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**Review**

# Direct comparison of the bond strength results of the different test methods: A critical literature review

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ABSTRACT

*Objective.* The goal of this paper is to undertake a literature search collecting all dentin bond strength data obtained for six adhesives with four tests (shear, microshear, tensile and microtensile) and to critically analyze the results with respect to average bond strength, coefficient of variation, mode of failure and product ranking.

*Method.* A PubMed search was carried out for the years between 1998 and 2009 identifying publications on bond strength measurements of resin composite to dentin using four tests: shear, tensile, microshear and microtensile. The six adhesive resins were selected covering three step systems (OptiBond FL, Scotch Bond Multi-Purpose Plus), two-step (Prime & Bond NT, Single Bond, Clearfil SE Bond) and one step (Adper Prompt L Pop).

*Results.* Pooling results from 147 references showed an ongoing high scatter in the bond strength data regardless which adhesive and which bond test was used. Coefficients of variation remained high (20–50%) even with the microbond test. The reported modes of failure for all tests still included high number of cohesive failures. The ranking seemed to be dependant on the test used.

*Significance.* The scatter in dentin bond strength data remains regardless which test is used confirming Finite Element Analysis predicting non-uniform stress distributions due to a number of geometrical, loading, material properties and specimens preparation variables. This reopens the question whether, an interfacial fracture mechanics approach to analyze the dentin–adhesive bond is not more appropriate for obtaining better agreement among dentin bond related papers.

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## 1. Introduction to the review

When an assignment for a literature review involves the subject of "dentin bonding" an instantaneous shiver runs down the backbone as the amount of data is a knock out (over 6000 papers just with the Keywords: "bonding" AND "dentin") and when combined with the sorting through testing parameters and variables, it becomes a nightmare. The reasons for such a popular topic of research is of course the rapid development of bonding adhesives to dentin and the fact that the product screening test methods such as shear, tensile and microtensile are inexpensive testing routines in most dental school or research laboratories. Dentin bonding however has been notorious for high spread in the results, whether within the same laboratory or between laboratories using the same tests, and even 15–20 years ago suggestions were raised to improve the standardization of bond strength testing after reviewing possible reasons for such variability [1–7]. The introduction in 1994 of the microtensile bond strength test [8] allowing measurements of the tensile bond strength on very small surfaces ($\sim$1 mm$^2$) opened the research to regional differences within dentin, and had the advantage of producing many specimens from the same extracted tooth. It was thought that with this test, the characteristic high bond strength spread obtained in conventional shear and tensile tests would diminish due to a better stress distribution over a very small surface during loading, generating more interface failures (i.e. fewer cohesive failures) in dentin [8]. In 1999, Pashley et al. [9] reviewed positively the microtensile bond test and summarized its versatile usage providing new insights into strength of adhesion of restorative materials to clinically relevant sites and substrates and advocated this test as a means for evaluating the long-term durability of resin-hard-tissue bonds. Since then, the microtensile bond strength test has become the most used dentin bond test.

The philosophical question however to be asked here is what is the final goal of measuring bond strength? Is it to measure interfacial bond strength? Is it to distinguish product A from product B? Is it to rank the products according to their results? Is it to understand localized degradation within a bonded surface? Is it a test to indicate reliability of the bonding? Which test should be used? Are the results dubious? Well, considering the information gathered from Finite Element Analyses (FEA) generated for shear [4,10–12], microshear [11], tensile [4,7] and microtensile [13], almost every possible testing variable (i.e. specimen's geometry, loading condition, film thickness, modulus of elasticity of the materials involved) has a significant influence on the stress state and thus on the bond strength values.

Therefore, the goal of this paper is to perform a 10 year literature search collecting dentin bond strength data obtained for six adhesives with four tests (shear, microshear, tensile and microtensile) and to critically analyze the results with respect to average bond strength, coefficient of variation, mode of failure and product ranking. No attempt is made to undertake additional statistical analysis, as the major focus of this paper is to point out limitations of the most popular methods for evaluating adhesion. Alternative approaches to bond strength evaluation including the use of Weibull statistics and fracture mechanics will be discussed.

## 2. Materials and methods

A PubMed search was carried out for the years between 1998 and 2009 to identify publications on bond strength measurements of resin composite to dentin using shear, tensile, microshear and microtensile. The search would poke around Keywords: such as "(Clearfil SE bond) AND (bond) AND (dentin) NOT (bovine) NOT (primary) NOT (enamel) NOT (root) NOT (fiber)" as an example. The six adhesive resins selected were: (a) two *three step* systems where the etching (and rinsing); priming; and bonding are carried out separately (OptiBond FL; Kerr; Orange; CA; USA; and Scotch Bond Multi-Purpose Plus; 3 M Espe; St. Paul; MN; USA); (b) two *two-step self-priming* systems which include etching (and rising) followed by a self-bonding primer (Prime & Bond NT; Dentsply/De Trey; Konstanz; Germany; and Single Bond; 3 M Espe; St. Paul; MN; USA); (c) a *two-step self-etching* system where the etch and prime are together followed by a bonding step (Clearfil SE Bond; Kuraray; Osaka; Japan); and (d) a one step "*all-in-one*" system where the etching; priming; and bonding are combined into a single step (Adper Prompt L Pop; 3 M Espe; Seefeld; Germany).

