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Apatite crystal protection against acid-attack beneath resin–dentin interface with four adhesives: TEM and crystallography evidence

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ARTICLE INFO

Article history:
Received 19 December 2011
Received in revised form
8 March 2012
Accepted 16 April 2012

Keywords:
Etch-and-rinse
Self-etch
Functional group
ABRZ
Electron crystallography

ABSTRACT

Objectives. Interaction between specific functional groups and apatite crystals may contribute to adhesion. The present study investigated effects of four adhesives with different compositions on protection of crystals beneath the hybrid layer against acid-attack using transmission electron microscopy (TEM) and selected area electron diffraction (SAED).

Methods. Human dentin was bonded with four adhesives; two with a carboxylic-based functional co-polymer (PAA): three-step etch-and-rinse Scotchbond Multi-Purpose (SMP, 3M ESPE) and one-step self-etch Adper Easy Bond (AEB, 3M ESPE), and two with a phosphate-based functional monomer (MDP): two-step etch-and-rinse Clearfil Photo Bond (CPB, Kuraray Medical) and two-step self-etch Clearfil SE Bond (CSE, Kuraray Medical). The specimens were the either left untreated (control) or subjected to acid–base challenge with demineralizing solution (pH 4.5) and 5% NaClO. All specimens were processed and observed by TEM. SAED was used to identify the presence or absence of apatite crystallites at the base or beneath hybrid layer before and after acid–base challenge.

Results. An apatite-rich zone was observed beneath the partially demineralized hybrid layer of CSE. The zone was thinner in AEB, but a demineralization-susceptible area was found beneath it. The etch-and-rinse adhesives (SMP and CPB) demonstrated completely or predominantly demineralized hybrid layers, which were devoid of the acid-resistant apatite-rich zone.

Significance. TEM/SAED evidence disclosed that the preserved dentin apatite crystals beneath the thin hybrid layer of the mild self-etch adhesives were protected against acid. Diffusion of reactive components beyond the hybrid layer, and their chemical bonding potential with the remaining crystals created the acid–base resistant zone.

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http://dx.doi.org/10.1016/j.dental.2012.04.025

1. Introduction

The ultimate goal in adhesive dentistry is to achieve a durable bond to dental tissues. Bonding to dentin was conventionally achieved by three main steps namely etching, priming and bonding, each applied as a separate agent [1]. Self-etching primer systems combined the etching and the priming steps into one, while the bonding agent remained as a separate agent. More recently, one-step self-etch or all-in-one systems combined the self-etching primer and the bonding agent into one application [1]. Adhesion to dentin substrate has been primarily attributed to micro-mechanical hybridization which involves infiltration and subsequent *in situ* polymerization of adhesive monomers within the demineralized microporous collagen scaffold [2,3]. In addition, it was suggested that chemical interactions between carboxyl or phosphate ester groups of functional monomers in adhesives and apatite contributed to bonding [4,5].

Despite extensive morphological studies on the interface and small-scale destructive mechanical tests, the interactions of adhesive components with the structural units of hard-tissues (*i.e.* biological apatite crystals in dentin) *in situ*, and the properties of the complexes formed to confirm actual intermolecular interactions have been investigated to a limited extent [6]. In addition to ultrastructural observation, application of local analytical techniques on the interface of biomaterial-hard tissue would benefit further progress of the adhesive technology. Among those analytical techniques, selected area electron diffraction (SAED) is a crystallography method that provides information on the local crystalline structure of thin sections under transmission electron microscopy (TEM); however, only few studies to date have employed crystallography on the interface after demineralization challenge [7].

Recent studies revealed an acid–base resistant zone (ABRZ) beneath the visible hybrid layer when dentin was treated with specific functional monomers as part of mild self-etch adhesives [7–9]. Based on the attributes of this zone, which was more resistant against acidic and basic attack compared to the underlying normal dentin, a dentin reinforcement concept was proposed [7]. However, it is unknown whether apatite crystals remaining beneath the hybrid layer were acid-resistant after phosphoric-acid-etching step in the etch-and-rinse approach, with adhesives containing similar functional monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP). In addition, little information is available on the acid-resistance of dentin treated with polyalkenoic acid (PAA)-based adhesives, considering the well-proven primary ionic interaction between the carboxyl groups of the PAA and the calcium (Ca) of apatite [10,11].

Therefore, the aim of the present study was to investigate the protection of crystals beneath the hybrid layer against acid-attack following application of commercially available PAA- or MDP-based etch-and-rinse or a self-etch adhesives. The null hypothesis tested was that the thickness of an apatite containing layer remaining after acid–base challenge was not different using various adhesive approaches containing different functional groups.

2. Materials and methods

2.1. Adhesives and bonding procedures

The chemical compositions and the manufacturers' instructions of the four adhesives used in this study are listed in Table 1. The three-step etch-and-rinse system, Scotchbond Multi-Purpose (SMP; 3M ESPE, St Paul, MN, USA) contains about 10–20 wt% carboxylic-based functional co-polymer of PAA. The two-step etch-and-rinse system, Clearfil Photo Bond (CPB; Kuraray Medical, Tokyo, Japan) contains about 5–10 wt% phosphate-based functional monomer of MDP. The two-step self-etch system, Clearfil SE Bond (CSE; Kuraray Medical) also contains about 25–30 wt% MDP. The one-step self-etch system, Adper Easy Bond (AEB; 3M ESPE) contains about 1–5 wt% PAA, and 5–15 wt% of a phosphoric acid monomer.

The study set-up is illustrated in Fig. 1. Twelve freshly extracted non-carious third molars were used following the guidelines approved by the Tokyo Medical and Dental University Ethical Committee. A 1.5-mm-thick dentin disk was prepared from mid-coronal dentin using a low-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) under water cooling. The disk was covered with acid-resistant nail varnish (Shiseido, Tokyo, Japan) except for a window (2.5 mm × 2.0 mm) in the center of the mid-coronal dentin surface. The discs were divided into two halves at the center of the window; the crosscut surface on each hemisection was wet-sanded with 600-grit SiC papers to create a standardized smear layer. Three pairs of half-disks obtained from three teeth were randomly assigned to each of the adhesives. The adhesives were then applied on the ground dentin surfaces of the teeth that were to be bonded with the etch-and-rinse adhesives were etched for 15 s with 37–40% phosphoric acid or treated with the self-etching adhesive according to the manufacturers' instructions. The adhesive was light-cured using a visible light curing unit (Optilux 501; Demetrom, Danbury, CT, USA) at an output of 600 mW/cm^2 prior to the incremental placement of a hybrid composite (Z100; 3M ESPE).

2.2. Acid–base challenge

After 24-h storage at 37 °C in water, the specimens were either left untouched (control) or subjected to the acid–base challenge as follows. First, 100 ml buffered demineralizing solution (pH 4.5, 2.2 mmol/l $CaCl_2$, 2.2 mmol/l NaH_2PO_4 and 50 mmol/l acetic acid) was used for 90 min to create artificial secondary caries [12]. After the acid-challenge, the specimen was subjected to 5% NaClO for 30 min with ultrasonic vibration, in an attempt to remove any denatured dentin collagen fibrils, and finally rinsed with running water for 30 s [9]. The specimens were subjected to the acidic and basic solutions (25 °C) in a beaker with a magnetic stirrer to ensure complete access of acid to all of the exposed surfaces.

2.3. Transmission electron microscopy

The interface of the adhesive and dentin in the control and acid–base challenged specimens was observed under transmission electron microscope (TEM). To facilitate

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