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Influence of crosshead speed on micro-tensile bond strength of two-step adhesive systems

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Summary *Objectives.* The purpose of this study was to determine the influence of crosshead speed on the micro-tensile bond strength of two separate adhesive systems to dentin.
Methods. The systems used were the Clearfil SE Bond (Kuraray Medical) and the Single Bond (3M ESPE) combined with a resin composite Clearfil AP-X (Kuraray Medical). Dentin surfaces of bovine madibular incisors were primed with self-etching primer followed by air blowing for Clearfil SE Bond, or etched with phosphoric acid followed by rinsing with distilled water for Single Bond, and adhesive was applied. The resin composite was then built up in three layers and light activated. After 24 h storage in water, specimens were sectioned and trimmed to a cross-sectional area of 1 mm^2 and subjected to a micro-tensile bond-strength test. Ten samples per test group were tested at crosshead speeds of 0.5, 1.0, 5.0 and 10.0 mm/min. Micro-tensile bond-strength values (in MPa) were calculated from the peak load at failure divided by the specimen surface area. Two-way ANOVA was performed at the 0.05 probability level.
Results. The mean dentin bond strength at different crosshead speeds ranged from 34.6 to 37.1 MPa for Clearfil SE Bond and from 44.3 to 50.4 MPa for Single Bond. There was no significant difference among the same adhesive systems with the different crosshead speeds tested.
Significance. The influence of the crosshead speed might be negligible when measuring micro-tensile bond strengths.

Introduction

Much of the research related to dentin bonding attempts to assess the integrity and strength of

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the interfacial bond [1-3]. Experimental approaches in dentistry that measure adhesive bond strengths consist primarily of tensile or shear bond-strength determinations performed within a defined area [4-7]. Although the testing procedures used are similar, the results presented in different studies can differ tremendously. It is well known that the coefficient of variation associated with such bond strength figures is high—commonly greater than 30%—with the difference between the extremes being marginally statistically significant [8]. Large variations in bond-strength determinations and the lack of standardized laboratory test procedures have contributed to ambiguities in the interpretation of data [9,10].

The design of the bond-strength apparatus is such that the maximum stress is transmitted along the adhesive interface, and any fracture that appears under the load will pass through the weakest area of the bonding agent, resin composite and dentin substrate [11]. It is generally accepted that the fracture strength of a brittle material is statistical in nature, depending on the probability that a flaw capable of initiating a fracture at a specific applied stress is present [12]. The stress applied at the point of failure between the resin composite and the dentin is recorded as the load at the time of failure divided by the surface area of the bonding interface; this is referred to as the nominal bond strength. A previous study employing finite-element stress analysis to measure the sensitivity of bond strengths in different testing conditions, reported that the nominal bond strength changed with regard to specimen geometry, loading configurations or the modulus of elasticity [13]. Because of the non-uniformity of the interfacial stress distribution, stresses are sensitive to the specificities of the loading geometry, and the shape and size of the resin composites.

A lower rate of load application is often employed with brittle materials compared to elastic materials. Although the dentin and resin composites are brittle materials, the rate of load application (crosshead speed) used to evaluate dentin bond strengths can vary significantly. According to a review by Oshida and Miyazaki [14], crosshead speeds varied widely ranging from 0.1 to 10.0 mm/min, and crosshead speeds of 0.5, 1.0, 2.0 and 5.0 mm/min were commonly employed to evaluate dentin bond strengths for both tensile and shear modes, although none of the reports mentioned their rationale for selecting these crosshead speeds.

The bond-strength test also incorporates the modulus of the adhesive area consisting of a bonding agent, a resin–dentin interdiffusion zone and dentin [15]. Decreasing modulus of elasticity will result in a more even stress distribution over the adhesive area, so as to become less concentrated at the point of load application. The rate of load application might be another influential factor that affects the results of bond-strength tests. It has been reported that different crosshead speeds could influence the dentin bond strength [14,16]. Moreover, relatively high crosshead speeds might cause abnormal stress distributions during the bond-strength test, which would influence the bond-strength value [17,18].

An increasing number of studies has used micro-tensile measurement to evaluate the dentin bond strength of adhesive systems. The number of defects within a specimen composed of a homogeneous brittle material affects the tensile-strength characteristics. When the specimen is loaded, the stress is concentrated at the defected areas, which initiate crack formations. The small adhesive interface used in the micro-tensile test contains fewer defects compared with larger interfaces, resulting in higher recorded bond strengths compared with other test methods, which use larger surface areas [19,20]. The bonding interface is a complex structure composed of the hybrid layer, resin tags, adhesive resin and dentin. As with any measurement method, one should question its accuracy and precision. Research should aim to verify how closely and consistently micro-tensile measurements represent the true interfacial bond strength. The influence of the cross-sectional shape and surface area on the micro-tensile bond test has previously been discussed [21-24]. Another essential issue is the crosshead speed at which the specimen is designed to fail. Although many studies that perform micro-tensile bond-strength tests use varying crosshead speeds, it is not fully understood how this factor affects the variations in the bond-strength value.

The purpose of this study was to determine the effect of the crosshead speed on the micro-tensile bond strength of two separate adhesive systems to dentin using a standard shear bond-strength test apparatus. The null hypothesis was that the crosshead speed affected the values of the micro-tensile bond strength.

Materials and methods

The dentin-bonding systems used in this study were the Clearfil SE Bond (Kuraray Medical, Tokyo, Japan) and Single Bond (3M ESPE, St Paul, MN, USA) as shown in Table 1. An Optilux 501 curing unit