifests itself differently, since the reliability, despite increased SBS values for sound and hypermineralized dentin, only improves with maturation for hypermineralized dentin. In that sense, the assumption can be made that SO stabilizes its bond towards hypermineralized dentin in the long run. Due to this increase in bond stability SO is able to display the same bond reliability as CF on hypermineralized dentin after 6 months. Furthermore, the reliability analysis in EF allows concluding that the bond between the hypermineralized layer and the underlying dentin surface is both unstable and prone to further degradation when exposed to artificial saliva.

5. Conclusion

The bond strength of CF is superior to that of EF and SO, regardless of aging and dentin condition. A partially demineralized dentin surface appears to be detrimental to the bond strength of both CF and EF only in the short term, whereas a hypermineralized dentin surface deteriorates the bond strength of EF irrespective of aging. SO is the only material unaffected by dentin condition, but this does not lead to superior bond strength or reliability.

With maturation, SO improves in bond strength on sound and hypermineralized dentin but in bond reliability only on hypermineralized dentin, while EF shows both improvements only on sound dentin.

CRediT authorship contribution statement

Vasil Christoff: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis. **Nicoleta Ilie:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Conceptualization.

Declaration of competing interest

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Supplementary materials

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