Evaluation of bond strength of different adhesive systems: Shear and Microtensile Bond Strength Test

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SUMMARY

Evaluation of bond strength of different adhesive systems: Shear and Microtensile Bond Strength Test.

Objectives. Aim of this work is the in vitro bond strength evaluation of three bonding agents comparing the results of two kinds of test, Microtensile Bond Strength Test and a Shear Bond Strength Test. Bond strength tests have been used to test both direct and indirect restorative techniques to investigate if methods could give different results.

Methods. 72 human third molars have been collected and stored in physiological solution. Three kinds of test were conducted: 1- SB, 2- "Slice" preparation μTBS1, 3- "Stick" preparation μTBS2. We tested three different adhesive systems (Groups 1-2-3 n=24), two restorative techniques (subgroup A-B n=12). The tested adhesives were: Optibond FL (OFL) (Group 1), Optibond Solo Plus (OSP) (Group 2), Optibond Solo Plus Self-Etch (OSSE) (Group 3). For all tests was used a universal load machine Instron Machine.

Results. Best values were found for Optibond FL with mean values of 45-50 MPa. Optibond Solo Plus resulted in values very similar and in some cases almost identical to FL. Optibond Solo Self Etch showed poorer adhesion in both direct and indirect restorative techniques. The parametric and non parametric statistical variance analysis pointed out the absence of significant differences between OFL and OSP, and demonstrated a significant difference for OSSE adhesive.

Significance. The results confirm that a total etch two-step adhesive is the best compromise between easiness and effectiveness.

Key words: dentin-bonding agents, Microtensile Bond Strength Test, Shear Bond Strength Test, direct restorations, indirect restorations.

RIASSUNTO

Valutazione della forza di adesione di sistemi adesivi: Shear e Microtensile Bond Strength Test.

Obiettivi. Lo scopo di questo lavoro è la valutazione in vitro della forza di adesione di tre sistemi adesivi mettendo a confronto i risultati di due tipi di test, un Microtensile Bond Strength Test e uno Shear Bond Strength Test. Entrambe le metodiche sono state utilizzate per testare sia restauri diretti che indiretti per evidenziare se la metodica di restauro possa influire sul risultato.

Materiali e Metodi. Sono stati raccolti 72 terzi molari umani estratti per motivi chirurgici e stoccati in soluzione fisiologica. Sono stati realizzati tre tipi di test: 1- SB, 2- "Slice" preparation μTBS1, 3- "Stick" preparation μTBS2. Sono stati sottoposti a test tre sistemi adesivi (Gruppi 1-2-3 n=24), utilizzati con due tecniche operative (sottogruppi A-B n=12). Gli adesivi testate sono: Optibond FL (OFL) (Gruppo 1), Optibond Solo Plus (OSP) (Gruppo 2) e Optibond Solo Plus Self-Etch (OSSE) (Gruppo 3). Tutti i test sono stati eseguiti mediante una macchina di carico universale Instron Machine.

Risultati. I migliori valori sono stati ottenuti dall'Optibond FL con valori medi di 45-50 MPa. L'Optibond Solo Plus ha prodotto risultati molto simili ed in alcuni casi sovrapponibili all'FL. L'Optibond Solo Self Etch si è dimostrato il peggiore dei tre adesivi testati sia nella tecnica diretta che indiretta. L'analisi statistica parametrica e non parametrica della varianza ha dimostrato l'assenza di differenze significative tra OFL and OSP e ha inoltre indicato una differenza significativa nei dati ottenuti con l'adesivo OSSE.

Conclusioni. I risultati ottenuti confermano che un adesivo *total etch* a due passaggi rappresenta il miglior compromesso tra semplicità d'uso e efficacia.

Parole chiave: adesivi smalto dentinali, Microtensile Bond Strength Test, Shear Bond Strength Test, restauri diretti, restauri indiretti.

Introduction

The Shear Bond Strength Test has represented for a long time the test of election to evaluate the adhesion strength of adhesive systems on enamel and dentin (1, 2). This test is still used in dental research for its simple execution. Recently the literature has doubted the validity of this test, though it is negatively influenced by several variables related to the dentin substrate (3, 4) such as: etching quality, substrate humidity level, quality of the adhesive bonds (operator variability), experimental tensile conditions (5, 6). According to the Griffith theory (7), a small area is statistically associated with higher tensile values while a large area with lower tensile forces; therefore has been suggested the microtensile bond strength test. The Microtensile Test (μTBS) is considered at the time the most valuable test for the evaluation of the adhesion bond strength (8, 9). Actually this test has some limitations, as the starting sample needs to be reduced to minimal specimen. The Microtensile test allows a more uniform stress distribution than the shear bond strength test, due to an axial tensile loading on a reduced interface, thus reducing the frequency of cohesive fractures in the dentin (10). Furthermore, the tested sticks of every single sample allow to evaluate the mean and the standard deviation of total bond strength. In any case, this test is highly influenced by procedures and preparation of the samples for testing. A side aim of this study is to verify if the μTBS is an accurate and reliable evaluation technique of bonds strength and if possible to determine the influence of sample preparations on the performance of the adhesive systems. In order to create a complete test, simulating different clinical conditions, tests were conducted with three adhesive systems under direct and indirect experimental restorations.
The aim of this study is not only to evaluate the bond strength of adhesive systems but also to compare the validity of different tensile tests.