The selected criteria for inclusion within the literature search were: (1) the bonding substrate: human coronal to mid-coronal dentin of molars, premolar and central incisors; (2) the storage media for the extracted human teeth: formalin, thymol, chloramine, sodium azide or saline solution; (3) the post-extraction storage time prior to sample preparation: 15 days to 6 months; (4) the dentin surface preparation: sandpaper from 180 to 1200 grit SiC, fine or medium diamond burs, tungsten carbide burs; (5) application of the adhesives to the prepared dentin surfaces following the manufacturers' instructions; (6) the storage time and media of the bonded specimens prior to testing: 10 min (immediate) or 24 h up to 14

**Table 1 – Average bond strengths from Figs. 1 to 6. CSE Bond (Clearfil SE Bond), SB (Single Bond), P&B NT (Prime & Bond NT), SBMP+ (Scotchbond Multi Purpose Plus), OptB FL (OptiBond FL), PLPop (Adper Prompt L Pop).**

| | CSE Bond | SB | P&B NT | SBMP+ | OptB FL | PLPop |
|---|---|---|---|---|---|---|
| Shear | 23.2 (7.1) | 12.4 (7.8) | 17.7 (5.2) | 17.0 (5.7) | 23.1 (7.9) | 13.4 (5.1) |
| m-Shear | 41.5 (11.6) | 38.9 (4.9) | 20.8[a] | 20.7 (3.0) | 22.7[a] | 22.8[a] |
| Tensile | 22.9 (5.5) | 13.8 (4.6) | 11.9 (2.4) | 10.1 (8.6) | 18.7 (5.5) | 4.5 (2.5) |
| m-Tensile | 42.5 (11.8) | 36.1 (10.4) | 31.5 (10.0) | 30.2 (8.5) | 48.0 (13.7) | 25.8 (13.5) |

[a] Values without a standard deviation have been obtained from one publication only.

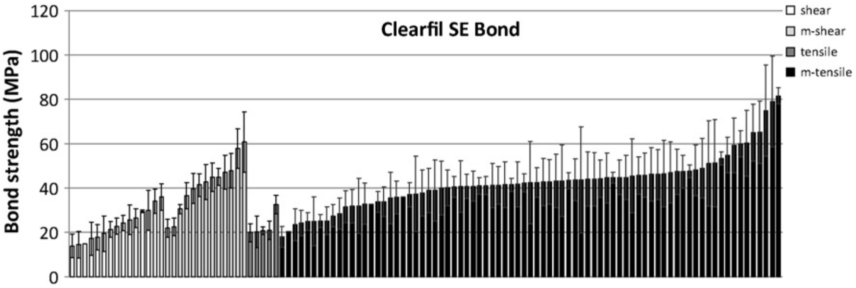

**Fig. 1 – Individual results of bond strength to dentin for Clearfil SE Bond. References are listed in sequence. References [96] and [118] listed twice in microtensile reported different values for different grit surfaces. Shear (15 publications [21–35]), microshear (13 publications [36–48]), tensile (5 publications [49–51,14,52]) and microtensile (77publications [53–90,46,91–118, 96,119–122,14,123,124,118,125–127]).**

days in a humid environment, (distilled) water, artificial saliva or 0.5% chloramine at 37 °C; (7) no thermocycling of specimens during storage or thermocycling not beyond 1500 cycles; (8) specimens tested without pulp pressure.

The data were compiled in spreadsheets and analyzed for: (1) variability in bond strength between tests for a same adhesive, (2) coefficient of variation of bond strength within tests for a same adhesive, (3) mode of failure, and (4) ranking dependence of products by test.

## 3. Results and discussion

### 3.1. *Variability in bond strength between tests for a same adhesive*

The bond strengths for six adhesive resins bonded to dentin listed in 147 publications using any of the four tests are summarized in Table 1 and in Figs. 1–6. Detailed references are given in the following graphs (1–6) depending on the test and product. The graphs confirm for all the selected adhesives the large discrepancies between dentin bond values for the same adhesive measured in different laboratories with the same test as well as when using different tests.

The overall trend (Table 1) is that macro-tests with bonding surfaces around 7 mm$^2$ as encountered in shear and tensile tests deliver lower bond strength values than their equivalent micro-tests with bonding surface around 1 mm$^2$. Hence, the adhesives for which the most publications were found in all four tests, Clearfil SE Bond and Single Bond, showed microbond values 2–3 times higher than their equivalent macrobond values (shear and tensile). The overall results of macro testing versus micro testing as expressed in Table 1 are in line with the findings of several authors showing that the tensile bond strength is inversely related to bonded surface area [8,14–17]. Hence, the smaller the bonding surface, the higher the bond strength values.

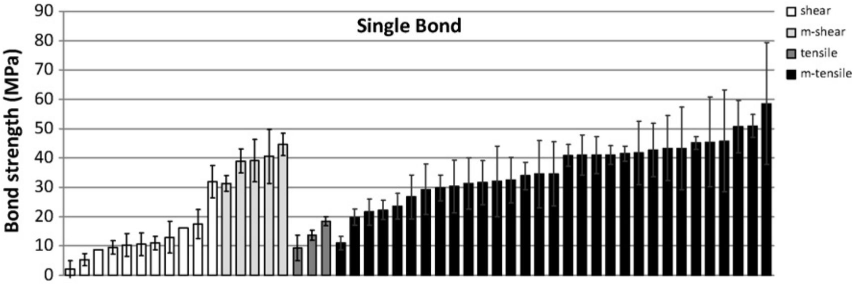

**Fig. 2 – Individual results of bond strength to dentin for Single Bond. References are listed in sequence. Shear (11 publications [128,27,23,129–132,15,133–135]), microshear (5 publications [38,41,40,47,44]), tensile (3 publications [15,50,51]) and microtensile (31 publications [132,136–138,93,60,66,57,109,55,122,76,92,89,139,15,83,79,73,82,80,99,106,110,140,104,91,141,90,142,65]).**

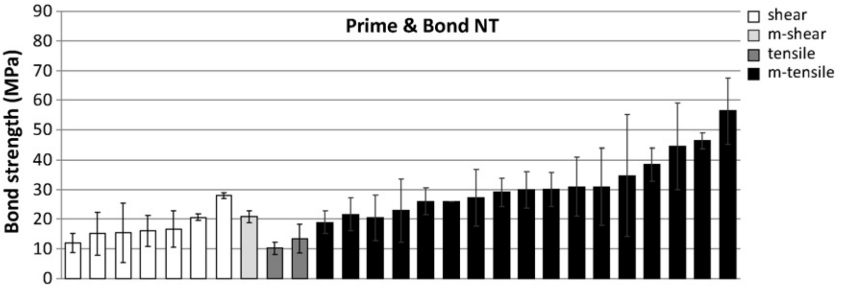

**Fig. 3 – Individual results of bond strength to dentin for Prime & Bond NT. References are listed in sequence. Shear (7 publications [28,24,143,25,144,145,32]), microshear (1 publication [36]), tensile (2 publications [52,50]) and microtensile (17 publications [146,137,93,91,60,54,71,110,147,106,76,75,112,79,99,104,70]).**

