

MARGINAL ADAPTATION OF CONTEMPORARY DENTIN BONDING AGENTS IN ENAMEL AND DENTIN UNDER THE SIMULATION OF DENTINAL FLUID.

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Abstract

Purpose: To compare the *in vitro* marginal adaptation of contemporary adhesive and restorative systems in mixed Class V restorations. Both etch & rinse and self-etch adhesives were evaluated before and after long-term thermal and mechanical loading under the simulation of dentinal fluid.

Materials and methods: Mixed class V cavities were prepared on buccal and lingual surfaces of extracted human molars and restored using sixteen adhesive systems with their corresponding composites. Scotchbond Multipurpose Plus and iBond were additionally tested in another two experimental groups by increasing the aggressiveness of enamel etching. Marginal adaptations were evaluated using computer assisted quantitative marginal analysis in a scanning electron microscope (SEM) on epoxy replicas of the restorations both before and after loading challenge. The percentages of "continuous margin" for the total marginal length ranged from 97.5 ± 2.9 to 56.2 ± 16.2 before, and from 91.2 ± 3.7 to 31.4 ± 16.6 after loading. The modified application procedure did not improve marginal adaptation for both Scotchbond Multipurpose Plus and iBond after loading.

Conclusion: None of the adhesive systems tested was able to eliminate marginal gap formation in enamel and dentin. The adhesion strategy (etch & rinse or self-etch) and the number of application steps may not be the only parameters to consider in marginal adaptation. Other factors, more related to the compositions of the adhesive and probably composite, may also be important.

Keywords: marginal adaptation, artificial aging, adhesive systems, enamel, dentin

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Introduction

Resin composites are commonly used materials in restorative dentistry ^{1,2}. Since they are not able to directly bond to the tooth substrates, adhesive systems are necessary to promote adhesion between the filling material and the tooth. Commercially available adhesive systems generally involve 1, 2 or 3 application steps ³. If an etch & rinse approach is followed, phosphoric acid may be used as the conditioning agent with selective application times for enamel and dentin, followed by water spray rinsing and blot drying as described in the process of wet bonding ⁴. If a self-etch method is used, acidic monomer-containing solutions, (so-called self etching primers), are applied simultaneously to enamel and dentin before the application of the bonding resin ⁵. In an attempt to simplify the clinical procedure and application steps, single component systems have been developed. With these, either the etching, priming and bonding components are contained in one bottle or solutions supplied in separate containers may be pre-mixed prior to a single or repeated application.

The selection of the appropriate adhesive system and composite material for a restoration is a difficult task for the practitioner faced with a diversity of available materials. Certainly, the most conclusive information regarding any material would be provided by its clinical performance over a significant period of time ⁶. Unfortunately there are inherent difficulties with clinical studies. These are related firstly to the oral cavity being an aggressive environment where several stressing factors act simultaneously, often making it difficult to define the determinant factor/s leading to failure ⁷. Secondly, long evaluation periods are required to test the products conclusively. Furthermore, once these findings have been satisfactorily analysed, there is often a risk that the product has already been replaced by a new material with properties claimed to be superior to those of its predecessor. For these reasons *in vitro* tests are often used as they provide a rapid and workable method prior to the clinical evaluation of a given material with the advantage of evaluating the effects of different variables separately.

A stress-resistant adhesion between tooth and restoration is crucial in the planning of a reasonably long prognosis for an adhesive dental restoration in the mouth ⁸. Non-bonded areas or defects between restoration and tooth substance are susceptible to microleakage, marginal discolouration, postoperative sensitivity and secondary caries ⁹. Secondary caries together with marginal discolouration have been described as the most common reasons for the replacement of

Table 1. Description of the experimental groups.