Materials and methods

For this study we selected seventy-two superior and inferior third molars, extracted for surgical reasons, caries-free, cleaned and stored in physiological solutions at room temperature. All teeth were randomly divided into groups and subgroups according to: tested adhesive, restorative technique and tensile test.

Group 1: 3 step adhesive Optibond FL (KerrHawe Scafati Italy) n = 24
- Subgroup A: Direct restorations n = 12
  - Sub- Subgroup 1: Shear Bond (SB) n = 4
  - Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4
  - Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4
- Subgroup B: Indirect restorations n = 12
  - Sub- Subgroup 1: Shear Bond (SB) n = 4
  - Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4
  - Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4

Group 2: 2 step adhesive Optibond Solo Plus (KerrHawe Scafati Italy) n = 24
- Subgroup A: Direct restorations n = 12
  - Sub- Subgroup 1: Shear Bond (SB) n = 4
  - Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4
  - Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4
- Subgroup B: Indirect restorations n = 12
  - Sub- Subgroup 1: Shear Bond Stregth test (SB) n = 4
  - Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4
  - Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4

Group 3: 2 step adhesive Optibond Solo Plus Self Etch (KerrHawe Scafati Italy) n = 24
- Subgroup A: Direct restorations n = 12
  - Sub- Subgroup 1: Shear Bond (SB) n = 4
  - Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4
  - Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4
- Subgroup B: Indirect restorations n = 12
  - Sub- Subgroup 1: Shear Bond (SB) n = 4

Sub- Subgroup 2: Microtensile "Slice" (μTBS^1) n = 4

Sub- Subgroup 3: Microtensile "Stick" (μTBS^2) n = 4

Teeth for test were fixed in aluminium boxes filled with Type 3 gypsum, dimensions 3.6 cm x 3.6 cm x 3 cm. All teeth were fixed in the cast up to the DEJ with major axis perpendicular to the base in order to simplify the correct alignment during cutting phases. For each tested adhesive we simulated both direct and indirect restorative techniques. Occlusal surfaces of the prepared teeth were cut horizontally using a precision miller mounted diamond disk to obtain the exposure of all occlusal dentin. To minimize the number of variables for the subsequent tests, during every step of teeth preparation, any sample showing abnormalities or macroscopic non-homogeneities were excluded. With the described set up of the samples, it was possible to obtain a dentinal surface, which is absolutely parallel to the base of the mounting box. After the first cutting step, the exposed dentin surface was polished with sand paper 1000 grit to simulate a realistic smear layer (Fig. 1) . To create the direct and indirect experimental restorations a silicone mould was prepared (Fig. 2). This was to create the same sample final shape for both kind of restorations; final restoration had dimensions of 5,4 mm x 5,4 mm x 5,4 mm. (Microhybrid restorative composite used was Point 4 shade A3 KerrHawe Scafati Italy). All materials were used strictly according to manufacturer indications. To initiate the polymerization of adhesives and resin composite, an halogen lamp curing unit Coltolux 75 (KerrHawe Scafati Italy). For the luting of all indirect restorations, was performed a "Polymerization under pressure technique" (11) using the same photo-curing restorative composite as cement. Luting load pressure generated by an hydraulic press was 1.5bar (measured equal to the pressure generated manually by a mean operator) and was maintained till the end of light curing time. To cut the restored teeth for the tensile tests, was used a hard tissues and bone micrometrical controlled microtome (Leika) with a toroidal diamond saw.

Samples for SB test did not undergo sectioning and were immediately ready for load machine (Fig. 3). For μTBS^1 (Slice preparation) three axial cuts were realised in order to obtain four samples for each tooth (Fig. 4). Each tested sample was then characterised by an adhesive surface of approximately 5,4 mm x 1,2 mm and was individually measured with a digital precision calliper before traction. For μTBS^2 (Stick preparation) were realised five

Figure 1
Tooth fixed into aluminium box filled with gypsum up to DEJ. Exposure of the dentinal surface to be prepared for adhesion procedures.

