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## Effect of double-layer application on bond quality of adhesive systems



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### ABSTRACT

**Objectives:** The aim of this study was to determine the effect of double-layer application of universal adhesives on the bond quality and compare to other adhesive systems.

**Methods:** Two universal adhesives used were in this study: Scotchbond Universal (SU), [3M ESPE] and Prime & Bond elect (PE), [Dentsply Caulk]. The conventional single-step self-etch adhesives G-aenial Bond (GB), [GC Corporation.] and BeautiBond (BB), [Shofu Inc.], and a two-step self-etch adhesive, Optibond XTR (OX), [Kerr Corporation], were used as comparison adhesives. Shear bond strengths (SBS) and shear fatigue strengths (SFS) to human enamel and dentin were measured in single application mode and double application mode. For each test condition, 15 specimens were prepared for SBS testing and 30 specimens for SFS testing.

**Results:** Enamel and dentin SBS of the universal adhesives in the double application mode were significantly higher than those of the single application mode. In addition, the universal adhesives in the double application mode had significantly higher dentin SFS values than those of the single application mode. The two-step self-etch adhesive OX tended to have lower bond strengths in the double application mode, regardless of the test method or adherent substrate.

**Conclusion:** The double application mode is effective in enhancing SBS and SFS of universal adhesives, but not conventional two-step self-etch adhesives.

**Significance:** These results suggest that, although the double application mode may enhance the bonding quality of a universal adhesive, it may be counter-productive for two-step self-etch adhesives in clinical use.

### 1. Introduction

After decades of research and development of resin composite materials and dental adhesive, tooth colored direct resin composite restorations have increased in popularity and are extensively accepted due to achieving minimally invasive treatment. However, the bonded restorations are threatened by many internal and external degradation factors in intraoral conditions. In particular, hydrolytic degradation and fatigue of the adhesive, biofilm attack, and enzymatic degradation by dentin-MMPs (matrix metalloproteinases) are important reasons for the failure of bonded restorations (Breschi et al., 2008; Carvalho et al., 2012; Nassar et al., 2014; Tezvergil-Mutluy et al., 2015). Adhesive technology is a key factor in achieving long term stability in the oral environment.

At present, dental adhesives are classified as either “etch-and-rinse” or “self-etch” systems. Furthermore, self-etch systems have been classified as “single-step” or “two-step” adhesives based on the application procedure (Van Meerbeek et al., 2011). When comparing single and two-step self-etch adhesive systems, although neither type of adhesive system requires a water rinsing step, the composition, application steps, etching capability, and thickness of the adhesive layer are different from each other (Van Meerbeek et al., 2011). In particular, single-step self-etch adhesives combine priming and bonding functionality into a single solution. Although, simplified application results in a valuable reduction in treatment time, there are some concerns about not only lower etching ability but also the vulnerability of the cured adhesive layer due to its hydrophilic nature (Erickson et al., 2009a, 2009b; Marchesi et al., 2013; Takamizawa et al., 2015a; Tay et al., 2002).

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Single-step self-etch adhesives tend to have increased residual water in the adhesive layer during polymerization, which may reduce the degree of conversion, resulting in poorer mechanical properties of the adhesive layer, when compared to two-step self-etch adhesives (Takahashi et al., 2011; Van Landuyt et al., 2006; Yamada et al., 2004). In addition, this hydrophilic adhesive layer may act as a semipermeable membrane that allows the movement of water (Tay et al., 2002, 2004). These characteristics of the adhesive layer may enhance its vulnerability to biodegradation and biomechanical forces when compared to the hydrophobic adhesive layer of the two-step self-etch adhesives (Takamizawa et al., 2015b).

Investigators have looked at ways to overcome the shortcomings of single-step self-etch adhesives. Approaches to reinforce or improve the vulnerable adhesive layer of single-step self-etch adhesives have included prolonging air-drying time (Chiba et al., 2006), warm air-drying (Ogura et al., 2012; Shiratsuchi et al., 2013) after adhesive application, multiple-coating of the adhesive layer (Hashimoto et al., 2004; Takahashi et al., 2010; Taschner et al., 2014; Wei et al., 2009), or adding a hydrophobic bonding agent (Muñoz et al., 2014; Perdigão et al., 2014; Reis et al., 2008; Sezinando et al., 2015) have been evaluated in efforts to reinforce or improve the vulnerable adhesive layer of single-step self-etch adhesives. Many of these attempts have shown increased initial bond strength from the modification of the adhesive layer. However, there have been few reports that focus on the bond durability of the improved adhesive layer of single-step self-etch adhesives.

Recently, a new category of self-etch adhesive has been introduced as “universal adhesive”, which has an ability to bond to different types of dental materials without pre-treatment of the adherent surface (Amaral et al., 2014; Seabra et al., 2014; da Rosa et al., 2015). Furthermore, universal adhesives can be used in total-etch mode with phosphoric acid without a negative effect on dentin bonding performance (Takamizawa et al., 2016a, 2016b). Although universal adhesives have been accepted as a new generation of adhesive, some skeptics argue that the bond quality of universal adhesives is similar to conventional single-step self-etch adhesives and they do not represent progress (Chen et al., 2015; Zhang et al., 2016). In addition, it is thought that the adhesive layer of universal adhesives may be as vulnerable as other self-etch adhesives.