| Class of adhesive | Group Adhesive system Filling composite | Manufacturer | Batch numbers |
|--------------------------------------|---|---|--|
| Etch & rinse (3 components, 3 steps) | Scotchbond MP Plus Filtek supreme | 3M ESPE AG, D-82229 Seefeld, Germany | H ₃ PO ₄ : 3BC Primer : 3AH Adhesive : 3NJ Composite: Shade A2B , 3BR |
| Etch & rinse (2 components, 2 steps) | Admira Bond Grandio | VOCO GmbH 27457 Cuxhaven, Germany | H ₃ PO ₄ : 371086 Adhesive: 351598 Composite: Shade A3, 391267 |
| | James 2 ELS | Saremco AG CH-9445 Rebstein, Germany | H ₃ PO ₄ :01.2008 – 41, Adhesive: 01.2006 – 20 Composite: Shade A2, 08.2007 – 05 |
| Self etch (3 components, 2 steps) | Fl-bond Beautiful | SHOFU Dental GmbH 40878 Ratingen, Germany | Primer A : 050383 Primer B : 050305 Bond : 050302 Composite: Shade A2, 080373 |
| | ART Bond Miris | Coltène AG 9450 Altstätten/Switzerland | Primer A: LH 728 Primer B : LH 728 Bond : LH 728 Composite: Shade S3 LI787 |
| | Tyrian SPE Micronew | Bisco, Inc. IL 60193, U.S.A | Primer : 0300006274 Bond : 0300006240 Composite: Shade Tr.A2, 0400000961 |
| Self etch (2 components, 2 steps) | Contax Ecusit | DMG Chemisch-Pharmazeutische Fabrik GmbH 22547 Hamburg, Germany | Primer : 506515 Bond : 502308 Composite: Shade A2, 511778 |
| | Exp. Bond Coltène Miris | Coltène AG 9450 Altstätten/Switzerland | Primer exp_04 : 240902P1 Bond exp_04 : MF02 Composite: Shade S3, LI787 |
| | Optibond Solo Plus Herculite | Kerr GmbH D-76185 Karlsruhe, Germany | Primer : 208670 Bond : 212017 Composite: Shade A2, 3-1287 |
| | Nanobond Simile | Pentron P.O. Box 724 Wallingford, CT U.S.A. 06492 | Primer : 96193 Bond : 91694 Composite: Shade A2 95668 |
| | Clearfil SE Bond Clearfil AP-X | Kuraray Medical Inc. Okayama 710-8622, Japan | Primer : 00442B Bond : 00661A Composite: Shade A2, 00746B |
| | One Coat SE Bond Miris | Coltène AG 9450 Altstätten/Switzerland | Primer : 110902 Bond : 120902 Composite: Shade S3, LI787 |
| Self etch (2 components, 1 step) | Exp.bond VoCo Grandio | VoCo GmbH 27457 Cuxhaven, Germany | Primer A : V25273 Primer B : V25487 Composite : Shade A3, 391267 |
| | Xeno III Ceram X Duo | Dentsply De Trey GmbH 78467 Konstanz, Germany | A : 0310000129 B : 0310000129 Composite: Enamel Shade E2, 0309000482 , Dentin Shade D2, 0309000484 |
| | Hybrid Bond Metafil CX | Sun Medical CO.,LTD. | Hybrid Base GV2\ Hybrid Brushes GG1 Composite : Shade A2, GV1 |
| Self etch (1 component, 1 step) | iBond Venus | Heraeus Kulzer GmbH, D-61273 & Co., Wehrheim, Germany | Single dose: 010046 Composite: Shade A2, 030023 |

adhesive restorations¹⁰. Therefore, assessing the marginal adaptation of a restorative material is an important parameter to be tested when predicting its long-term behaviour^{3,11}.

It has been suggested that dental restorations clinically fail as a result of fatigue or cumulative damage^{12,13}. Fatigue is an inevitable process that occurs in every material in the mouth due to the constant stresses that are generated by masticatory forces. Consequently, the *in vitro* evaluation of marginal adaptation of restorative systems under fatigue loading may be an important factor to be considered when evaluating its clinical potential. If clinical conditions can be mimicked as

closely as possible in the *in vitro* evaluation, i.e. by the simulation of the dentinal fluid^{14,15,16}, by the use of natural antagonistic cusps, by reproduction of the 3-dimensional tooth cavity¹⁷, by repeated thermal cycling and by cyclic sub-critical occlusal mechanical loads applied during a pre-determined period of time^{18,19,20,21}, then the *in vitro* test may be considered to be clinically relevant^{3,22,23}.

The purpose of this study was to evaluate the marginal adaptation potential of sixteen contemporary adhesive systems together with their “manufacturer-recommended” composites in the enamel and dentin margins of mixed class V restorations

TABLES 2A AND 2B.

