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Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin

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ABSTRACT

Objectives: To examine the interfacial chemical and morphological characteristics of four self-etching adhesives bonded to dentin with different functional monomers. Further, to evaluate the effects of this interaction between functional monomers and dentin on short-term *in vitro* bonding performance of the four adhesives.

Methods: Clearfil SE Bond (CSE) and Scotchbond Universal (SU) containing 10-methacryloxydecyl dihydrogen phosphate (10-MDP), Optibond XTR (OX) containing glycero-phosphate dimethacrylate (GPDM), and Adper Easy One (AEO) containing 6-methacryloyloxyhexyl dihydrogen phosphate (6-MHP) were applied to the dentin surface according to the instructions supplied with each. Interaction between the functional monomers and dentin was characterized using thin-film X-ray diffraction (TF-XRD) and scanning electron microscopy (SEM). The hydrophilicity of each acidic monomer was also assessed by chemical structure drawing software. Microtensile bond strength (μTBS) and nanoleakage were used to evaluate the bonding effectiveness of the adhesives, either immediately or after thermo-cycling (5 °C–55 °C) for 5000 cycles.

Results: TF-XRD showed that both CSE and SU exhibited 10-MDP–Ca nano-layering at the adhesive interface, but with different intensity when reacted with dentin. OX, that contains GPDM, demineralized the dentin surface more severely, forming long resin tags into the dentinal tubules, and gained the highest μTBS at the immediate time-point. Thermo-cycling adversely affected the μTBS and nanoleakage of AEO and OX, but had no significant influence on CSE and SU which contain 10-MDP.

Conclusions: Self-etching adhesives containing different structures/concentrations of functional monomers produced adhesive interfaces with obviously different chemical and morphological characteristics, which may have a direct impact on bonding effectiveness.

Clinical Significance: Our findings support the concept that the stable chemical bonding produced by 10-MDP to the Ca of hydroxyapatite is advantageous for durability of adhesive–dentin bonds. In contrast a higher immediate bond strength was achieved with the functional monomer GPDM that etched and wetted the dentin surface better.

1. Introduction

The bonding performance of the various commercially-available self-etching adhesives evaluated either by laboratory or clinical studies varies greatly, relying on the actual composition [1,2] or rather the specific functional monomer included in it [3]. With functional monomers, self-etching adhesives can simultaneously demineralize and infiltrate the tooth surface. The chemical and morphological characteristics of the adhesive–tooth interface and the closely-related quality of the hybrid layer depend to a large extent on the interaction between functional monomers and the tooth substrate [4,5].

With different chemical structures, the functional monomers containing acidic groups may interact quite differently with hydroxyapatite (HAp) and thus tooth tissue, and even small changes may influence their polarity and consequently, influence their interaction behavior and bonding efficacy [6,7]. According to the concept of 'Adhesion–Decalcification' (A–D concept) [8] that describes the way acidic molecules interact with HAp, all acids bond ionically to the calcium of HAp in the first phase. The molecule will then remain bonded if the ionic bond is hydrolytically stable, or it will de-bond and calcium will be removed from deep within the tooth surface, leading to a relatively deep demineralization of the dentin if the ionic bond is unstable in the

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second phase.

A functional monomer such as 10-methacryloxydecyl dihydrogen phosphate (10-MDP) can form stable calcium–phosphate complexes, and self-assemble into the form of a regular layered structure at the apatite surface. The good hydrolytic stability of 10-MDP-Ca salts may be attributed to the presence of long and relatively hydrophobic spacer chains of 10-MDP. The unique chemical structure of 10-MDP and the resultant intense and stable adhesion to the calcium in HAp has been shown to contribute particularly to the durability of the bond as well as enhance the initial bonding performance of self-etching adhesives [3,9,10]. Several recently marketed 'universal' adhesives such as Scotchbond Universal (3M ESPE, Seefeld, Germany), All-Bond Universal (Bisco, Schaumburg, IL, USA), and Clearfil 3S Bond Plus (Kuraray Noritake Dental, Tokyo, Japan) have been formulated with 10-MDP. The bonding effectiveness of universal adhesives has therefore been a hot topic since they came into use [11–14].

Certainly, apart from 10-MDP, functional monomers such as glycero-phosphate dimethacrylate (GPDM) also appear to be used in 'universal' adhesives (OptiBond XTR [Kerr, Orange, CA, USA]). However, to our knowledge there are no publications concerning the characterization of the interaction of GPDM with HAp. In fact, current work with regard to the chemical interaction of acid monomer with HAp-based substrates mainly focused on 10-MDP, 4-methacryloxyethyl trimellitic acid (4-MET) and 2-methacryloxyethyl phenyl hydrogen phosphate (Phenyl-P) [9,15,16]. Furthermore, self-etching adhesives are generally mixtures of components including an acid functional monomer, hydrophobic monomers, water, and an organic solvent, and thus functional monomers account for only a small proportion. Very few investigations have specifically adopted commercial adhesives as the object of studies to clarify the actual interaction between functional monomer and dentin and analyzed their practical effects on bonding performance, considering that both the concentration of functional monomer and other components in adhesives may influence the interaction.

