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## Influence of different etching modes on bond strength and fatigue strength to dentin using universal adhesive systems

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### ABSTRACT

**Objectives.** The purpose of this study was to determine the dentin bonding ability of three new universal adhesive systems under different etching modes using fatigue testing.

**Method.** Prime & Bond elect [PE] (DENTSPLY Caulk), Scotchbond Universal [SU] (3M ESPE), and All Bond Universal [AU] (Bisco) were used in this study. A conventional single-step self-etch adhesive, Clearfil Bond SE ONE [CS] (Kuraray Noritake Dental) was also included as a control. Shear bond strengths (SBS) and shear fatigue strength (SFS) to human dentin were obtained in the total-etch mode and self-etch modes. For each test condition, 15 specimens were prepared for the SBS and 30 specimens for SFS. SEM was used to examine representative de-bonded specimens, treated dentin surfaces and the resin/dentin interface for each test condition.

**Results.** Among the universal adhesives, PE in total-etch mode showed significantly higher SBS and SFS values than in self-etch mode. SU and AU did not show any significant difference in SBS and SFS between the total-etch mode and self-etch mode. However, the single-step self-etch adhesive CS showed significantly lower SBS and SFS values in the etch-and-rinse mode when compared to the self-etch mode. Examining the ratio of SFS/SBS, for PE and AU, the etch-and-rinse mode groups showed higher ratios than the self-etch mode groups.

**Significance.** The influence of different etching modes on dentin bond quality of universal adhesives was dependent on the adhesive material. However, for the universal adhesives, using the total-etch mode did not have a negative impact on dentin bond quality.

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## 1. Introduction

Because of increased patient desire for tooth color restorations and minimally invasive treatments, direct application of resin composites has become universally accepted and widespread over the past few decades. This treatment has relied heavily upon the development of adhesive technology, and both resin composite and adhesive technology have rapidly advanced over the years.

Currently, dental adhesives are generally classified into either “etch-and-rinse” or “self-etch” systems. Furthermore, the priming and bonding components can be separated or combined, resulting in three steps or two steps for etch and rinse systems, and two steps or one step for self-etch adhesives [1]. In recent years, self-etch adhesive system have become very popular due to the efficiency offered by the simplified bonding procedures. In addition, it is thought that the incidence of post-operative sensitivity appears to be lower relative to etch-and-rinse systems thanks to chemical bonding and reduced demineralization of dentin [2]. However, some laboratory studies have indicated that self-etch adhesive systems are not able to etch enamel as effectively as the phosphoric acids used in etch-and-rinse adhesive systems due to their lower acidity [3–9]. In order to achieve a durable bond to enamel, when using self-etch adhesive systems, selective etching with phosphoric acid prior to application of the self-etch adhesive has been recommended [10–15]. However, clinically, it may be difficult to precisely etch only the enamel region without affecting exposed dentin [16]. Therefore, inadvertent pre-etching of dentin could be a clinical risk, as resin monomers of self-etch adhesives may not be able to penetrate the entire depth of the deeply demineralized dentin, resulting in reduced dentin bonding quality [11,17–19].

Recently, a new type of single-step self-etch adhesive has been introduced. This type of self-etch adhesive is categorized as “universal” or “multi-mode” as they can be used either with the etch-and-rinse or the self-etch approaches [20–23]. Therefore, universal systems allow application of the adhesive with phosphoric acid pre-etching in the total-etch or selective-etch approaches, which purportedly enhances enamel bond durability. In addition, it also provides a simplified procedure of the self-etch approach on dentin.

Investigation of initial bonding effectiveness is considered essential to grasp the general characteristics of adhesive systems for screening purposes. Currently, the most widespread method for the evaluation of bonding performance is measuring bond strength by shear bond strength (SBS) or tensile bond strength testing ( $\mu$ -TBS). However, clinical bonds between restorations and teeth are not typically subjected to a monotonically increasing force until the bond fails from tensile or shear forces, but rather bonded restorations are subjected to repeated sub-critical loading during normal function. The repeated loads typically encountered in the oral cavity are insufficient to provoke acute failure, but they induce damage by generating cracks that grow over time and eventually result in deterioration of adhesively bonded restorations through marginal failure or, in extreme cases, bulk fracture [24–28]. Fatigue can be defined as the degradation or failure of mechanical properties after repeated applications of stress, at a level

well below the ultimate fracture strength of the material or interface [29]. Consequently, fatigue tests provide not only information on the ability of a material or interface to resist the development of cracks, but also the endurance characteristics of a bonding system.

Although there are several studies of enamel and dentin bonding performance of universal adhesives [20–23], only limited information is available on the bonding quality of universal adhesives when used in different application modes [30]. The purpose of this laboratory investigation was to determine the dentin bond quality of universal adhesives in different application modes using fatigue testing.

## 2. Methods

### 2.1. Study materials

The materials used in this study are shown in Table 1. The three universal adhesives used were: (1) Prime & Bond elect [PE] (DENTSPLY Caulk, Milford, DE USA), (2) Scotchbond Universal [SU] (3M ESPE, St Paul, MN USA) and (3) All Bond Universal [AU] (Bisco, Schaumburg, IL USA). A conventional single-step self-etch adhesive, (4) Clearfil Bond SE ONE [CS] (Kuraray Noritake Dental, Tokyo, Japan) was used as a control. The phosphoric acid pre-etching agent used was Ultra-Etch (Ultradent, South Jordan, UT USA). Z100 Restorative [Z100] (3M ESPE, St Paul, MN USA) was used as a restorative material for bonding to dentin.

### 2.2. Specimen preparation

Extracted caries-free deidentified human molars were selected for use in this study under a protocol reviewed and approved by the Ethics Committee for Human Studies of the Nihon University School of Dentistry (#2015-06). The dentin bonding sites were prepared by sectioning the teeth medio-distally and then removing approximately two-thirds of the apical root structure. The buccal and lingual tooth sections were mounted with Triad Dualine (DENTSPLY International, York, PA, USA) in 25 mm diameter brass rings. The dentin bonding surfaces were ground flat using a water coolant and a sequence of carbide polishing papers ending with 4000 grit (Struers Inc., Cleveland, OH, USA). 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 to confine resin composite on dentin surfaces for shear bond strength (SBS) and shear fatigue strength (SFS) tests. The bonding procedure resulted in a resin composite cylinder inside the ring that approximated 2.35 mm in diameter and 2.5 mm in height. The ring was left in place for the tests.

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

Fifteen specimens were used for each test group to determine the SBS to dentin in total-etch mode (phosphoric acid was applied for 15 s, prior to the application of the adhesive) or in self-etch mode (without phosphoric acid etching). The adhesive agents were used in accordance with the manufacturers' instructions as shown in Table 2. Following the treatment of

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