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## The effect of the loading method and cross-head speed on resin–dentin microshear bond strength



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## ABSTRACT

This study compared the effect of different loading methods and crosshead speeds on resin–dentin microshear bond strength ( $\mu$ SBS) using two etchandrinse adhesive systems. Sixty molar teeth had their dentin surfaces exposed and were randomly distributed into 12 groups ( $n=5$ ), according to a combination of the factors: loading methods (orthodontic-looped wire and chisel systems), cross-head speed (0.5, 1.0 and 5.0 mm/min) and adhesive system (Adper Single Bond 2 and XP Bond). Five tygon tubes were positioned over each sample, filled with composite resin and photoactivated. After 24 h, they were tested. The data were analyzed with a two-way ANOVA and Tukey test ( $\alpha=0.05$ ). The  $\mu$ SBS of the adhesives was higher with the chisel methods, compared to the orthodontic-looped wire ( $p < 0.05$ ). The cross-head speed was only significant for the chisel ( $p < 0.05$ ). The evaluated test variables affect the  $\mu$ SBS for both adhesives and therefore should be standardized; however the loading method proved to have the most effect on  $\mu$ SBS values.

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### 1. Introduction

Bonding tests have been extensively employed by different authors to investigate the performance of adhesive systems and techniques [1,2]. The rationale is that the stronger the adhesion between the tooth and a biomaterial, the better it will resist the stress imposed by resin polymerization and oral function.

The shear bond strength test was the most commonly used [1,2] among the available bond strength tests. Improvements in dentin bonding, however, have made this test inappropriate for screening materials and techniques [3]. Cohesive failures in the substrates have been more frequently observed with the newest adhesives [1,3–6]. By using this conventional bonding test, researchers rapidly approached the point where product improvements were not distinguishable as dentin cohesive failures, rather than interfacial failures [1,3–6].

This limitation of the shear test led to the development of the microtensile test [7,8], which relies on the application of tensile stresses to areas with reduced bonds. The smaller the area, the

higher the bond strength value and the higher the sensitivity of the test in detecting subtle differences among groups [8,9]. This new test was claimed to have a better stress distribution, while preventing cohesive failures within the substrates [5,7,8].

Based on the same rationale, some authors have advocated a new test method using specimens with reduced dimensions, as a substitute for the conventional shear test: the so-called microshear test [4,10]. This permits regional mapping or depth profiling of different substrates. In addition, multiple specimens from the same tooth can be prepared, as in the microtensile test, but without the need for sectioning procedures that might, by themselves, induce early cracking [11], mainly when bonded to brittle substrates.

Despite all the advantages of microshear [4], there is a lack of methodological standardization among the different studies. For this reason, analyses of the same adhesive system unavoidably produce different data on the bonding resistance [12]. Although the need for standardization has been widely recognized [13], few efforts have been made towards this end. Two common sources of variation are loading methods and crosshead speed used for testing [14,15]. Authors have employed crosshead speeds of 0.5 mm/min [4,16–18] and 1.0 mm/min [19–23]. This small variation was shown to significantly affect conventional shear testing [14]; however, whether this variable has any impact on microshear

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testing has yet to be addressed. Another source of variation, which also affects the conventional shear test, is the methods used for load application [15]. Although the ISO (International Organization for Standardization) [24] recommends the use of a chisel, which is followed by some researchers [4,22], several other authors have employed orthodontic-looped wire to stress the bonding interface [10,18,21,25,26].

Therefore, the aim of this study is to compare the effects of different loading methods (chisel (ISO standard [24]) and orthodontic-looped wire) and different crosshead speeds (0.5, 1.0, and 5.0 mm/min) on resin–dentin microshear bond strength ( $\mu$ SBS) using two etch-and-rinse adhesive systems. The null hypothesis is that there will be no difference among the various parameters tested.

## 2. Materials and methods

This study used 60 extracted, caries-free third human molars. The teeth were obtained after obtaining the patients' informed consent. The local Ethical Research Committee of University reviewed and approved this study under Protocol number 07733/08.

