

Case Report

One-Year Clinical Aging of Low Stress Bulk-Fill Flowable Composite in Class II Restorations: A Case Report and Literature Review

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Abstract: Bulk-fill flowable composites provide functional and aesthetic restorations while eliminating incremental composite layering and saving time. The degradation of the adhesive interface with subsequent gap formation is a concern when adhesively luted restorations are placed. Moreover, the number of adhesive interface failures increases when they are exposed to long-term water storage. The aim of the present study was to evaluate the morphological characteristics of the tooth-composite interface in class II cavities restored with a low stress bulk-fill flowable composite after aging in an oral environment. We describe a case of a patient with class II cavities in four premolars restored with a low stress bulk-fill flowable composite Surefil SDR (Dentsply DeTrey GmbH, Konstanz, Germany). The occlusal part was restored with nano-hybrid resin composite Ceram X Mono (Dentsply DeTrey GmbH). After one year of clinical function, the teeth were extracted and examined in a scanning electron microscope (SEM). It can be concluded that the application of bulk-fill covered with conventional composite seems to provide the homogeneous and stable bond to tooth structure after one year of aging in an oral environment. However, some defects within the dentin-resin composite interface were observed.

Keywords: bulk-fill; SEM; adhesive; dentin; enamel; resin

1. Introduction

Resin composites have evolved significantly over the last decades and now we are witnessing the greatest development of these materials [1–3].

Despite numerous advantages, composites exhibit some shortcomings that may adversely influence clinical results. The marginal micro-leakage and polymerization shrinkage still occur, particularly in class II restorations; thus, the adhesive interface becomes the most vulnerable site for the restoration failure [4–7]. Moreover, the interface between tooth structure and high viscosity resin composites that was expected to provide the long-term and stable result may reveal many defects with time. The major cause of this phenomena is the difficulty in homogenous adaptation to the cavity surface [8]. In fact, the gap formation may occur at the margins in enamel and dentin or even along the adhesive system–tooth interface [9–11]. It is believed that there are three major factors leading to this marginal defect. Firstly, the lack of compensation of initial polymerization shrinkage stress occurs prior

to the first occlusal loading. Secondly, the occlusal load results in repeated stress exerted on the resin–tooth interface. Thirdly, the biochemical stress via biofilm accumulation at the restoration margin. Consequently, these phenomena might cumulate and cause damage to the adhesive bond [12].

In an attempt to overcome these drawbacks, a new class of composite materials was introduced into the market [1]. Low-stress bulk-fill flowable composites with enhanced chemical and mechanical properties have been developed in an attempt to provide the durable and uniform seal on dental substrate [13]. Furthermore, these materials have established a new class of resin-based composites that can be adequately photo-polymerized up to (or over) 4 mm of thickness [14]. Thus, the risk of entrapping air voids between the resin increments has been reduced, resulting in the improvement in mechanical strength [15].

Numerous resin composite materials were studied over the past years, including conventional and bulk-fill composites [16–22]. With regards to laboratory outcomes, the bulk-fill materials show better or similar performance than the conventional materials in terms of volumetric shrinkage, polymerization stress, cusps deflection and marginal quality [23]. Moreover, according to the latest systematic review and meta-analysis, the clinical performance of conventional resins and bulk resins for carious lesion restorations is similar after 1 to 10 years of follow up [24].

Surefil SDR (Dentsply, Konstanz, Germany) is a light-cured bulk-filled, fluoride-containing material capable of generating an intimate contact with cavity surfaces [25]. In fact, low-viscosity materials, such as SDR with a reduced amount of inorganic filler (45% in volume), undergo volumetric shrinkage, but with minimal contraction stress [26]. Indeed, it results from the ‘stress decreasing resin’ technology used in this material, that provides greater flexibility when compared with traditional resin materials [27]. However, due to the lower filler content in these composites, they were said to exhibit a lower creep resistance [28]. Therefore, an occlusal capping with a conventional resin-based composite is recommended [29].