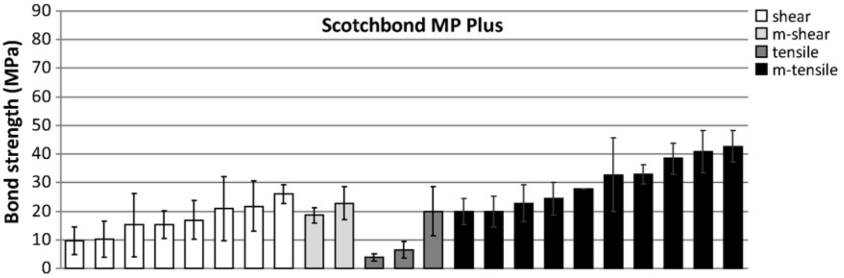

**Fig. 4 – Individual results of bond strength to dentin for Scotchbond Multi Purpose Plus. References are listed in sequence. Reference [152] listed twice in microtensile reported different values for different dentin location. Shear (8 publications [15,26,148–150,30,24,134]), microshear (2 publications [36,17]), tensile (3 publications [150,15,151]) and microtensile (9 publications [152,17,149,152,72,15,60,57,82,80]).**

The overall increase in bond strength for small surfaces can be explained by the Weibull distribution showing mathematically that an increase in specimen size increases the probability of encountering a strength-limiting flaw and that the specimen's flaws are size distributed [18].

Direct comparisons between tensile and microtensile for all six adhesives showed microtensile bond values 2–5 times higher (Table 1). Direct comparison between shear and microshear could only be made for three adhesives (Clearfil SE Bond, Single Bond, Scotchbond Multi Purpose (MP) Plus) for which minimum of two microshear values were reported. For these products, the microshear bond strengths were 1.2–3 times higher than their shear values.

Average macroshear bond values were higher than macrotensile for four adhesives (Prime & Bond NT, Scotchbond MP Plus, OptiBond FL and Prompt L-Pop) but not for Clearfil SE Bond and Single Bond for which we had the most references (Table 1). In a meta-analysis reviewing factors involved in dentin adhesion, Leloup et al. [19] also reported significantly higher shear bond data compared to tensile. Nevertheless, these comparisons between shear and tensile should be carefully made in full knowledge of the differences in their stress distributions as these tests have non-uniform tensile or shear stress states at the interface due to variation in the specimens' geometries, loading configuration (i.e. wire loop, knife edge, blade, hook, point of loading, alignment,

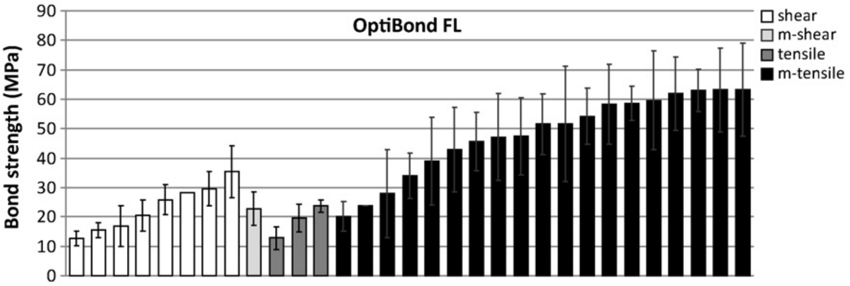

**Fig. 5 – Individual results of bond strength to dentin for OptiBond FL. References are listed in sequence. Reference [96] listed twice in microtensile reported different values for different grit surfaces. Shear (8 publications [130,153,134,28,22,23,32,154]), microshear (1 publication [17]), tensile (3 publications [155,52,154]) and microtensile (18 publications [17,72,78,156,157,74,158,141,117,87,107,159,123,85,75,96,88,112,96]).**

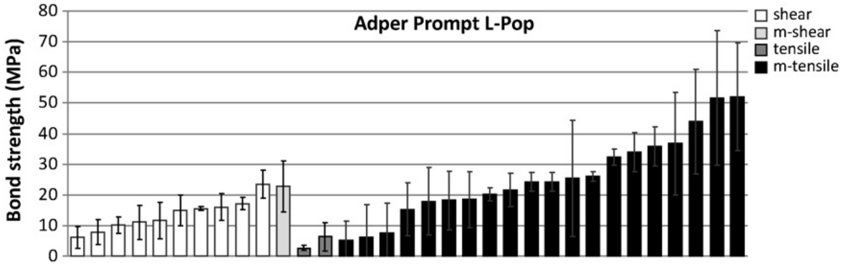

**Fig. 6 – Individual results of bond strength to dentin for Adper Prompt L-Pop. References are listed in sequence. Reference [118] listed twice in microtensile reported different values for different grit surfaces. Shear (10 publications [24,143,153,28,22,35,145,34,150,31]), microshear (1 publication [45]), tensile (2 publications [150,52]) and microtensile (19 publications [146,123,118,118,88,70,79,160,161,53,162,93,120,104,127,85,163,125,126,140]).**

stressing rate) and modulus of elasticity of the restorative resins, all of which influence the final stress state and stress concentration location and thus the measured bond strength [4,7,10,20].

In order to better understand the literature findings, Table 2 summarizes the percentage of the reviewed published data that used a specific surface finish grit, crosshead speed, thermocycling and time prior to testing. As one can read from the table, the majority (highlighted in the table) of the reported studies used a 600 grit SiC surface finish, a 0.5 or 1 mm/min crosshead speed, no thermocycling, and 24 h in water prior to testing.