The purpose of this study was to determine the effect on bond quality of a double-layer application of universal adhesives compared to conventional single-step or two-step self-etch adhesives. The null hypotheses to be tested were: (1) a double-layer application of a universal adhesive will not affect bond durability; (2) efficacy of double-layer application will not differ between the different kinds of self-etch adhesive system; and (3) the influence of different application modes will not differ between the enamel and dentin substrates.

## 2. Materials and methods

### 2.1. Study materials

The materials used in this study are shown in Table 1. The two universal adhesives used were: Scotchbond Universal (SU), [3M ESPE, St. Paul, MN, USA], and Prime & Bond elect (PE), [DENTSPLY Caulk, Milford, DE, USA]. Two conventional single-step self-etch adhesives were also used: G-aenial Bond (GB), [GC Corporation., Tokyo, Japan], and BeautiBond (BB), Shofu Inc., Kyoto, Japan]. In addition, a conventional two-step self-etch adhesive was also used in this investigation: OptiBond XTR (OX), [Kerr Corporation, Orange, CA USA]. Z100 Restorative [3M ESPE] resin composite was used for bonding to both enamel and dentin. During the course of the study, a visible-light curing unit (Spectrum 800 Curing Unit, [DENTSPLY Caulk]) was used and set at a light irradiance of 600 mW/cm<sup>2</sup>.

### 2.2. Specimen preparation

De-identified extracted human molars were used in this investigation under a protocol reviewed and approved by the Ethics Committee for Human Studies of the Nihon University School of Dentistry (#2015-05). Human molars of regular size and shape were carefully selected and teeth with any signs of cracking of the enamel or caries were excluded to assure homogeneity. Acceptable teeth from all sources were mixed and selected randomly for each specimen group. The enamel and dentin bonding sites were prepared by sectioning the teeth mesio-distally and then removing approximately two-thirds of the apical root structure. Each tooth segment was then mounted in aluminum rings with a diameter of 25 mm with Triad DualLine (DENTSPLY International, York, PA, USA). The enamel and dentin bonding surfaces were ground flat using a water coolant and a sequence of carbide polishing papers (Struers Inc., Cleveland, OH USA) to create a flat-ground surface ending at 4000 grit. In the present study, in an effort to determine the influence of double-layer application, the adherent surface condition was standardised by grinding the flat enamel surface to 4000 grit to eliminate the influences of scratches and the smear layer thickness. Metal rings machined from 304 stainless steel with an inner diameter of 2.4 mm, an outer diameter of 4.8 mm and a length of 2.6 mm were used as a mold-enclosed bonding system (Erickson et al., 2009a, 2009b) for placement of a resin composite on enamel and dentin surfaces for shear bond strength (SBS) and shear fatigue strength (SFS) tests. Following the treatment of the flat ground enamel or dentin surfaces with the adhesive agents, the metal ring was positioned over the bonding sites and secured in place by clamping in a modified Ultradent Bonding Jig [Ultradent Products Inc., South Jordan, UT, USA]. Z100 resin composite was placed into the ring with a condensing instrument and polymerized for 40 s with the curing unit. The bonded specimens were stored at 37 °C in distilled water for 24 h before testing. The bonding procedure resulted in a resin composite cylinder inside the ring that approximated 2.36 mm in diameter and 2.5 mm in height. The ring was left in place for the both the SBS and SFS tests (Takamizawa et al., 2016c).

### 2.3. Adhesive application protocol

The adhesive application protocols are shown in Table 2. For the single application mode, the adhesive agents were used in accordance with the manufacturers' instructions. For the double application mode, the adhesive was applied twice, each time according to the manufacturers' instructions. For the two-step OX, the second application was performed without priming, and the bonding agent was applied followed light irradiation.

### 2.4. Shear bond strength tests (SBS)

For each test group, fifteen (15) specimens were used to determine SBS to enamel and dentin in self-etch mode (without phosphoric acid pre-etching). Specimen size of 15 for SBS testing was used following the recommendation of ISO Standard 29002:2013(E). In addition, following data collection, the sample size was confirmed using two statistical software systems (G Power calculator; <http://www.gpower.hhu.de/>, and Sigma Plot version 13.0; Systat Software, Inc., Chicago, IL, USA). These tests indicated that the sample size was adequate.

The specimens were loaded to failure at a crosshead speed of 1.0 mm/min using a universal testing machine (ElectroPuls E1000, Instron Worldwide Headquarters, Norwood, MA, USA). A metal rod with a chisel-shaped end was used to apply the load to the metal ring immediately adjacent to the flat ground tooth surface. The SBS values (MPa) were calculated from the peak load at failure divided by the bonded surface area. After testing, the bonding sites of the tooth surface and the resin composite cylinders were observed under an optical microscope (MZ16, Leica Microsystems Ltd., Heerbrugg, Switzerland) at a

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