Percentages of continuous margin at the total margin length (enamel and dentin together) before and after thermal and mechanical loading. Significant differences between initial and terminal values for each group are represented with an * (Wilcoxon signed-rank test $p < 0.05$). Differences among groups were statistically evaluated with Kruskal-Wallis and Bonferroni's test ($p < 0.05$). Groups not connected by the same letter are significantly different

| 2A. TOTAL MARGIN LENGTH BEFORE LOADING. | | | | 2B. TOTAL MARGIN LENGTH AFTER LOADING | | | |
|---|-------|-------------|--|---------------------------------------|---------|---------------|--|
| GROUPS | | MEAN ± SD | | GROUPS | | MEAN ± SD | |
| ART Bond | A | 97.5 ± 2.9 | | ART Bond | A | 91.2 ± 3.7 * | |
| One Coat SE Bond | A | 97.4 ± 0.9 | | One Coat SE Bond | A B | 83.2 ± 9.3 * | |
| Nanobond | A B | 94.1 ± 1.7 | | Clearfil SE Bond | A B C | 4.9 ± 10.7 * | |
| FL-Bond | A B C | 92.1 ± 5.9 | | Coltène Exp. | A B C D | 69.0 ± 6.8 * | |
| Admira Bond | A B C | 90.3 ± 5.4 | | Nanobond | A B C D | 65.9 ± 9.8 * | |
| Coltène Exp. | A B C | 84.0 ± 11.3 | | FL-Bond | A B C D | 65.7 ± 14.4 * | |
| Hybrid Bond | A B C | 82.6 ± 7.9 | | VoCo Exp. | B C D | 61.6 ± 23.2 * | |
| Clearfil SE Bond | A B C | 82.0 ± 7.0 | | Admira Bond | B C D | 61.6 ± 16.7 * | |
| Xeno III | A B C | 78.8 ± 11.6 | | Xeno III | B C D E | 56.2 ± 10.6 * | |
| James 2 | A B C | 78.6 ± 12.3 | | Scotchbond MP Plus | B C D E | 55.9 ± 12.1 * | |
| VoCo Exp | A B C | 78.2 ± 16.7 | | iBond | B C D E | 55.2 ± 11.0 * | |
| Scotchbond MP Plus | B C D | 73.6 ± 13.9 | | Contax | C D E | 51.8 ± 15.7 * | |
| Contax | C D | 72.5 ± 14.1 | | Hybrid Bond | C D E | 49.2 ± 16.2 * | |
| iBond | C D | 64.8 ± 8.1 | | Optibond Solo Plus | D E | 43.3 ± 4.2 * | |
| Tyrian SPE | C D | 64.7 ± 5.3 | | Tyrian SPE | D E | 41.8 ± 10.2 * | |
| Optibond Solo Plus | D | 56.2 ± 17.1 | | James 2 | E | 31.4 ± 16.6 * | |

in extracted human molars. Evaluation was performed using the quantitative replica evaluation method with a scanning electron microscope (SEM) immediately after placement of the restorations, and after simultaneous thermal and mechanical loading under the influence of simulated dentinal fluid. The null hypotheses tested were: 1) that no difference exists between the marginal adaptation of self-etch and etch & rinse adhesive systems and 2) that there are no differences in the ability of these adhesive-composite systems to perfectly seal both enamel and dentinal margins.

Materials & Methods

Preparation of test teeth

Fifty four caries-free human upper molar teeth with completed root formation were stored in 0.1% thymol solution prior to the experiment. After scaling and pumicing, the teeth were randomly assigned to eighteen experimental groups (sixteen groups in which the manufacturers' instructions were strictly followed and two additional groups where the application procedure was modified) and mounted on custom made specimen holders with their roots centrally placed using cold-polymerizing resin (Technovit 4071, Heraeus Kulzer GmbH, Wehrheim, Germany).

The root apices of each tooth were sealed with two coats of nail varnish prior to the mounting procedure. A cylindrical hole was drilled into the pulpal chamber approximately in the middle third of the palatal root and a metal tube with a diameter of 1.4 mm was then inserted using a dentinal adhesive (Syntac Classic, IvoclarVivadent AG, Schaan, Liechtenstein). The pulpal tissue was not removed. This tube was connected by a flexible silicone hose to an infusion bottle placed 34 cm vertically above the test tooth. The infusion

bottle was filled with horse serum diluted in a 1:3 ratio with 0.9 % NaCl^{15, 16} in order to simulate dentinal fluid under normal hydrostatic pressure of approximately 25 mm Hg^{14, 24}. Twenty-four hours before starting the cavity preparations the pulp chambers were evacuated with a vacuum pump and subsequently filled with bubble-free, diluted horse serum. The intra-pulpal pressure was subsequently maintained at 25 mm Hg throughout the testing, i.e. during cavity preparation, restoration placement, finishing and stressing.