To address this issue, in the present study, we chose four commercial self-etching adhesives: the two-step self-etching adhesives Clearfil SE Bond containing 10-MDP, universal adhesives Scotchbond Universal also containing 10-MDP but at a lower concentration and Optibond XTR containing GPDM, and the one-step self-etching adhesive Adper Easy One containing 6-methacryloyloxyhexyl dihydrogen phosphate (6-MHP). The aim of this study was first to characterize the interaction between the functional monomers in the adhesives and the dentin chemically, using X-ray diffraction (XRD), and also ultrastructurally, using scanning electron microscopy (SEM). The effects of this interaction on short-term *in vitro* performance of the adhesives bonded to human dentin were then analyzed, by assessing micro-tensile bond strengths (μTBS) and nanoleakage. The null hypotheses were that : (1) adhesive interfacial characteristics of the four self-etching adhesives with different functional monomers applied to dentin are almost the same; (2) there is no difference in short-term *in vitro* bonding performance of the four adhesives tested.

2. Materials and methods

One hundred and twenty-six caries-free extracted human third molars were collected after obtaining informed consent from donors under a protocol approved by the Ethics Committee for Human Studies of the Shanghai Ninth People's Hospital. The teeth were cleaned, stored in 1% chloramine at 4 °C and used within 1 month following extraction. The compositions, manufacturers and application instructions of the four adhesives examined in the present study are listed in Table 1.

2.1. Thin-film X-ray diffraction (TF-XRD) analysis

Fifteen dentin disks (10 × 8 × 1 mm) were cut using a low-speed diamond saw (ISO Met 4000, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. The prepared disks were polished with wet 600-grit SiC paper for 1 min. All dentin surfaces were carefully verified, by stereomicroscopy, for the absence of enamel/pulp tissue. Each of the four self-etching adhesives (Table 1) were applied to dentin disks by lightly rubbing each dentin surface using a microbrush according to the instructions supplied with each (n = 3, referred to as 'CSE_dentin', 'SU_dentin', 'OX_dentin' and 'AEO_dentin'). The samples were then vigorously air-dried before being analyzed by thin-film XRD (Bruker AXS D8, Germany) operated at 40 kV acceleration and 200 mA current, with the angle of the incident X-ray beam fixed at 2.0° and a scanning time of 0.02°/s for 2θscan. Untreated dentin ('dentin') specimens were examined by XRD as reference.

2.2. Morphological assessment under SEM

The morphological assessment consisted of two parts: the first part consisted of observation of the surfaces of adhesive-treated dentin, with and without rinsing with ethanol or distilled water following a protocol similar to that described by Yoshihara et al. [6]. In the second part, vertical sections of the adhesive interface were further prepared to assess the extent of adhesive infiltration into dentin.

2.2.1. Part 1

Further 600-grit SiC-paper-ground thirty-nine dentin specimens were prepared. Thirty-six of the disks were treated with each of the four self-etching adhesives whilst the other three dentin disks were used as untreated controls (n = 9). The "self-etching adhesives + dentin specimens" and "self-etching adhesives + dentin ethanol/water rinsed specimens" (three specimens per subgroup) were mounted on aluminum stubs, dehydrated in silica gel, gold sputter-coated, and imaged by SEM (S3400, Hitachi, Tokyo, Japan) at 15 kV.

2.2.2. Part 2

Twelve teeth were selected for this part. The enamel and superficial dentin of these teeth were removed to expose the mid-coronal dentin. The exposed dentin surfaces were then polished using 600-grit SiC paper for 60 s under running water to create standardized surfaces. The adhesives were applied to the dentin by lightly rubbing each dentin surface using a microbrush following the respective manufacturers' instructions, then air-dried and light-cured for 10 s under an LED light-curing unit (Elipar Trilight, 3 M ESPE) with an output intensity of 750 mW/cm^2. Composite resin crowns were built with a nanofilled composite resin (Filtek Z350 XT, 3 M ESPE, St. Paul, MN, USA) in two increments of 2 mm each. Each increment was light-cured for 40 s under the LED light-curing unit mentioned above. After storing in distilled water for 24 h at 37 °C, all resin–dentin specimens were longitudinally sectioned perpendicular to the bonded interface with a low-speed diamond saw (ISO Met 4000) under sustained water-cooling to obtain resin–dentin slabs (the central slabs, three specimens per group) with a thickness of 1 mm.

The surfaces of the slabs were treated with 37% phosphoric acid for 30 s to completely eliminate any smear layer created by cutting. Surfaces were then treated with 5% sodium hypochlorite for 5 min to provide evidence of infiltration of the adhesive into dentin. Finally, prepared slabs were sputter-coated with gold and examined under SEM at an accelerating voltage of 15 kV.

2.3. Hydrophilicity assessment of acidic functional monomer

The number of carbon atoms and/or ester/polyether groups in spacer chains may influence the hydrophilicity of the functional monomers and also their interaction with calcium and dentin [7]. As displayed in Fig. 1, the chemical structures of the three phosphate functional monomers evaluated in the present investigation were different from each other considering both of number of carbon atoms and presence of hydrophilicity group in spacer chains. The hydrophi-

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