It is well-proven that long-term success of restorations, beside the technique used, relies on the bond strength and the durability of adhesion to enamel and dentin [30]. The instability and susceptibility to degradation of adhesive systems lead to the gap formation [31] and may be responsible for the retention loss of the restoration [32]. Interestingly, the resin–dentin interface produced by two-step etch-and-rinse adhesives, degraded with time when directly exposed to water or oral fluids [33]. When exposed to long-term water storage, an increase in the number of adhesive interface failures was demonstrated in *in vitro* studies [34,35].

Thus, the aim of the present study was to evaluate the morphological characteristics of the tooth–composite interface in class II cavities restored with a low-stress bulk-fill flowable composite after aging in an oral environment.

2. Case Presentation

2.1. Materials and Methods

2.1.1. Tooth Restoration and Aging

This study was performed after the approval of the Ethical Committee no.#USJ-2014-43 (Saint-Joseph University).

An 18-year-old female with no significant medical history and with good oral hygiene, permanent dentition and with a severe tooth-arch discrepancy presented to the private practice with multiple proximal carious lesions (score 5 International Caries Detection and Assessment System (ICDAS)) including first upper and second lower premolars: 14, 24, 35, and 45.

The treatment plan included the restoration of compromised teeth after the assessment of radiographic X-ray preoperatively.

After informed consent was obtained, class II cavities (14—II OM, 24—II OD, 35—II OM, and 45—II OD) were prepared using a cylindrical with round carbide burs (010 ISO) with a high-speed handpiece working at 250,000 to 300,000 rpm (under copious water

cooling), and with maximum preservation of sound tooth structure. The caries excavation was performed with round steel burs (014 ISO) with a low-speed handpiece working at 20,000 to 30,000 rpm. The internal line was rounded; the enamel and dentin margins were then prepared with a butt joint using 80 µm grit diamond burs and finished with 25 µm grit diamond burs (Intensiv, Viganello-Lugano, Switzerland) under copious water cooling. Next, the rubber dam isolation (Nic Tone Dental Dam, thick, mint, MDC dental, Zapopan, Mexico) was performed and Palodent V3 Sectional Matrix combined with a separation ring (Palodent, Dentsply Detrey GmbH) and wooden wedge (Polydentia, Mezzovico, Switzerland) were introduced. The cavities were etched with 36% phosphoric acid gel (Conditioner36, Dentsply DeTrey); the enamel was etched for 30 s, and dentin for 15 s. Next, the cavities were rinsed with water for 30 s. The excess of humidity was then removed using absorbent paper points, leaving a glossy and slightly wet surface. Afterwards, a two-step etch-and-rinse adhesive Prime&Bond XP (Dentsply Caulk, Mildford, DE, USA) was applied and photopolymerized according to the manufacturer's recommendation. The low-stress bulk-fill flowable composite Surefil SDR (Dentsply DeTrey GmbH, Konstanz, Germany) was then placed in two increments (each approximately 2 mm). The nano-hybrid resin composite Ceram X Mono (Dentsply DeTrey GmbH) in a contrasting shade (A3) was placed as the last layer, not exceeding 2 mm (occlusal capping). Each layer was polymerized for 20 s by using a light curing unit equipped with a LED light SmartLite iQ2 (Model No. 200, Dentsply Caulk) and an irradiance of 800 mW/cm². The restorations were finished using fine-grit diamond burs and Enhance[®] PoGo[®] (Dentsply Caulk) polishers combined with Prisma[®] Gloss[™] polishing paste (Dentsply Caulk) for 30 s, using a light rotation movement. The applied materials are described in Table 1.

Table 1. Manufacturer and composition of the materials used for performing the restorations.

