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Limited interaction of a self-adhesive flowable composite with dentin/enamel characterized by TEM

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ABSTRACT

Objectives. A new category of composite which does not require any acid etching or bonding protocol prior to application has been introduced. The purpose of this study was to characterize the interfacial ultra-structure at enamel and dentin surfaces by means of transmission electron microscopy (TEM).

Methods. Non-carious human third molars were randomly divided into 6 groups (bur-cut dentin, SiC-ground dentin, fractured dentin, bur-cut enamel, SiC-ground enamel, and un-cut enamel). After preparation of the respective surfaces, the self-adhesive flowable composite (Vertise Flow, Kerr) was applied. Non-demineralized/demineralized and non-stained/stained sections of 70–90 nm were prepared following common TEM-specimen processing procedures. Additional specimens were immersed in an ammoniacal silver nitrate solution.

Results. The composite–dentin interface was free of voids and no de-bonding occurred during specimen preparation. For bur-cut and SiC-ground dentin, no surface demineralization was observed and the smear contained residual hydroxyapatite. On fractured dentin (i.e. without smear interference), a very thin interaction area of 100–200 nm without apparent signs of surface demineralization was seen. When the composite was bonded to enamel, a distinct separation between the self-adhesive composite and enamel was present in all three groups. A tight interaction, yet without distinct dissolution of hydroxyapatite, could only be seen in some regions at bur-cut enamel where smear was absent or sparse. Silver nitrate infiltration was associated with the presence of smear.

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Significance. The self-adhesive composite showed limited interaction with smear-covered substrates and aprismatic enamel, which explains its inferior diminished bonding capacity in comparison with current adhesives.

1. Introduction

Decayed/fractured teeth can be reconstructed minimal-invasively and nearly invisibly using adhesive technology. The first clinical breakthrough came after the introduction of 3-step adhesive systems, which made use of phosphoric-acid etching, primer and bonding. A simplified adhesive protocol in order to reduce clinical time is a highly desired property in adhesive dentistry. Recently, a new category of restoratives, self-adhesive (flowable) composites, has been developed. According to the manufacturers' instructions, these self-adhesive (flowable) composites do not require any acid etching or bonding protocol prior to application. The self-adhesive composite Vertise Flow (Kerr, Orange, CA, USA) comprises phosphoric-acid ester methacrylate and glycerol phosphate dimethacrylate (GPDM) as functional monomers. Rahimian-Imam et al. revealed that this self-adhesive flowable composite exhibited less microleakage than conventional fissure sealants [1]. However, when used as a sealant in a split-mouth clinical trial, retention rates of the self-adhesive flowable composite were significantly lower compared to those of three conventional flowable composites bonded with an adhesive [2]. After 24 months, the retention rate of the self-adhesive composite was only 62.9% [2].

An important part of dental adhesive technology research is focused on bond-strength tests to tooth substrates in the laboratory. With these bond-strength tests, bonding effectiveness of the self-adhesive composite was found to be lower than that of 3-step etch-and-rinse [3–7], 2-step etch-and-rinse [8], 2-step self-etch [4,7], and 1-step self-etch [6,9,10] adhesives. Our research group found a general trend that multi-step adhesives bonded more effectively than simplified one-step adhesives, which on their turn performed better than the self-adhesive flowable composite Vertise Flow in both a micro-tensile bond strength [11–13] and interfacial fracture toughness [12,13] test. However, a detailed affirmation of the cause of this lower bonding effectiveness of the self-adhesive composite is not yet available.

Much knowledge on the underlying mechanisms of adhesion to enamel and dentin has been derived from microscopic imaging adhesive–tooth interfaces. Transmission electron microscopy (TEM) has the great advantage of rendering a high structural resolution with a relatively low artifact incidence [14,15]. Fu et al. analyzed the interface between the self-adhesive composite and 600-grit SiC-paper grounded dentin, and detected bubble formation and empty dentin tubules [16]. To date, this is the only publication available in which TEM was used to evaluate the interfacial ultra-structure of a self-adhesive composite at dentin. No TEM studies regarding the self-adhesive composite–enamel interface have been conducted so far. The purpose of this study was to characterize ultra-morphologically the interface complex of a self-adhesive flowable composite bonded to different by prepared surfaces of enamel and dentin using TEM.

2. Materials and methods

Twelve non-carious human third molars were stored in 0.5% chloramine at 4 °C and used within one month after extraction. The teeth were randomly divided into 6 groups in accordance with the prepared dentin (bur-cut, SiC-ground, fractured) and enamel (bur-cut, SiC-ground, un-cut) surfaces. All teeth were mounted in gypsum blocks in order to ease manipulation.

To prepare the bur-cut dentin specimens, the occlusal third of the crown was removed at the level of mid-coronal dentin using a slow-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), after which a smear layer was produced using a medium-grit 100-μm diamond bur (842, Komet, Lemgo, Germany) in a water-cooled high-speed contra-angle handpiece mounted in the MicroSpecimen Former (University of Iowa, Iowa, IA, USA). SiC-ground dentin specimens were prepared by grinding with wet 600-grit silicone-carbide (SiC) paper to produce a thinner smear layer. For fractured dentin specimens, a shallow 1–2 mm deep groove was cut circumferentially around the tooth at the level of mid-coronal dentin after which the coronal part was fractured using a forceps to render a surface free of smear debris. All dentin surfaces were carefully verified for absence of enamel and/or pulp tissue using a stereo-microscope (Wild M5A, Heerbrugg, Switzerland). The bur-cut and SiC-ground enamel specimens were prepared in analogy with the respective dentin specimens. For the bur-cut enamel specimens, lingual and buccal enamel surfaces were flattened using the MicroSpecimen Former and a medium-grit 100-μm diamond bur (842). SiC-ground enamel specimens were further ground with wet 600-grit SiC paper. From the third set of enamel substrate specimens, the lingual/buccal surface was cleaned with pumice using a soft-bristle brush mounted in a handpiece to produce a clean enamel surface free of debris (un-cut enamel).

A very thin layer (<5 mm) of the self-adhesive flowable composite Vertise Flow was applied to each flat substrate, strictly according to the manufacturer's instructions (Table 1). Specimens were light-cured for 20 s using a halogen curing light (Optilux 500, Kerr) with an output of at least 600 mW/cm^2. Thereafter, specimens were stored for 1 day in distilled water at 37 °C. The specimens were processed for TEM according to the procedure described in detail by Van Meerbeek et al. [17]. Non-demineralized and lab-demineralized ultra-thin sections were cut (Ultracut UCT, Leica, Vienna, Austria) and examined using TEM (JEM-1200EX II, JEOL, Tokyo, Japan), unstained and positively stained (5% uranyl acetate for 12 min/saturated