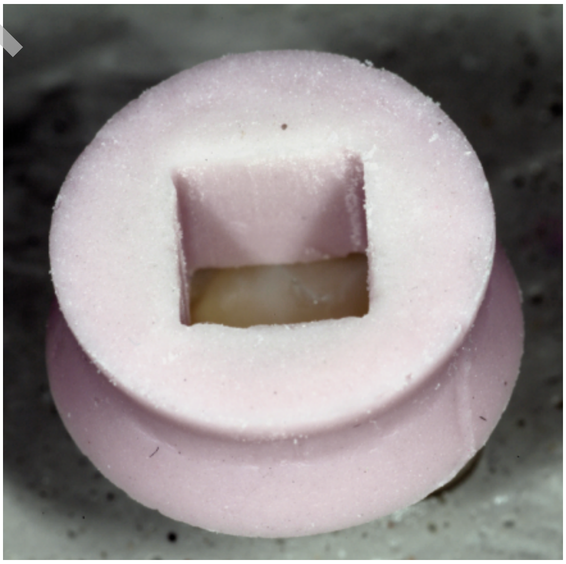

Figure 2
Putty mold in place for the building of both direct and indirect simulated restorations.

Figure 3
Sample ready for SB test or for cutting procedures for microtensile tests.

axial cuts followed by other five cuts to 90° rotation in order to obtain 16 samples for each tooth (Fig. 5). The adhesive surface expected for these samples was approximately 1,2 mm x 1,2 mm and was individually measured with a digital precision calliper before traction. For the tensile test was used a universal testing machine (Instron Machine) with the following settings:

- SB maximum load 500 Kg; speed: 0,5 mm/min.
- μTBS[1] maximum load 150 Kg; speed: 0,5 mm/min.
- μTBS[2] maximum load 120 Kg; speed: 0,5 mm/min.

The collected data were statistically analysed with parametric and nonparametric tests of the variance and of the median values using SPSS software.

Results

All data collected from tensile testing are reported in Table 1 (for direct restorations) and in Table 2 (for indirect restorations) as mean adhesion bond strength and Standard Deviation. The samples that fractured during preparation procedures (especially in Microtensile testing) or that presented cohesive fractures were excluded from the statistical analysis. As some of the samples were excluded for premature failing, in the tables are reported the number of tested samples over the number of prepared samples.

The parametric statistical analysis of the variance showed the absence of statistically significant differences between tests and the lack of interactions between the tests variables and the adhesives.

The Kruskal-Wallis non parametric test confirmed the absence of statistically significant differences between the test groups of the single adhesives. A Post Hoc Test with Tukey HSD ($p<0.5$) showed that the adhesive OSSE (self etching primer) differs significantly from the other two systems that don't present significant statistical differences.

The T-test ($p<0.5$) was used to compare the direct and the indirect techniques: for the OFL adhesive the difference between the mean and the variance is significant; for the OSP adhesive the difference between the mean and the variance is significant; for the OSSE adhesive the difference between mean values is not significant while the difference between the variances are border-line.

The analysis of the results obtained from the direct and indirect techniques for each adhesive system analysed separately with the non-parametric Mann-Whitney test highlighted statistically significant differences

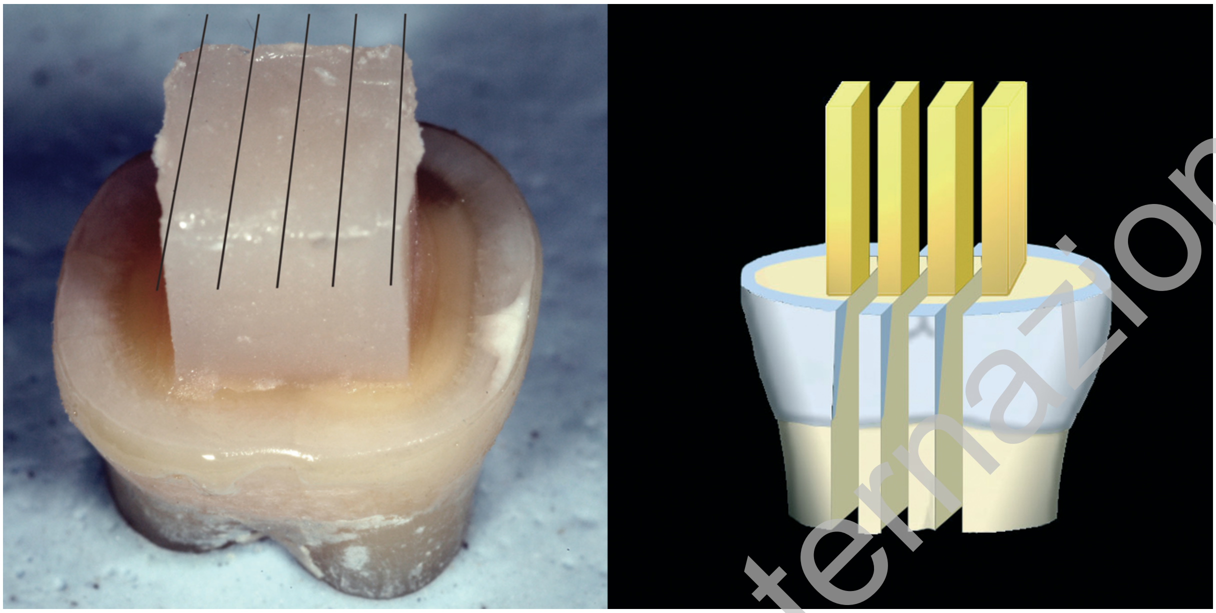

Figure 4
Cutting scheme for µTBS[1].