**Table 2 – Percentage of the reviewed published data using a specific surface finish grit, crosshead speed, thermocycling and time prior to testing.**

| | | Shear | m-Shear | Tensile | m-Tensile |
|---|---|---|---|---|---|
| Surface finish | 180-grit | 0 | 0 | 0 | 15 |
| | 220-grit | 0 | 0 | 0 | 2 |
| | 320-grit | 12 | 0 | 0 | 2 |
| | 400-grit | 3 | 0 | 12 | 1 |
| | 500-grit | 0 | 0 | 0 | 1 |
| | 600-grit | 38 | 100 | 59 | 55 |
| | 800-grit | 12 | 0 | 29 | 4 |
| | 1000-grit | 12 | 0 | 0 | 0.5 |
| | 1200-grit | 12 | 0 | 0 | 2 |
| | Fine diamond bur | 0 | 0 | 0 | 10 |
| | Regular diamond bur | 0 | 0 | 0 | 2 |
| | Coarse diamond bur | 0 | 0 | 0 | 0.5 |
| | 100 μm bur | 0 | 0 | 0 | 0.5 |
| | Carbide bur | 8 | 0 | 0 | 2 |
| | Diamond saw | 0 | 0 | 0 | 2 |
| | Isomet | 0 | 0 | 0 | 0.5 |
| | Ecomet 3 | 3 | 0 | 0 | 0 |
| Crosshead speed (mm/min) | 0.50 | 39 | 9 | 88 | 43 |
| | 0.75 | 7 | 0 | 0 | 0 |
| | 1.00 | 32 | 91 | 0 | 57 |
| | 1.20 | 2 | 0 | 0 | 0 |
| | 2.00 | 3 | 0 | 0 | 0 |
| | 3.00 | 2 | 0 | 0 | 0 |
| | 5.00 | 15 | 0 | 12 | 0 |
| Thermocycling | Yes | 21 | 0 | 0 | 6 |
| | No | 79 | 100 | 100 | 94 |
| Time prior to testing | Immediate | 12 | 0 | 0 | 2 |
| | 1 h | 0 | 9 | 0 | 0 |
| | 12 h | 0 | 0 | 0 | 2 |
| | 24 h | 61 | 60 | 100 | 86 |
| | 48 h | 2 | 9 | 0 | 2 |
| | 72 h | 0 | 0 | 0 | 4 |
| | 3 d | 0 | 0 | 0 | 1 |
| | 7 d | 23 | 22 | 0 | 3 |
| | 10 d | 2 | 0 | 0 | 0 |

**Table 3 – Mean coefficient of variation (±standard deviation, in %) obtained from bond strength data as a function of type of test and adhesive.**

| Adhesive | Bond strength test | | | |
|---|---|---|---|---|
| | Shear | m-Shear | Tensile | m-Tensile |
| Clearfil SE Bond | 25 ± 13 | 15 ± 4 | 20 ± 10 | 23 ± 10 |
| Single Bond | 42 ± 35 | 14 ± 6 | 22 ± 21 | 22 ± 10 |
| Prime & Bond NT | 31 ± 22 | 9 ± 0[a] | 28 ± 12 | 28 ± 14 |
| Scotchbond MP Plus | 45 ± 18 | 20 ± 8 | 40 ± 6 | 22 ± 9 |
| OptiBond FL | 24 ± 8 | 25 ± 0[a] | 21 ± 11 | 26 ± 10 |
| Prompt L-Pop | 33 ± 18 | 36 ± 0[a] | 53 ± 28 | 49 ± 44 |

[a] Values without a standard deviation have been obtained from one publication only.

### 3.2. *Coefficient of variation of bond strength within tests*

The large spread in the results of bond testing has been explained by many papers using FEA. These authors have continuously alerted the scientific community of massive stress concentrations in all dentin bond tests. Major differences in the stress states, non-uniform stress distributions were pointed out for shear [4,7,10–12], microshear [11], tensile [4,7] and microtensile [13]. Almost every possible testing variable (i.e. specimen's geometry, loading condition, film thickness, modulus of elasticity of the materials involved) has a significant influence on the stress state and thus on the bond strength values and is responsible for the inconsistencies in the results.

Table 3 shows the average coefficients of variation (CVs) obtained from 147 publications on bond strength data for each test and adhesive with the corresponding standard deviations.

Overall, for all six adhesives, the CV in microtensile ranged between 22% and 49%, in tensile between 20% and 53%, in shear between 24% and 45%. Due to the lack of references, no comments can be made for the microshear test. Nevertheless, the CV seemed to be rather product dependent as the one step adhesive (Prompt L-Pop) systematically showed very high variation in all tests.

As expected shear test data showed a high CV. The differences are related to the fact that shear bond strength tests have non-uniform stresses generated within the reaction zone, which can have a significant effect on the mode of failure. As shown with FEA, complex stress states occur at the interface in which high tensile stresses are generated due to the bending moment resulting during shear testing [4,7,10–13]. These tensile stresses will predispose the crack to deviate into dentin resulting in dentin pull-out [12]. Bond strength values obtained from a fractured surface containing cohesive failure of dentin or resin will be meaningless as the measured "nominal" bond strength will reflect a mixture of mechanical properties of both dentin and resin rather than the performance of the adhesive tested [7,10,12] (see results in Section 3.3). In addition, the results are not coming from one single standard shear test but from different test designs using a loop or a chisel for load application as well as different crosshead speeds (from 0.5 mm/min to 5 mm/min, see Table 2). These variations in shear test design have been shown to influence the bonds results and therefore contribute to the high scatter in the data [164].

Although the microtensile bond test has been developed among other purposes to lower the CV due to its smaller bonding surface area and lower number of possible strength limiting flaws [8,9], our direct comparison of the microtensile with the tensile test did not show a smaller CV for any of the adhesives with the exception of Scotchbond MP Plus. Explanation for our findings in Table 3 come from a recent Finite Element Analysis by Ghassemieh [13] as well as critical analysis of different parameters influencing the microtensile bond strength by Poitevin [74]. Variables such as specimen shape (stick, dumbbell, hourglass), flaws (e.g. air bubbles) in the adhesive or grinding flaws during specimen preparation, thickness of the adhesive, modulus of elasticity differences, jigs and angle of load application will influence to various