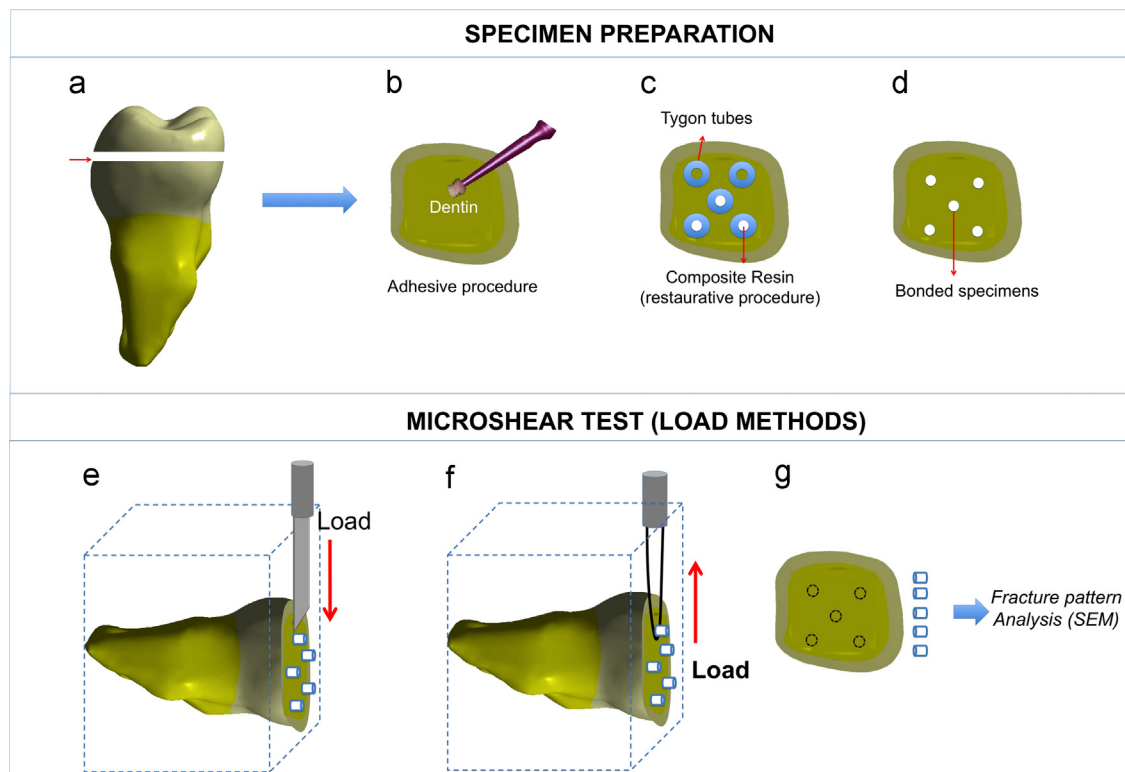
The teeth were extracted and disinfected in 0.5% chloramine, stored in distilled water and used within 6 months after extraction. Then, the teeth had their roots cut off approximately 1 mm below the cement–enamel junction with a diamond disc at slow speed (Isomet, Buehler, Lake Bluff, IL, USA) under water-cooling. Similarly, flat dentin surfaces were exposed after removing the occlusal enamel (Fig. 1a). Then, they were embedded in polyvinyl chloride (PVC) tube using acrylic resin (Jet, Artigos Odontológicos Clássico, SP, Brazil—Fig. 1b). The enamel-free, exposed dentin

surfaces were further polished on wet #600-grit silicon-carbide paper for 60 s to standardize the smear layer.

The teeth were divided randomly into 12 experimental conditions, according to the combinations of the main factors ( $n=5$  teeth): load methods (chisel and orthodontic-loop wire), cross-head speed (0.5, 1.0, and 5.0 mm/min) and adhesive system (Adper Single Bond 2 [SB2], 3M ESPE, St. Paul, MN, USA and XP Bond [XPB], Dentsply, Caulk, Milford, DE, USA).

The adhesive systems were applied onto the dentin surfaces (Fig. 1b) according to the instructions reported in Table 1. Before adhesive light-curing, five vinyl tygon tubes (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) with internal diameters of 0.7 mm and height of 0.5 mm were placed on the dentin surface (Fig. 1c) and carefully filled with composite resin (Filtek Z250, shade A2, 3M ESPE, St. Paul, MN, USA). Then, each resin cylinder was light-cured for 40 s (Optilux 500, Demetron, Danbury, CT, USA) with a power density of 600 mW/cm<sup>2</sup>. We avoided irradiating the other composite resin cylinders by protecting them with an aluminum foil. The power density of the curing device was regularly checked with a curing radiometer (Demetron, Orange, CA, USA). The teeth were stored in water at 37 °C for 24 h. The tygon tubes were carefully removed with a blade (Fig. 1d) and then checked with a light stereomicroscope at 10 $\times$  magnification. Specimens with evident air bubbles or gaps at the interface or with a flash of composite extending beyond the base were discarded.

We used a universal testing machine (Kratos Dinamometros, Cotia, SP, Brazil) for the microshear bond test. Each tooth was taken to the universal testing machine (Fig. 1e and f). Then, each resin cylinder bonded to the teeth was subjected to a load application—either the orthodontic-wire loop or the chisel method (Fig. 1e and f). For the former, a thin wire (0.2 mm diameter, Morelli Ortodontia, São Paulo, Brazil) was looped around half the



**Fig. 1.** Schematic drawing showing the specimen preparation and testing. (a) The teeth were cut with a diamond disk to obtain a flat dentin surface. (b) After dentin exposure and standardizing the smear layer, the adhesive systems were applied. (c) Then, the tygon tubes were placed on the dentin surface and (c) the lumen was filled with composite resin and light-cured. (d) After storage, each tygon tube was removed, so that only the bonded resin composite cylinder specimens could be observed. (e, f) Each tooth was placed in a jig and assembled in the universal testing machine for a microshear test with (e) a chisel or (f) an orthodontic-loop around the composite resin specimens. (g) The fracture pattern of the specimens was analyzed using SEM.

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