Adhesive System	Type of Adhesive	Main Components (Lot No.)
Prime&Bond XP (Dentsply Caulk)	Two-step etch-and-rinse	PENTA, UDMA, HEMA, TEGDMA, TCB, tert-butanol, nanofiller, camphorquinone, stabilizer (1105001715)
Resin Composite	Type of Resin Composite	Main Components
Surefil SDR flow (Dentsply DeTrey GmbH)	Bulk-fill flowable	Barium-alumino-fluoro-boro-silicate glass, strontium alumino-fluoro-silicate glass, modified urethane dimethacrylate resin, EBPADMA, TEGDMA, CQ, BHT, UV stabilizer, titanium dioxide, and iron oxide pigments (1105121)
Ceram X Mono (Dentsply DeTrey GmbH)	Nano-hybrid composite	BisGMA, CQ, TEGDMA, UDMA, Ba–Al–borosilicate glass, methacrylate, functionalized silicon dioxide nanofiller, iron oxide pigments, titanium oxide pigments, aluminum sulfo silicate pigments (1106000932)

PENTA: dipentaerythritolpenta-acrylate phosphate; UDMA: urethane dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; TCB: butan-1,2,3,4-tetracarboxylic acid di-2-hydroxyethylmethacrylate ester; EBPADMA: ethoxylated bisphenol A dimethacrylate; BHT: butylated hydroxytoluene; CQ: camphorquinone; Bis-GMA: bisphenol-a-glycidyl methacrylate.

One year later, the patient returned to the clinic looking for orthodontic treatment. The treatment plan included extraction of the upper first and lower second premolars. Once the informed consent was obtained, the premolars were scheduled for extraction.

2.1.2. SEM Study

Premolars were preserved in 0.2% thymol solution for one week in order to minimize bacterial contamination [36]. Next, samples for scanning electron microscope (SEM) images were prepared as described by Lapinska et al. [37]. First, the root was sectioned perpendicularly, and their crowns were embedded in acrylic resin, allowing the buccal enamel surface to be exposed. Then, the vestibular enamel was abraded with an orthodontic grinder (Essencedental, Araraquara/SP, Brazil) until the exposure of a flat medium dentin. The exposed dentin surface was wet polished with increasing grit sizes of SiC abrasive papers: P180 followed by P400 (Carbimet, Buehler, Lake Bluff, IL, USA) and thoroughly

rinsed with water for 10 min. The samples were later dried carefully using absorbent paper (Kimwipes; Kimberly-Clark Professional, Roswell, GA, USA). After 24-h storage in artificial saliva in 37 °C, specimens were etched with 37% orthophosphoric acid for 30 s and rinsed with water. Next, 10% NaOCl was applied for 2 min and rinsed with water.

In this sense, the cross-sections were prepared as followed: enamel/bulk-fill, enamel/dentin, bulk-fill/nano-hybrid composite, enamel/nano-hybrid composite and dentin/bulk-fill. All samples were sputter-coated with a thick layer of gold-palladium (30 nm, Bal-Tec SCD 005, Sputter Coater) for 120 s. SEM EVO50XVPLaB6 (Carl Zeiss; Cambridge, UK) with a lanthanum hexaboride filament for high-resolution images was used for the evaluation. The representative SEM images were captured at 60×, 150×, 650×, 1500×, 4000× and 6000× magnification. SEM operating conditions included 10 kV accelerating voltage, 10 mm working distance and 1.2 mA probe current, spot size 4.0, image resolution 2048 × 1768 pixels, horizontal field width 90.13 μm with a resolution of 45 nm. The observations were made under variable pressure at 0.75 torr using both BSE and SE1 detectors.

2.1.3. Results

Representative SEM micrographs of the tooth–composite interfaces are shown in Figures 1–3. Figure 1a–c show the homogenous interface between tooth structure and composite at different cavity levels. A good adaptation of bulk-fill and nano-hybrid composite to dentin and enamel is presented (Figure 1a,b). In Figure 1c (black arrow) the gap between dentin and bulk-fill-composite is visible; that was the only sample where a gap was observed.

