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Smear layer-deproteinizing improves bonding of one-step self-etch adhesives to dentin

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ABSTRACT

Objectives. Smear layer deproteinizing was proved to reduce the organic phase of smear layer covered on dentin surface. It was shown to eliminate hybridized smear layer and nanoleakage expression in resin–dentin bonding interface of two-step self-etch adhesive. This study aimed to investigate those effects on various one-step self-etch adhesives.
Methods. Four different one-step self-etch adhesives were used in this study; SE One (SE), Scotchbond™ Universal (SU), BeautiBond Multi (BB), and Bond Force (BF). Flat human dentin surfaces with standardized smear layer were prepared. Smear layer deproteinizing was carried out by the application of 50 ppm hypochlorous acid (HOCl) on dentin surface for 15 s followed by Accel® (*p*-toluenesulfinic acid salt) for 5 s prior to adhesive application. No surface pretreatment was used as control. Microtensile bond strength (μTBS) and nanoleakage under TEM observation were investigated. The data were analyzed by two-way ANOVA and Tukey's post-hoc test and *t*-test at the significant level of 0.05.
Results. Smear layer deproteinizing significantly improved μTBS of SE, SU, and BB ($p < 0.001$). Hybridized smear layer observed in control groups of SE, BB, and BF, and reticular nanoleakage presented throughout the hybridized complex in control groups of BB and BF were eliminated upon the smear layer deproteinizing.
Significance. Smear layer deproteinizing by HOCl and Accel® application could enhance the quality of dentin for bonding to one-step self-etch adhesives, resulting in the improving

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μTBS, eliminating hybridized smear layer and preventing reticular nanoleakage formation in resin–dentin bonding interface.

1. Introduction

Recently, one-step self-etch adhesives with quicker application times and easier handling are being increasingly used in the clinic. Their bond strengths [1,2] and polymerization behavior [3] have improved over time. They are known to form hybridized smear layer on the authentic hybrid layer at the adhesive interface, because they cannot completely remove the smear layer due to their mild acidity [4]. Remnants of the smear layer on the adhesive surface have been purported to adversely affect the dentin bonding performance of self-etching adhesives, because they act as a selective barrier for monomer infiltration [5], giving rise to a physical weak link in the interface [6]. Moreover, their porous characteristics incorporate a certain amount of water [7], which lowers the degree of resin monomer conversion [8,9] and forms nanoleakage in the adhesive layer [10,11].

The dentin smear layer is composed of disorganized organic debris binding mineral particles [12]. Generally, self-etching adhesives can dissolve and remove the mineral phase in the smear layer, but they leave organic debris on the dentin surface, which is not dissolved [13]. Some researchers have demonstrated that treatment with an oxidizing/deproteinizing agent, such as sodium hypochlorite (NaOCl) and hypochlorous acid (HOCl) solutions, can dissolve and remove the organic phase of smear layer, leading to an increased mineral to organic ratio at the smear layer-covered dentin surface [14,15] and thinning of the smear layer [16,17]. Smear layer deproteinizing with HOCl solution, using in combination with a two-step self-etch adhesive (Clearfil SE Bond), can eliminate the hybridized smear layer and prevent nanoleakage formation at the resin–dentin interface [15]. These results indicated that removal of the organic phase of the smear layer would promote further infiltration of resin monomer into the underlying dentin without formation of hybridized smear layer [15]. Additionally, increasing the mineral/organic ratio on dentin surface by smear layer deproteinizing might be advantageous for chemical interaction of acidic functional monomers with hydroxyapatite [18–20]. Regarding NaOCl solution, several researchers have demonstrated that the residual oxidizing effect on NaOCl-treated dentin would affect resin polymerization, leading to a reduction in bond strengths [17,21] and an increase in nanoleakage expression [18,22]. However, these negative effects of NaOCl pretreatment on dentin bonding can be reversed by the subsequent application of reducing/antioxidant agents [21,23]. On the other hand, single pretreatment with HOCl solution had shown to significantly improve bond strengths of self-etching adhesive to caries-affected dentin [16]. Although there were no improvements in bond strengths to normal dentin [24], the quality of hybrid layer was improved as the hybridized smear layer and reticular nanoleakage were eliminated [15]. However, the effects of smear layer deproteinizing with oxidizing agents on bond strengths and nanoleakage expression at the adhesive interface might be dependent upon the type of self-etching adhesive, because of the differences in acidic functional monomers, hydrophilic and hydrophobic monomer compositions, polymerization catalyst, organic solvent etc. There is little information on the effect of smear layer deproteinizing on the dentin interface bonded to one-step self-etch adhesives. Therefore, the aim of this study was to evaluate the effect of smear layer deproteinizing by pretreatment with HOCl solution on dentin bond strengths and nanoleakage expression at the interface using one-step self-etch adhesives.

The null hypothesis was that there was no difference neither in microtensile bond strength nor nanoleakage expression at the adhesive interface of smear layer-deproteinized dentin and no-pretreated smear layer-covered dentin bonded to each one-step self-etch adhesive.

2. Materials and methods

2.1. *Specimen preparation*

Following ethical approval by the Ethics Committee of Tokyo Medical and Dental University under protocol number 725, extracted human third molars were collected, and stored in distilled water containing 0.1% thymol solution at 4 °C within a six-month period prior to the experiments. Fifty-six flat dentin surfaces were ground using a model trimmer perpendicular to long axis of the tooth under water lubrication, and then wet-polished using 600-grit SiC paper for 30 s to create a standardized smear layer. Half of the specimens was subjected to smear layer deproteinizing procedure by treating the smear layer-covered dentin surface with 50 ppm HOCl (Comfosy®, Haccpper Advantec Co., Tokyo, Japan) solution for 15 s, and then rinsed with water for 30 s. After air-drying, a reducing agent (*p*-toluenesulfinic acid salt; Accel®, Sun Medical Co. Ltd., Kyoto, Japan) was applied to the HOCl-treated dentin surface for 5 s and air-dried. The remaining half of the specimens was used as controls (without smear layer deproteinizing). The specimens in smear layer deproteinizing and control groups were randomly divided into 4 subgroups (n = 7). One of four kinds of one-step self-etch adhesives; Clearfil™ Bond SE One (SE; Kuraray Noritake Dental Inc., Japan), Scotchbond™ Universal (SU; 3 M ESPE, USA), BeautiBond Multi (BB; Shofu, Japan), and Bond Force (BF; Tokuyama Dental, Japan) was applied to dentin surface according to manufacturers' instructions (Table 1). Subsequently, three increments of resin composite (Clearfil AP-X; Kuraray Noritake Dental Inc., Japan) were built up on the dentin surface with each increment being light cured (830 mW/cm^2; Optilux 501, Kerr, Orange, CA, USA) for 20 s. The specimens were stored in 37 °C water for 24 h.

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