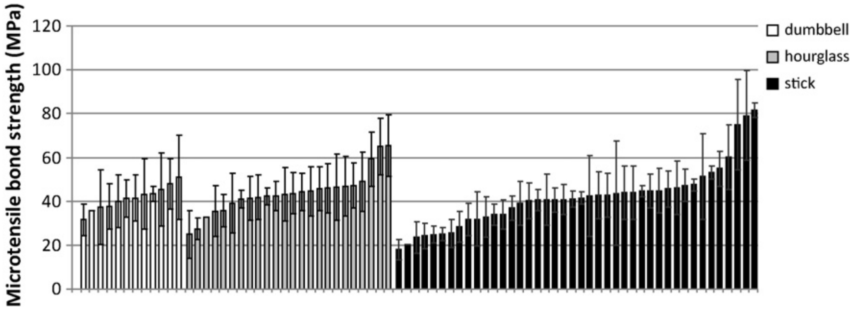

**Fig. 7 – Microtensile bond strength for Clearfil SE Bond for different specimen shapes: dumbbell (11 publications [63,72,74,75,78,87,88,96,97,107,117,96]), hourglass (23 publications [58,61,67,70,71,77,85,86,90,46,92,95,98,102,103,109,111–114,118,122,124,118]), and sticks (42 publications [53–57,59,60,62,64–66,68,69,73,76,79–84,89,91,93,94,99–101,104–106,108,110,115,116,119–121,123,125–127]). Data are based on results from Fig. 1. References [96] and [118] listed twice reported different values for different grit surfaces.**

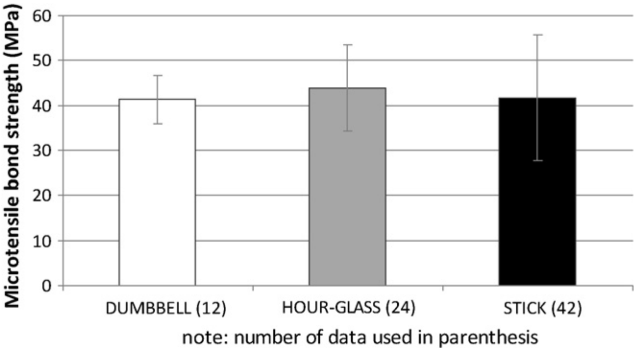

**Fig. 8 – Average microtensile bond strength from individual data shown in Fig. 7 for Clearfil SE Bond for different specimen shapes: sticks, dumbbell and hourglass.**

degrees the stress distribution in each specimen design and thus contribute to the variations in the bond strength results [13]. When comparing the microtensile bond strength results for Clearfil SE Bond between sticks, dumbbell or hourglass-shaped specimens (Figs. 7 and 8), our findings confirm the FEA of Ghassemieh [13] showing no difference in the bond strength values between sticks and dumbbell-shaped specimens (Fig. 8). However, the hourglass average bond strength was slightly higher (Fig. 8) than for the two other shapes which is not in accordance with the FE predicting lower bond strength values for hourglass configurations [13]. The spread within each shape was still quite important with 28.6% coefficient of variation for the dumbbell-shape followed by the hourglass with 23.8% and 21.5% for the sticks.

An additional source for the scatter in the microtensile test results is the pre-test failure. Spontaneous interfacial debonded specimens are not treated in the same statistical manner by all research papers. The majority of the papers reporting pre-test failures will include these as zero values in the statistical analysis, whereas 30% will report the number of pre-test failures but not include them in the statistical analysis. Overall, only 30% of the papers mentioned pre-test failures.

### 3.3. *Mode of failure for each test*

Care should be taken when interpreting the failure mode as there is no clear consensus in the literature regarding their classification, nor have these failure modes been diagnosed based on the same microscopic analytical tools [6]. If large cohesive failures within dentin or resin can be evaluated with a stereomicroscope at low magnification, the decision on the mode of failure for the adhesive interface or mixed failures can only be properly made using a Scanning Electron Microscope at high magnification [156,165].

Table 4 reports percentage of failure modes as described in the articles reviewed. Thus, “cohesive” (coh) regroups all cohesive failures in dentin or resin as well as the non-specified cohesive failures. “Adhesive” (adh) represents failures at the adhesive interface, and “mixed” (mix) designates a mixture of adhesive and cohesive failure within the same fractured surface. The problem arises when interpreting the “mixed” (mix) failures from the literature. One does not know if “mixed” means the crack path remains mainly within the adhesive interface but also includes some small areas of resin and/or dentin, or if a larger region of the fractured surface also includes cohesive failure in dentin and/or resin. The latter should clearly be rejected from the data [12,13].

The high amount of cohesive failure with shear was no surprise [4,11,12] whereas the pooled results for the microtensile bond test, which is thought to be less prone to cohesive failures due to the smaller bonding surface area [8,9], showed, respectively, 20% and 39% cohesive failure for the two leading bond strength products, Clearfil SE Bond and OptiBond FL (Table 3). Cohesive failure in dentin or resin can occur with the microtensile bond test due to errors in the alignment of the specimen along the long axis of the testing device [165] or from the introduction of microcracks during cutting or trimming of the specimens [166,167]. These specimens should be discarded from the data set [13,165].

If one considers the additional uncertainty arising from the mixed failures for all bond tests, which may well be predominantly within resin or dentin, the overall bond strength results become very unreliable as they represent breaking stresses resulting from different materials with different mechanical properties and thus no longer are representative of adhesive bond strength values. Needless to say on the basis of the modes of failure summarized in Table 4 of the reviewed papers and the uncertainty regarding which fractured surface was involved, the average bond strength data summarized in Table 1 reflects a massive mix of strength values which cannot be related to a “true” interfacial bond.