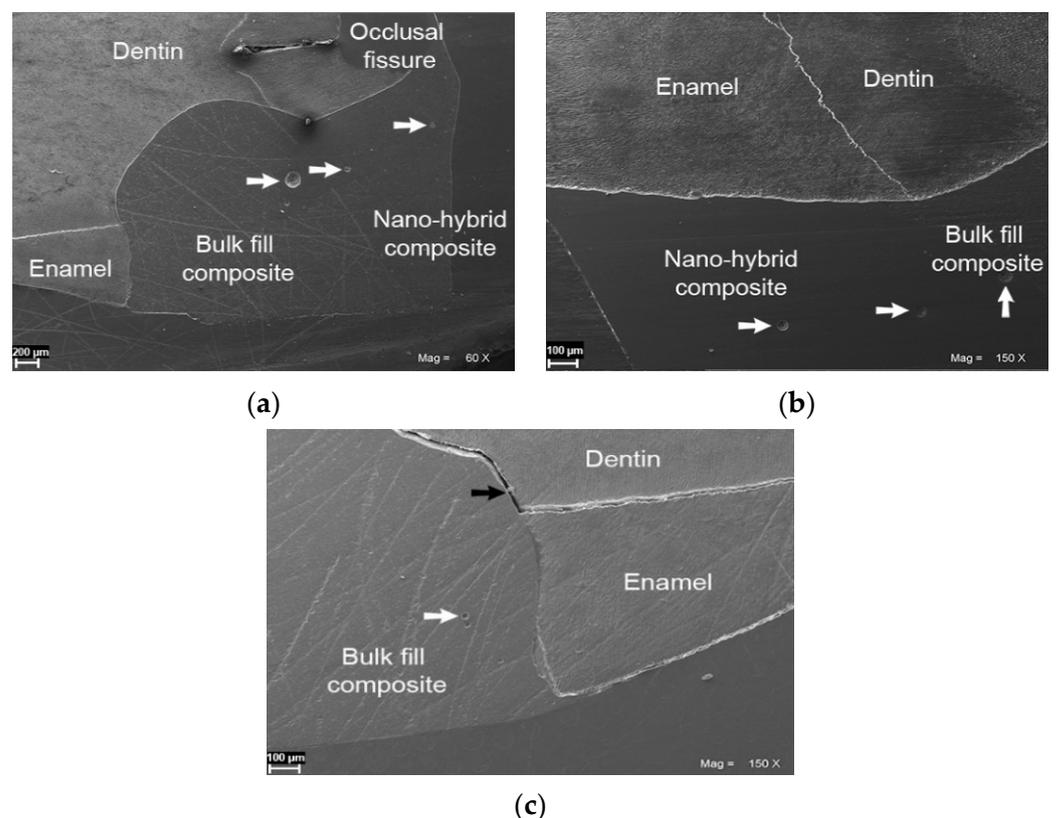


Figure 1. SEM images of (a) the cross-section of the restored tooth, 60×; (b) the interface between enamel, dentin, bulk-fill and nano-hybrid composite at occlusal surface, 150×; (c) the interface between enamel, dentin, bulk-fill composite at gingival margin, 150×. White arrows indicate voids, black arrow—gap.

In Figure 2a, the homogeneous interface between enamel and bulk-fill composite is demonstrated. Figure 2b shows a uniform hybrid layer along with well-formed resin tags in the pulpal wall. Additionally, the sizeable resin tags with numerous lateral branches and a well-defined intact and adequate hybrid layer are highlighted.

