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Correlative analysis of cement–dentin interfaces using an interfacial fracture toughness and micro-tensile bond strength approach

Evelise M. Souza[a,b], *Jan De Munck*[a], *Pong Pongprueksa*[a], *Annelies Van Ende*[a], *Bart Van Meerbeek*[a,*]

[a] *KU Leuven (University of Leuven), Department of Oral Health Sciences, BIOMAT & University Hospitals Leuven, Dentistry, Leuven, Belgium*

[b] *Pontifícia Universidade Católica do Paraná (PUCPR), School of Life Sciences, Graduate Program in Dentistry & Federal University of Paraná, Dental School, Restorative Dentistry Department, Curitiba, PR, Brazil*

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ABSTRACT

Objectives. To determine the interfacial fracture toughness (iFT) and micro-tensile strength (μTBS) of composite cements bonded to dentin.

Methods. Fifty feldspar ceramic blocks (Vita Mark II, Vita Zahnfabrik) were luted onto dentin using two self-adhesive (G-CEM LinkAce, GC; SpeedCEM, Ivoclar Vivadent), two self-etch (Multilink Primer & Multilink Automix, Ivoclar Vivadent; Scotchbond Universal & RelyX Ultimate, 3 M ESPE), and one etch-and-rinse (Excite F DSC & Variolink II, Ivoclar Vivadent) composite cement ($n = 10$). After 48 h in 100% relative humidity at 37 °C, one half of each tooth was sectioned in sticks with a chevron notch at the cement–dentin interface and tested in a 4-point bending test setup (iFT). The remaining half of the tooth was sectioned in micro-specimens and stressed in tension until failure (μTBS). The mode of failure was determined with a stereomicroscope at 50× magnification. Data were submitted to Weibull analysis and Pearson's correlation ($\alpha = 0.05$).

Results. At 10% probability of failure, no significant differences could be found using iFT, while the etch-and-rinse composite cement Variolink II presented a significantly higher μTBS at this level. At 63.2% probability of failure, the self-adhesive composite cement G-CEM LinkAce revealed a significantly lower μTBS and iFT, and the self-etch cement Multilink Automix also revealed a significantly lower μTBS than all other cements. The correlation found between iFT and μTBS was moderate and not significant ($r^2 = 0.618$, $p = 0.11$).

Significance. Overall, the etch-and-rinse and 'universal' self-etch composite cements performed best. The micro-tensile bond strength and interfacial fracture toughness tests did not correlate well.

* *Corresponding author at*: KU Leuven (University of Leuven), Department of Oral Health Sciences, Kapucijnenvoer 7, Block A—Box 7001, 3000 Leuven, Belgium.

E-mail addresses: bart.vanmeerbeek@kuleuven.be, bart.vanmeerbeek@uzleuven.be (B. Van Meerbeek).

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1. Introduction

In the mid 90's, the micro-tensile strength test was introduced by Sano et al. [1]; they applied this method to measure the ultimate tensile strength and modulus of elasticity of mineralized and demineralized dentin. Concurrently, this method was used to measure bond strength to tooth enamel and dentin (μTBS) [2]. A number of advantages have been attributed to the μTBS approach when compared to conventional tensile and shear bond strength test methods, among which the limited number of teeth needed (although today micro-specimens originating from a single tooth are no longer considered statistically independent), the occurrence of more adhesive than cohesive failures and thus measurement of a bond strength representing more the interfacial adhesive-tooth strength, the μTBS mean and variance that can be calculated per single tooth, the lower probability to incorporate interfacial defects that may falsely lower bond strength, the potential to test different experimental conditions in parallel on a single tooth (hereby enabling statistical comparison on tooth level), the potential to test different cavity configurations, and allowing high-resolution examination of failed specimens using SEM/TEM [3–6]. Principal variables identified to affect μTBS are specimen size and geometry [4,7,8], dentin region [9], size and shape of the cross-sectional area [10,11], misalignment of the applied load axis [11], as well as the selected method to handle pre-testing failures to calculate the μTBS [12]. Limitations of μTBS testing include, among others, the labor-intensive and technically demanding specimen preparation, the difficulty to measure low bond strengths (<5 MPa), the potential dehydration of specimens, the risk on specimen damage when removing it from the jig to which it was glued, and the difficulty to fabricate specimens with a consistent geometry without the aid of special equipment such as a Micro-Specimen Former (University of Iowa, Iowa City, IA, USA) [13].