A further issue with strength tests be they tensile or shear are that they measure the stress to *initiate* fracture from the largest flaw. As such the subsequent extension of the crack through the remnant cross-section does not reflect on the crit-

**Table 4 – Percentage of failures reported as cohesive in resin or dentin (coh), adhesive (interface), or mixed (mix) (cohesive resin or dentin and adhesive interface) for 147 publications (see Figs. 1–6 for references).**

| | Shear | | | m-Shear | | | Tensile | | | m-Tensile | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | coh | adh | mix | coh | adh | mix | coh | adh | mix | coh | adh | mix |
| CSE | 27 | 48 | 26 | 13 | 17 | 70 | 57 | 20 | 24 | 20 | 32 | 49 |
| SB | 25 | 41 | 35 | 15 | 50 | 35 | 7 | 52 | 42 | 5 | 31 | 63 |
| P&BNT | 26 | 57 | 17 | – | – | – | 0 | 70 | 30 | 14 | 53 | 33 |
| SBMP+ | 17 | 23 | 61 | 0 | 67 | 33 | – | – | – | 15 | 63 | 22 |
| OptFL | 65 | 21 | 14 | 0 | 75 | 25 | 28 | 56 | 16 | 39 | 20 | 41 |
| PLPop | 16 | 80 | 4 | 36 | 0 | 64 | 0 | 100 | 0 | 19 | 50 | 31 |

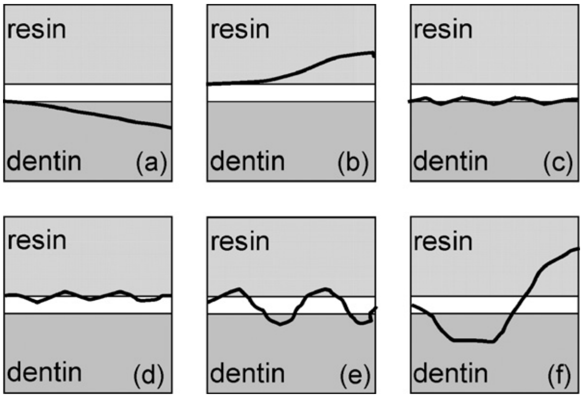

**Fig. 9 – Possible failure modes: (a) cohesive in dentin, (b) cohesive in resin, (c) adhesive (interface dentin–adhesive), (d) adhesive (interface resin–adhesive), (e) mixed dentin–adhesive–resin (small portions of dentin or resin involved in the fracture surface), (f) mixed dentin–adhesive–resin (large portions of dentin or resin involved).**

ical load for the failure event. Ideally one should use classical fractography techniques (with SEM) to identify the fracture initiation site and determine whether this lies at the interface or elsewhere.

Fig. 9a–f is a schematic representation of possible failure modes in adhesive resin bonding to dentin. Depending on the fracture path, failure modes as in (a), (b) and (f) should be rejected from the bond strength data [4,7,10,12].

### 3.4. *Ranking dependence by test*

Although the comparison of products was not the purpose of this review, the data of all six adhesives were pooled from all four tests and the ranking compared. As seen in Fig. 10, the ranking changes depending on the test used. For instance, OptiBond FL ranked first in microtensile, second in tensile and shear, and fourth in microshear. Similarly, Single Bond ranked second in microshear, third in tensile and microtensile, but fifth only in shear. Inconsistencies in ranking were pointed out by Sudsangiam [168] and Moll [154] who also showed an operator dependency. Nevertheless, with the exception of microshear, the three-step adhesive OptiBond FL always ranked in the top products as well as the two-step adhesive Clearfil SE Bond. Similarly, the one-step Promp L-Pop adhesive always ranked low.

### 3.5. *The use of Weibull statistics for bond strength data*

The strength of brittle materials such as ceramics, resin composites and tooth structure (i.e. enamel and dentin) cannot be specified by a single value due to the variability in strength-controlling flaws existing in these structures. Instead a probabilistic approach for the likelihood of failure is used which accounts for the area, size and stress variation throughout the object of relevance. The basis of the Weibull statistical approach is that with increasing size the chance of finding a critical defect or flaw of a structure increases. Such a size dependence of the strength does not form the basis of most other common statistical analysis treatments. This approach forms the basis for safely predicting the useable design stresses in service for bodies much larger and more complex than can be tested in the laboratory.

In the same category of brittleness and thus presence of strength-controlling flaws, lays the adhesive bond of filled or unfilled resin to dentin. The overall performances of these brittle materials and adhesives bonded to dentin can be better evaluated by means of predicting the likelihood of failure at specific stress levels using the Weibull distribution function expressed by two parameters, the Weibull characteristic strength ($\sigma_0$) and the Weibull modulus ($m$) which reflects the flaw distribution, and thus the variability of the results [18,169,170].

The ISO/TS 11405:2003 [171] recommends the use of the Weibull distribution for the analysis of bond strengths that are non-normally distributed. Unfortunately, most bond strengths papers used means and standard deviation, rather than a probabilistic failure approach which would provide insights as to the reliability (Weibull $m$ parameter) and the probability of failure for a given stress level. As indicated in Table 3, the high coefficient of variation of 20–50% for shear, tensile and microtensile indicate that the data do not fit a Gaussian distribution. High variability (i.e. high spread) in the bond strength to dentin for an adhesive will translate into a low Weibull $m$, which means low reliability of the characteristic bond strength (Weibull $\sigma_0$) due to the presence of critical flaws. Adhesives showing high Weibull moduli are to be favored for achieving similar bond strength and are generally less technique sensi-

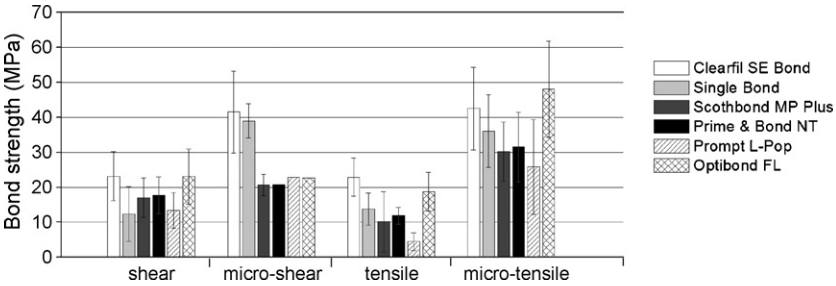

**Fig. 10 – Comparison of the bond strength results of the six adhesives in all four tests based on 147 publications (see Figs. 1–6 for references). The ranking seems to be dependent on the test used.**