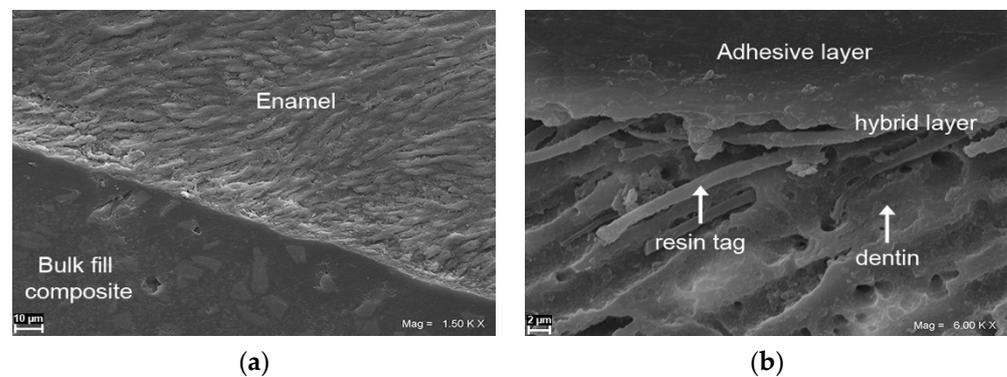


Figure 2. SEM images of (a) the interface between enamel and bulk-fill composite at gingival margin, 1500 \times ; (b) the hybrid layer and resin tags in the pulpal wall, 6000 \times . White arrows indicate resin tag and dentin.

Figure 3a,b show the homogenous interface between bulk-fill and nano-hybrid composite. However, there are voids within the nano-hybrid composite (arrows in Figures 1a–c and 3a,b). Moreover, at higher magnification, small voids are clearly visible (Figure 3b).

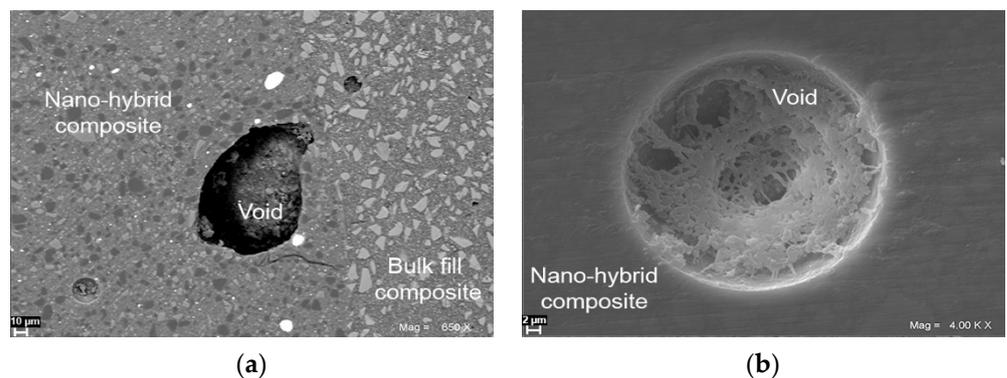


Figure 3. SEM image presenting voids (a) at the interface between bulk-fill composite and nano-hybrid composite, 650 \times ; (b) within the nano-hybrid conventional composite, 4000 \times .

3. Discussion

The stable and homogenous marginal seal to both enamel and dentin is one of the major factors influencing the clinical success of resin-based restorations [38]. While bonding to enamel is a well-established technique with predictable outcomes [39], the adhesion to dentin still remains a challenge [40]. In the present study, the qualitative evaluation of teeth–resin composite interfaces through SEM micrographs showed a good marginal adaptation at all investigated interfaces. However, few defects could be observed at the interface and within the resin composite.

The effective adhesion to enamel requires a micromechanical retention of resin composite on etched enamel by using phosphoric acid, which in turn increases the wettability of the adherent surface and also yields a durable bond [39]. Thereafter, the penetration of bonding resin into the porous zone could be observed which results in the formation of ‘prism-like’ resin tags after the polymerization [41]. It is well-known that the depth of enamel dissolved during the etching process depends on several factors: the duration of etching, the type of acid, the acid concentration and the chemical composition of the enamel [42]. In this study, a strong phosphoric acid was used on enamel, dissolving the

hydroxyapatite [41], and creating a deep enamel etching pattern. The aim of the acid etch in the enamel structure is to modify the surface contour by superficial cleaning and removing approximately 10 μm of nonreactive crystals, leading to an increase in the surface energy and a greater moisture due to the smaller contact angle of the adhesive with the tissue. Structurally speaking, acid etch reacts with the release of carbon and the detachment of calcium and phosphorus, forming irregularities in the inter- and intracrystalline space [43].

