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Interfacial fracture toughness of aged adhesive–dentin interfaces

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ABSTRACT

Objective. To assess interfacial fracture toughness of different adhesive approaches and compare to a standard micro-tensile bond-strength (μTBS) test after 6 months water storage.
Methods. Chevron-notched beam fracture toughness (CNB) was determined using a modified ISO 24370:2005 standard. Adhesive–dentin micro-specimens (1.0 mm × 1.0 mm × 8–10 mm) were stressed in tensile until failure to determine the micro-tensile bond strength (μTBS).
Results. The highest mean μTBS and interfacial fracture toughness were measured for the multi-step adhesives Clearfil SE Bond (Kuraray Noritake) and OptiBond FL (Kerr). While large differences were observed in the bond strength values (from 7.4 to 27.2 MPa) of the one-step self-etch adhesives tested, interfacial fracture toughness was less different (from 0.7 to 1.0 MPa $m^{1/2}$). The adhesive with the lowest mean toughness (All-bond Universal, Bisco) had however the highest Weibull reliability, which might be a better parameter in regard to more consistent clinical performance. The self-adhesive composite Vertise Flow (Kerr) scored significantly lower at all levels.
Significance. Although the ranking of the adhesives tested using CNB and μTBS corresponded well, the outcome of CNB appeared more reliable and less variable.

1. Introduction

Bond strength testing still is the most common method to evaluate bonding effectiveness to dentin, despite they are criticized on many aspects [1]. From a previous study [2] it was concluded that after 1 week of water storage a chevron-notched beam interfacial fracture toughness (CNB-iFT) test set-up is a more accurate and reproducible alternative compared to the micro-tensile bond strength (μTBS) test. Nevertheless, the μTBS correlated well with the CNB-iFT, is more versatile and less laborious and time-consuming [2]. Bond durability is deemed to be the most relevant parameter to predict clinical performance [3–7]. Commonly used methods

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are 3 months to 1 year water storage [8], thermo-cycling [9], fatigue [10] and chemical aging [11]. The most validated and commonly used method is water storage of the test specimens [8,12,13]. Water storage may however not only affect the interaction with dentin, but also the bulk properties of dentin, adhesive resin and/or composite, increasing test variability and hampering correct interpretation of the results.

An interfacial fracture toughness test that more consistently tests the interfacial interaction by concentrating stress at a very specific small area along the adhesive joint, may resolve these issues. Self-etch adhesives based on the 10-MDP functional monomer are reputed for their clinical durability [14], probably to their unique interaction with hydroxyapatite [15,16], where the reacted salts reassemble in nano-layered structures that stabilize the resin–dentin joint. Different formulations/application techniques may however affect this interaction [17]. Therefore, the purpose of this study was to assess the durability adhesives bonded to dentin using both a μTBS and CNB-iFT test approach. The hypotheses tested were that (1) artificial aging has no influence on CNB-iFT and μTBS; and that (2) there is no correlation between CNB-iFT and μTBS data upon artificial aging.

2. Materials and methods

This study is a follow-up of a previous study [2] that investigated the short-term CNB-iFT and μTBS of different commercial adhesives. Specimens used for the present study originate from the same teeth as only half of the specimens were tested at baseline. Therefore, all materials and techniques presently employed were exactly the same and testing was executed by the same operators. Full methodological details were described before [2] and are repeated briefly underneath.

2.1. *Interfacial fracture toughness*

The fracture toughness of adhesive–dentin interfaces was determined using a chevron-notched beam (CNB) test, adapted from the modified ISO 24370 standard to measure fracture toughness of monolithic ceramics [18]. Rectangular sticks (3.0 mm × 4.0 mm wide; 25–30 mm long) with the composite/dentin interface positioned in the middle were prepared using a water-cooled diamond saw. At the composite/dentin interface, a chevron notch was prepared using an ultra-thin diamond blade (150 μm, M1DO8, Struers A/S) at a feed speed of 0.015 mm/s and a wheel speed of 1000 rpm. The tip of the chevron was positioned at the adhesive–dentin interface using a stereo-microscope. The specimens were stored in water for six months, transferred to the universal testing machine (Instron 5848 Micro Tester) and tested in a 4-point bend test setup with a crosshead speed of 0.05 mm/min. The outer and inner span was 20 and 10 mm, respectively. Next, the exact dimensions of the chevron notch were measured using a traveling microscope, after which the K_{IC} was calculated in MPa m$^{1/2}$ according to the ISO standard [18]. In order to determine fracture location, crack propagation and possible imperfections, all fractured surfaces were processed for scanning electron microscopy (SEM, JSM-6610LV, JEOL, Tokyo, Japan) using common preparation procedures, including fixation, dehydration and gold-sputter coating.

2.2. *Micro-tensile bond strength (μTBS)*

The bond strength to dentin was determined using a standardized micro-tensile bond strength protocol [19]. Adhesive–dentin micro-specimens (1.0 mm × 1.0 mm × 8–10 mm) were prepared using an automated precision water-cooled diamond saw (Accutom-50, Struers A/S, Ballerup, Denmark) and stored for 6 months in 0.5% chloramine solution at 37 °C. Then specimens were glued to a BIOMAT jig [19] and stressed in tension at a crosshead speed of 1 mm/min using a universal testing machine (Instron 5848 Micro Tester, Instron, Norwood, MA, USA). The number of pre-testing failures (ptf) was explicitly noted. The mode of failure was determined with a stereomicroscope at 50× magnification.

2.3. *Study setup and statistical analysis*

Both the CNB-iFT and μTBS of five adhesives and one self-adhesive composite (Table 1) were measured. For GB, SBU and ABU the adhesive was used in a self-etch mode. The CNB-iFT and μTBS data were statistically analyzed using Weibull analysis; pivotal confidence bounds were calculated using Monte Carlo simulation [20]. The different groups were compared at the 10% unreliability level (b10) and at the characteristic strength (b63.2 or 63.2% unreliability). To compare the CNB-iFT and μTBS, a correlation analysis on the respective means was performed. All tests were performed at a significance level of $\alpha = 0.05$ using a statistical software package (R3.01 and abrem package, R Foundation for Statistical Computing, Vienna, Austria).

3. Results

The μTBS results are presented in Table 2 and Fig. 1. The highest mean μTBS was measured for CSE, which performed, nevertheless, not significantly better than OFL. The latter scored not significantly different from SBU. The other two one-step self-etch adhesives (1-SEAs) revealed a significantly lower μTBS, but were not different among each other. The self-adhesive composite VF recorded a significantly lower μTBS with 19 ptf's out of 20 specimens. The ranking we obtained in this study confirmed the results of our previous test after 1 week of water storage [2]. Compared to that study, particularly CSE scored well with a higher Weibull modulus and thus lower variability. The 1-SEAs and VF scored significantly lower after 6 months of water storage. Except for CSE, all the adhesives revealed a lower Weibull modulus and thus increased variability after water storage. Failure analysis of the μTBS specimens resulted in a predominant cohesive failure in composite/dentin for OFL and CSE (Table 2). While 1-SEAs mainly failed interfacially, as observed by light microscopy, SEM analysis revealed that most specimens actually failed within the adhesive resin (Figs. 2(3a) and 3(1a)).

For CNB-iFT, the highest scale values were obtained for the multi-step adhesives (CSE and OFL), followed by the 1-SEAs (GB, SBU and ABU) and VF. Compared to the 1-week