Figure 5
Cutting scheme for µTBS[2].

between the two techniques for the adhesives OFL and OSP, while it confirmed the not significativity of the variances for the OSSE.
Comparing the two restorative techniques with the T-Test without considering the single adhesives, we found a significant statistical difference for both variance and means. Within the same technique, the comparison of the self etching adhesive with the etch and rinse showed highly significant differences for both variance and means.

Table 1 - Bond Strength mean values expressed in MPa and Standard Deviation (DS) resulted by tests on simulated direct restorations. Number of samples reports the real number of tested specimens out the total.

Adhesive	Test	Samples	Mean (MPa)	SD
Optibond FL	SB	4/4	41.90	7.63
	μTBS¹	15/16	52.21	15.66
	μTBS²	48/64	45.59	17.35
Optibond Solo Plus	SB	4/4	31.79	6.82
	μTBS¹	15/16	45.26	12.70
	μTBS²	45/64	43.69	15.66
Optibond Solo Plus Self Etch	SB	4/4	17.93	5.04
	μTBS¹	14/16	15.35	5.63
	μTBS²	38/64	14.25	5.47

(exclusions of samples were due to premature fail or other adverse condition). SB: test shear bond stregth test; μTBS¹: microtensile bond strength test with "slice" preparation; μTBS²: microtensile bond strength test with "stick" preparation.

Table 2 - Bond Strength mean values expressed in MPa and Standard Deviation (DS) resulted by tests on simulated indirect restorations. Number of samples reports the real number of tested specimens out the total.

Adhesive	Test	Samples	Mean (MPa)	SD
Optibond FL	SB	4/4	37.96	3.22
	μTBS¹	15/16	42.69	10.04
	μTBS²	55/64	38.70	13.44
Optibond Solo Plus	SB	4/4	31.68	5.93
	μTBS¹	12/16	37.85	10.12
	μTBS²	51/64	36.61	11.91
Optibond Solo Plus Self Etch	SB	4/4	18.61	5.84
	μTBS¹	6/16	15.37	4.76
	μTBS²	45/64	13.36	4.63

(exclusions of samples were due to premature fail or other adverse condition). SB: test shear bond stregth test; μTBS¹: microtensile bond strength test with "slice" preparation; μTBS²: microtensile bond strength test with "stick" preparation.

Discussion

The analyzed data suggest that in general all tested etch and rinse adhesives had a better behaviour than the tested self etching one. The two etch and rinse adhesives gave similar results in terms of mean values but, probably due to the complexities of the technique, Optibond FL resulted in higher standard deviation values.

From the comparison of the results of direct restorative technique with the indirect technique, the data showed a better performance of the adhesion in direct technique. This result is probably due to a relatively less degree of conversion of the polymerized composite and adhesives in the indirect system during cementing phases.

The comparison of all tests gives the opportunity to draw up some considerations:

1. Shear Bond (SB): the simplicity of the test seams not a good reason to chose this test as the only way to evaluate the bond strength. For the number of measurements per sample and for the difference with values obtained from more sophisticated tests, SB seam not able to produce accurate results.

2. Microtensile Bond Strength Test (μTBS[1] e μTBS[2]): agreeing with literature, these tests on small size samples are more precise but can be highly affected by adverse events such as premature fails and need a larger number of samples (12-15).
3. Only to compare within the test the efficacy of different adhesives, and not to measure the exact individual bond strength of the adhesive, SB seams reliable due to ease of use. More sophisticated microtensile tests seam more oriented to find out maximum and minimal local adhesion of the specific tested adhesive on a specific substrate.

The statistical analysis showed a better behaviour of the "etch and rinse" adhesives rather than the tested self etch adhesive. Moreover, the statistical analysis suggested that the adhesive interface resulting in the direct restoration has a better behaviour than in the indirect restoration.

With the limitations of a test in which many variables were not included, such as, for example, the aging of the samples (16), this study, without pointing out the single values, gives us the possibility to compare different adhesive's performances in simulated clinical conditions. On the basis of collected data and among tested adhesives, the V generation adhesive (Optibond Solo Plus) confirmed the best characteristics between opertaing difficulties and the gained adhesion in both direct and indirect method.

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