It has been extensively acknowledged that resin–enamel bonds are durable and reliable [44,45]. Therefore, the presence of enamel at the cavity margins may provide a perfect seal against the ingress of bacteria and oral fluids. As a consequence, the unstable and vulnerable bond to dentin is being protected. In other words, when cavity margins are surrounded by enamel, the dentin bond is sealed by the overlaying enamel bond, providing long-lasting and stable results [35]. Though controversial [46], increasing the surface area for bonding by beveling the enamel can increase the durability of a restoration [47]. These findings are supported by the present study, as a good marginal seal at the composite–enamel interface was observed (Figures 1a–c and 2a).

Furthermore, the acid etching process demineralizes 5 to 8 μm of the intertubular dentin matrix and creates nanoscale porosities in the underlying collagen fibrillar matrix. As a result, the smear layer is removed and dentinal tubules become opened, exposing the collagen fibers [48]. Thereafter, monomers infiltrate this surface, creating a hybrid layer [49]. The solvent may provide higher stability and compatibility with both polymerizable resins and water [38], increasing the physicochemical stability of the adhesive interface over time [50]. Thus, the hybrid layer is a mixture of dentin, hydroxyapatite, resin monomers and residual solvents; hence, its stability ultimately depends on the resistance of the individual components to the degradation [51]. In general, the more compact and homogeneous the hybrid layer, the more stable resin–dentin bond is created [34,52] (Figure 2b).

In the present study, there were no gaps observed within the enamel–composite interface that provided a perfect seal and prevented bacterial microleakage. However, the composite–dentin interface exhibited gaps in some areas of one sample (Figure 1c). The presence of gaps among this interface may be caused by the insufficient adaptation or adhesion to dentin or the shrinkage of bulk-fill composite [53]. The polymerization shrinkage generates stress at the adhesive interface, and when the polymerization shrinkage is higher than the bond strength, it leads to the formation of marginal cracks, and consequently, gap formation and caries development. It is worth mentioning that gaps within the range of 0 to 70 μm were able to allow biofilm growth in the tooth–restoration interface. [54,55]. Another possible reason for gap formation might be the sample preparation procedure including cutting, polishing, and dehydration. However, if this is true, gaps would be observed both in enamel and dentin, but this was not the case.

As the dentin bonding agent, a two-step etch-and-rinse adhesive Prime&Bond XP was used in this study due to its well-established clinical performance [56–58]. Moreover, the *in vitro* study showed an increase in its reliability (μTBS) by nearly 110% after one year of aging [59]. The formation of a stable and long-lasting hybrid layer by Prime&Bond XP was widely proven [60,61]. Contrary to the findings of our study, a recently published systematic review concluded that, for load-bearing restorations, the performance of two-step etch-and-rinse or one-step self-etch adhesives was not satisfactory [62]. In this sense, it should be highlighted that for a long survival rate for composite restorations, other factors such as the patient and operator should be taken into account.

According to Furness et al. (2014), more gap-free areas at internal margins were found when using a 2-mm compared to a 4-mm thickness increment of bulk-fill composite, although this difference was not statistically significant [63]. An adequate depth of cure of bulk-fill composite is achieved by the reduction in filler content (45% vol) [4] in comparison to traditional resin composites (76% vol) [64]. As a result, SDR was reported to show higher polymerization shrinkage (4% vol) and lower elastic modulus (4–5 GPa) when compared to conventional resins (2.3% vol and 8.5 GPa, respectively) [4,65]. High polymerization shrinkage stress can lead to internal and marginal gap formation [66]. Consequently, marginal

staining, microleakage, secondary caries, enamel cracks, and postoperative sensitivity may occur [67].

The occlusal capping is proven to provide good clinical results in extensive class II cavities in permanent teeth [68]. Advantages of this technique include an increased color stability due to the nearly natural optical properties (value, refractive index) of the enamel layer of the conventional resin [69,70]. These characteristics optimize the final appearance, leading to a more aesthetic restoration [69]. In addition, covering the flowable bulk-fill composite protects it from potential wear [71]. However, it was shown that SDR can be used in deciduous teeth without capping by conventional resin [72].