Today, the μTBS test is the most frequently employed bond-strength test [6]. It however remains criticized, since μTBS data reported in different studies vary highly [6]. This should to a great extent be ascribed to the wide disparity in micro-specimen preparation and actual test parameters employed in the different research centers [14]. Other major criticism concerns the alleged inhomogeneous load distribution within the micro-specimen with the de-bonding stress imposed during tensile loading not necessarily concentrated at the adhesive-tooth bond and uniformly distributed across the actual interface. Previous research using finite element analysis (FEA) to compare the stress-concentration factor (Kt) for stick-shaped homogeneous and bi-material specimens with different notch geometries concluded that dumbbell and stick-shaped specimens are favored for μTBS testing; these micro-specimen geometries were shown to induce uniform stress distribution [15]. There is however no consensus in literature, since another more recent FEA study demonstrated that the main stress is not concentrated at the interface, but is located within the dentin and composite parts near the adhesive interface [16].

Hence, a fracture mechanics approach is considered more appropriate to assess bonding effectiveness [17,18]. This approach considers flaw size and features, component geometry, loading conditions and fracture toughness to predict fracture resistance at a flawed site [19]. By definition, fracture toughness is a property that describes the ability of a material containing a crack to resist fracture. Interfacial fracture toughness (iFT) has been proposed as an alternative method to measure bonding effectiveness in the laboratory. Having been applied in various forms, such as 'short rod chevron notch' [20,21], 'notchless triangular prism' [22], 'chevron notch beam' (CNB) [23,24], the tests generally appeared more accurate and reproducible; they were less test-dependent and revealed the interfacial bonding properties better [17,18]. The correlation between μTBS and iFT has been investigated in previous researches showing controversial results. While a moderate and non-significant correlation was found when bonding systems of different adhesive approaches were compared immediately [23], the same correlation tested after aging revealed a strong and highly significant correlation [24]. Our group recently miniaturized iFT to a so-called mini-iFT [25]. A significant and strong positive correlation was found between mini-iFT and μTBS. The new mini-iFT test appeared more discriminative and valid than the μTBS test to assess bonding effectiveness.

Fracture toughness tests have been widely used to investigate ceramics, composites, glass-ionomers, as well as enamel- and dentin–composite adhesive interfaces [17]. However, up to the date, no studies employed this approach to assess bonding effectiveness of composite cements to dentin. Therefore, the aim of this study was to assess iFT of composite cements bonded to dentin and to correlate the iFT data with μTBS data that were gathered in parallel for the same cements. The null hypotheses tested were that (1) there is no difference in interfacial bond strength among the composite cements tested and (2) iFT and μTBS of the cement–dentin interface do not correlate.

2. Methods

2.1. Tooth preparation

Fifty non-carious human third molars (gathered following informed consent approved by the Commission for Medical Ethics of KULeuven under the file S57622) were stored in 0.5% chloramine solution at 4 °C no longer than 6 months after extraction. The teeth were embedded in gypsum blocks and the occlusal third of the crowns was removed with a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), thereby exposing a flat mid-coronal dentin surface. The surfaces were checked for remaining enamel and exposed pulp tissue using a stereomicroscope (Wild M5A, Heerbrugg, Switzerland). The specimens were excluded in case the pulp chamber was exposed and if enamel was observed, it was promptly eliminated with a diamond bur.

A standardized bur-cut smear layer was produced by removing a thin layer of the dentin surface using a Micro-Specimen Former (University of Iowa, Iowa City, IA, USA) equipped with a high-speed cylindrical regular-grit (107 μm) diamond bur (842, Komet, Lemgo, Germany).