**Table 5 – Weibull modulus (*m*) for some adhesives bonded to dentin tested in shear, tensile or microtensile.**

| | Bradna et al. Shear *n* = 20 [28] | Braga et al. Tensile *n* = 10 [175] | Armstrong et al. m-TBS d-bell *n* = 21–22 [172] | Armstrong et al. m-TBS d-bell *n* = 20–22 [174] | Armstrong et al. m-TBS d-bell *n* = 20 [173] | Burrow et al. m-TBS hrglass *n* = 10–19 [18] | Sattabanasuk et al. m-TBS d-bell *n* = 18–19 [96] | Knobloch et al. m-TBS sticks *n* = 20 [54] | Vachiramon et al. m-TBS d-bell *n* = 30 [176] |
|---|---|---|---|---|---|---|---|---|---|
| Prompt L-Pop | 2.1 | | | | | | | | |
| Adper Prompt | 7.5 | | | | | | | | |
| Prime & Bond NT | 4.3 | 3.4 | | | | | | 3.1 | |
| OptiBond FL | 4.4 | | 3.4 | | 3.0 | | 4.7 | | |
| OptiBond Solo Plus SE | 7.2 | | | | | | | 3.1 | |
| OptiBond Solo | | 2.8 | | | | | | | |
| Clearfil SE Bond | 6.5 | | | 3.0 | | | 3.1 | 2.8 | |
| Gluma CB | 8.3 | | | | | | | | |
| AdheSE | 7.6 | | | | | | | | |
| Excite | 5.1 | | | | | | | | |
| iBond | 6.4 | | | | | | | 3.8 | |
| Xeno III | 4.3 | | | | | | | | |
| Single Bond | | 3.6 | | 4.6 | | | | | |
| Single Bond Plus | | | | | | | | | 3.3–5.1 |
| Scotchbond MP | | | | 3.7 | | | | | |
| EBS-Multi | | | | | | 3.1 | | | |
| Gluma One Bond | | | | | | 5.0 | | | |
| One Coat | | | | | | 3.0 | | | |
| Prime & Bond | | | | | | 1.9 | | | |
| Prime & Bond 2.1 | | 3.4 | | | | | | | |
| PermaQuick | | | | | | 3.5 | | | |
| Solid Bond | | | | | | 1.7 | | | |
| Clearfil Liner B 2V | | | | | | 1.8 | | | |
| G-Bond | | | | | | | | 3.7 | |
| Clearfil S3 Bond | | | | | | | | 3.2 | |

**Table 6 – Interfacial fracture toughness ($K_{Ic}$) or strain energy release rate ($G_{Ic}$) reported for some adhesives bonded to dentin. Legend: CVSrod = chevron short rod, CVSbar = chevron short bar, SENB = single edge notched beam, NTP = notchless triangular prism, SB2 = Scotchbond 2, SBMP = Scotchbond Multipurpose, SBMP+ = Scotchbond Multipurpose Plus, AB2 = All-Bond 2, CLB2 = Clearfil Liner Bond 2.**

| Author | Test specimen | Product | $K_{Ic}$ (MPa $\sqrt{m}$) | $G_{Ic}$ (J/m$^2$) | Shear (MPa) | Tensile (MPa) |
|---|---|---|---|---|---|---|
| Tam and Pilliar [179] | CVSrod | SB2 | 0.20 (0.14) | | | 1.7 (0.9) |
| | | SBMP | 0.34 (0.21) | | | 2.7 (1.9) |
| | | AB2 | 0.69 (0.41) | | | 8.4 (4.0) |
| Lin and Douglas [182] | CVSbar | SB2 | | 42.8 (7.8) | | |
| | | SBMP | | 75.0 (10.5) | | |
| Armstrong et al. [181] | CVSbar | AB2 | 0.88 (0.24) | | | |
| Toparli and Aksoy [180] | SENB | SBMP | 0.74 (0.04) | | | |
| Tantbirojn et al. [183] | CVSrod | SB | | 107.0 (26.0) | 14.8 (3.9) | |
| | | SBMP+ | | 93.0 (24.0) | 14.6 (2.3) | |
| Tam et al. [178] | CVSrod | AB2 | 0.64 (0.41) | | | |
| | | CLB2 | 0.43 (0.14) | | | |
| Far and Ruse [177] | NTP | SB | 0.94 (0.12) | | | |

tive. As a reference, typical Weibull *m* value for stainless steel is 100, for engineering ceramics 10 and for chalk 5 [18].

During the review, only a few papers were found that used Weibull statistics for commercial adhesives bond strength evaluation [18,28,54,96,172–176]. The reported *m* parameter using shear, tensile or microtensile tests is summarized in Table 5. Six studies using the microtensile bond test [18,54,172–174,176] reported Weibull *m* values ranging from 1.7 to 5 for 16 adhesives bonded to dentin indicating high dispersion and a rather poor reliability (low *m*). Similarly, *m* data of four adhesives using a tensile bond test [175] ranged between 2.8 and 3.6. Recently, Bradna et al. [28] applied Weibull analysis to 11 adhesives bonded to dentin and tested in shear. The reported Weibull moduli ranged between 2.1 and 8.2 showing significant reliability differences among the products.

Another indication of the significance of the Weibull approach is that most tests have been conducted with relatively small bonded areas, 1 mm$^2$ for the microtensile and 7 mm$^2$ for the tensile tests. These areas are often much smaller than clinically considered which may be greater than 70 mm$^2$ for a large MOD molar cavity. Based on the ratio of the pooled data in Fig. 10 for the microtensile and tensile results for all the materials considered the tensile values were approximately only half of the microtensile. If we then use the simple Weibull relationship for the strengths being related to the areas, that is $\sigma_0 = \sigma\,(A/A_0)^{1/m}$, where $\sigma_0$ and $A_0$ are the microtensile strength and area, $\sigma$ and $A$ are the tensile strength and area. The ratio of $\sigma_0/\sigma$ is approximately 2 (see Fig. 10). Ratio of $A/A_0$ is 7. Therefore from $\sigma_0/\sigma = (A/A_0)^{1/m}$ we have $2 = (7)^{1/m}$. Thus $m = 2.8$. On this basis one can then predict the consequence for the reduction in strength associated with the area of “real” large bonded cavity say 70 mm$^2$ or 25 mm$^2$ for a medium sized cavity. So, let us assume $\sigma_0 = 40$ MPa, $A_0 = 1$ mm$^2$, $A_{(\text{tooth cavity})} = 70$ mm$^2$ and $m = 2.8$, we would expect a bond strength $\sigma_{(\text{tooth cavity})}$ of 8.8 MPa which would be approximately half that of the 7 mm$^2$ results. With the same assumption as before, if we are dealing with a medium size cavity of 25 mm$^2$, the bond strength would be of 12.7 MPa. These values are very low and if the bonded composite resin were to develop appreciable shrinkage or thermo-elastic stresses the likelihood of interface failure would be very high.