The micrographs obtained in this study revealed voids within the nano-hybrid composite (Figure 3a,b). The presence of voids within restorations was reported to amount up to 86.4 to 100% of samples [73]. Voids within the composite, also known as bubbles or porosities, could be a result of air entrapment within the material during the manufacturing process. Consequently, they can be incorporated into restoration during cavity reconstruction procedure [74]. Next, the placement method can contribute to the insertion of submicron bubbles or formation of structures and pockets that can easily trap air at the surface [74–77]. However, porosities can be minimized through vacuum loading of syringes and the use of light-cured materials that can be applied in one layer, i.e., bulk-fill materials. The latter provides better adaptation to the surrounding tissue and is less prone to operator-dependent failures [78]. Unfortunately, in such a protocol polymerization, shrinkage of the bulk-fill composite remains a problem [79,80].

Currently, one of the major issues discussed among researchers relates to the validation of *in vitro* tests results in order to determine whether they can correlate positively with the clinical performance of adhesive restorations [81,82]. The best available evidence in this field gives us some clear indications for the correlation of laboratory bond strength tests with the clinical retention rates of class V restorations [83]. In this sense, when an adhesive is tested in *in vitro* conditions, the clinical investigation should be conducted immediately to examine the effectiveness of the adhesive [84]. The *in vitro* analysis of teeth restored and loaded in clinical conditions may contribute to a better understanding of failure modes and mechanisms. Thus, SEM analysis of the adhesive interface is an important and frequently used method to investigate tooth structure and bonding mechanisms. This method gives the opportunity for better understanding of the complexity and three-dimensional variations of the tooth structure [85].

In the present study, class II cavities restored with bulk-fill composite that underwent one year of aging in the oral cavity were evaluated in SEM. This study design provides an exceptional opportunity to evaluate the clinical performance of a restoration using an advanced imaging technique. Furthermore, the application of vital teeth with pulp, dentinal fluid and odontoblastic processes provided a scenario that cannot be studied in *in vitro* conditions. This is a great advantage of the present study over laboratory studies that are typically performed on extracted teeth [86–88]. Moreover, the natural saliva of the patient was used as the ageing fluid; therefore, the one-year aging process was performed in a perfect environment. Those conditions are almost impossible to obtain in *in vitro* studies.

The limitations of this study should be taken into consideration. First of all, the low number of teeth acquired from one subject included in the investigation means that the present results cannot be extrapolated to the general population. Furthermore, collecting information from numerous patients with different tested protocols should be researched in future clinical studies. Next, further studies are needed to test more dental adhesives and composite resins to demonstrate comparison between different materials. In the present study, only a one-time interval of interface aging was used; therefore, longer studies at differentiated time spans should be performed. Moreover, gap measurement and chemical analysis of the interface could also be investigated. In addition, the SEM study creates uncertainty about whether the gap is formed before or after light curing, or even during specimen preparation. The gap observed in only one specimen may

be a consequence of specimen preparation, including sectioning procedures and drying steps [89]. Further studies could be performed with an accurate, safe, and non-destructive method such as dye penetration [90] or non-invasive OCT (optical coherence tomography) instead of SEM [91]. Last, some tubule walls could be dissolved during demineralization; thus, tubule-covering sheaths persisted which may otherwise denote the so-called lamina limitans—in other words, the inner sheath of the peritubular dentin matrix containing glucosaminoglycans. The distinction of the tag-like structures from laminae limitantes in demineralized specimens might be the scope of further studies using energy-dispersive X-ray spectroscopy (EDX) [92].

4. Conclusions

Within the limitations of this study, the application of a bulk-fill flowable material covered with a conventional composite together with a two-step etch-and-rinse adhesive seems to provide a homogeneous and stable bond to tooth structure after one year of aging in an oral environment. Despite some minor defects at the dentin–resin composite interfaces, it seems that restoring the cavity surrounded by enamel margin should provide more favorable outcomes.

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Data Availability Statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.

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