The question that remains is how different would these Weibull scale ($\sigma_0$) and shape (*m*) parameters look if all the specimens showing cohesive failures were rejected from the data.

Overall, the reliability performances of bond strength data as reported in Table 5 remained low, confirming the limitations of bond tests prone to high scatter from non-uniform stress states as already discussed as well as a variety of strength-controlling flaws present in the specimen.

### 3.6. *Fracture mechanics approach for interfacial bond assessment*

Considering the still high discrepancies in the bond strength results found in this literature review (1998–2009) regarding the same adhesive tested by different laboratories and by different tests, one should consider other testing approaches [10]. The main question is what is the final goal of measuring bond strength? If we are interested in evaluating bonding differences among products, or overall degradation susceptibility with time we should move to fracture mechanics approaches and use the potential power of stable crack propagation within an interface using either; (1) $K_{Ic}$, the fracture toughness, which is the material's resistance to crack propagation [177–181] or, (2) its related sister, $G_{Ic}$, the strain energy release rate or work to separate the adhesive resin from its bond to dentin [182,183]. The concept is to initiate and propagate in a *stable manner* a crack through the bonded interface using either the chevron notch short rod or bar design [178,179,181–183], or a modification of the chevron notched short rod known as the Notchless Triangular Prism [177,184], or the single edge notched beam [180].

Table 6 summarizes reports of interfacial $K_{Ic}$ or $G_{Ic}$ for some adhesives bonded to dentin [177–183]. Regardless of which test method was used, all authors reported true interfacial failure with minimal cohesive fractures in dentin or resin, thus testing the adhesive's ability to resist crack propagation or peeling resistance from the substrate.

In our view, the interfacial fracture mechanics approach should gain more recognition among the "dentin bonding community" and be promoted as it has been for other bi-materials interfaces such as the ceramic–metal [185–189], the ceramic–resin [190,191] or the resin–metal interface [191–193]. Miniaturization of chevron notches [177–179,183,190] can be readily used on human dentin as these are small sized specimens. Larger specimens in beam shape for the bi-materials interface energy release calculations [185–190] can be obtained from using bovine dentin.

It would be very interesting to see comparisons from fracture mechanics tests of the interfacial bond for three-step, two-step and one-step adhesives to dentin from various research groups and correlate the results with in vivo findings.

## 4. Conclusions and perspectives

This literature review compiling and comparing bond strength data from 1998 to 2009 for six adhesives bonded to dentin using four tests has pointed out that:

(a) despite similar sample preparation description and testing, the scatter in bond strength is present regardless of which test and which adhesive has been used.

(b) the high scatter and coefficients of variation are due in part to the inclusion of cohesive failures in dentin and composite as well as pre-testing failures into the statistical analyses.

(c) the modes of failure as described in the reviewed literature are often only evaluated with low power microscope magnification which add to the errors of interpretation of the materials involved at the fractured surface and to the distinction of failure modes.

(d) the reported "mixed" failure modes are often not describing the percentage of cohesive failure and within which material (dentin, adhesive resin or restorative composite). A high percentage of cohesive failure (in dentin and composite) will have an impact on the scatter of the bond strength data.

(e) the statistical analysis most often includes all broken specimens whether they are cohesively, adhesively or pre-test failed, which adds to the scatter of the bond strength data.

It seems that as a first step, there is a real need for a consensus regarding a thorough screening to determine which specimen should be used before and after testing (i.e. discard predamaged specimens, discard fractures that are mainly cohesive), which statistical analysis to apply and how to describe failure modes. In addition, the researcher has to be aware of the known problems arising from non-uniform tensile or shear stress states at the interface due to variation in the specimens' geometries, loading configuration and modulus of elasticity of the restorative resins as well as the existence of various strength-controlling flaws.

In light of the above remarks and findings of this literature review, a few recommendations can be suggested.

*Recommendation 1:* If traditional bond strength tests (shear, microshear, tensile, microtensile) are to be used in full knowledge of the difficulties of interpretation of the bonding performance of adhesion to dentin, all broken specimens that show cohesive failure in dentin or resin composite should be discarded as these data are not representative of an interface bond strength, but rather reflect a mixture of mechanical properties of the different materials involved (i.e. dentin, restorative resin). Only adhesive failures or mixed failures with small (<10%) resin or dentin involvement should be considered for the bond strength calculation (Fig. 9). This requires thorough microscopic evaluation (stereo and SEM) of the fractured surface.

*Recommendation 2:* The use of Weibull statistics should be systematically applied to evaluate bond strength data to provide more relevant information regarding failure probability as a function of stress level as well as reliability information of the bond. A minimum of 30 non-cohesively failed specimens (see Fig. 9 for rejection criteria) should be made available. Improvement will come from higher Weibull $m$ values when several adhesives or different surface preparations are involved. So, for the sake of showing bond strength degradation by introducing an aging or a fatigue variable to the study, it is critical to have a significant amount of valid baseline specimens ($\geq$30) and to thoroughly evaluate the Weibull scale ($\sigma_0$) and shape ($m$) parameters.

*Recommendation 3:* The authors would like to encourage the adhesion community to move to a more fracture mechanics approach when evaluating the adhesive bonded interface. Fracture toughness ($K_{Ic}$) or the strain energy release rates ($G_{Ic}$) are tests that are considered more meaningful to measure the energy or work to separate the adhesive resin from its bond to dentin.

The findings of this literature review emphasize the need to approach bond strength tests with the awareness of some current deficiencies and then strive to eliminate these in the future.

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