Cavity preparation

One V-shaped standardized Class V cavity was prepared on the buccal and one was prepared on the lingual surface of each tooth with half of the margin located in enamel and half in dentin. Tooth preparation was performed using 80 µm diamond burs (Diatech Dental, Coltène-Whaledent, Altstätten, Switzerland) with continuous water coolant. Each bur was replaced after four dental preparations. The dimensions of the V-shaped cavities were 3.0 - 3.5 mm (length) by 2.5 - 3.0 mm (height) and 1.5 mm depth. The margin in enamel was bevelled to a crescent-shape with a maximum width of 1.2 mm. The entire cavity was finished using 15 µm finishing diamond burs (Diatech Dental, Coltène-Whaledent, Altstätten, Switzerland). Finally, the cavity preparations were checked for marginal imperfections such as fractures or chipping with a stereo microscope (Wild M5, Wild AG, Heerbrugg, Switzerland) at 12x magnification and corrected wherever necessary.

According to the methodology established in a previous similar study²⁵, three teeth with two Class V cavities each were randomly assigned to each of the experimental groups.

TABLES 3A AND 3B.

Percentages of continuous margin at the **enamel** margins before and after thermal and mechanical loading. Significant differences between initial and terminal values for each group are represented with an * (Wilcoxon signed-rank test $p < 0.05$). Differences among groups were statistically evaluated with Kruskal-Wallis and Bonferroni's test ($p < 0.05$). Groups not connected by the same letter are significantly different.

| 3A. ENAMEL MARGINS BEFORE LOADING. | | | | 3B. ENAMEL MARGINS AFTER LOADING. | | | |
|---|---------|------------------|--|--|---------------|------------------|---|
| GROUPS | | MEAN ± SD | | GROUPS | | MEAN ± SD | |
| Admira Bond | A | 96.6 ± 1.2 | | Admira Bond | A | 88.7 ± 6.1 | * |
| One Coat SE Bond | A | 96.2 ± 2.2 | | One Coat SE Bond | A B | 85.4 ± 9.7 | * |
| ART Bond | A | 96.2 ± 3.4 | | Xeno III | A B | 82.9 ± 7.2 | * |
| James 2 | A | 95.6 ± 4.9 | | ART Bond | A B | 82.1 ± 7.4 | * |
| Nanobond | A B | 92.2 ± 3.4 | | Coltène Exp. | A B C D | 70.8 ± 7.5 | * |
| Xeno III | A B C | 88.9 ± 4.6 | | Contax | A B C D E | 65.2 ± 12.6 | * |
| Contax | A B C | 87.4 ± 4.9 | | Clearfil SE Bond | A B C D E | 64.7 ± 16.0 | * |
| FL-Bond | A B C | 85.3 ± 11.2 | | James 2 | A B C D E F | 64.3 ± 30.2 | * |
| Coltène Exp. | A B C | 79.5 ± 16.0 | | Tyrian SPE | A B C D E F G | 55.9 ± 10.2 | * |
| Tyrian SPE | A B C | 74.4 ± 10.4 | | Scotchbond MP Plus | B C D E F G | 55.1 ± 21.6 | * |
| Clearfil SE Bond | A B C | 73.0 ± 14.3 | | FL-Bond | B C D E F G | 52.4 ± 16.2 | * |
| VoCo Exp. | B C D | 69.2 ± 18.5 | | Nanobond | B C D E F G | 52.2 ± 11.2 | * |
| Hybrid Bond | B C D E | 68.9 ± 13.9 | | VoCo Exp. | D E F G H | 45.9 ± 29.1 | * |
| Scotchbond MP Plus | C D E | 64.3 ± 19.5 | | Hybrid Bond | G H | 25.9 ± 12.8 | * |
| iBond | D E F | 44.9 ± 16.2 | | iBond | G H | 24.6 ± 17.0 | * |
| Optibond Solo Plus | F | 36.0 ± 19.8 | | Optibond Solo Plus | H | 17.3 ± 8.8 | * |

Placement of the restorations:

In sixteen groups (Table 1), the adhesive systems and their corresponding restorative composites were applied strictly following the manufacturers' instructions. In the other two groups, modified application techniques from the manufacturers' recommendations for Scotchbond Multipurpose Plus and iBond were employed for evaluation. The modification of Scotchbond Multipurpose Plus consisted of extending the enamel etching time with 37% H₃PO₄ to 60 seconds instead of 15 seconds as originally proposed by the manufacturer. In the modified iBond group, the enamel margins were etched with 37% H₃PO₄ for 60 seconds prior to the application of the one-component adhesive system.

After the placement and light-curing of the adhesive with a halogen light-curing unit (Optilux 501, Kerr/Demetron, Danbury, CT, USA) with a constant relative power density

output of 800 mW/cm² (Curing Radiometer Model 100, Serial No. 134089, Demetron Research Corp. Danbury, CT, USA), the composites were inserted into the cavity in two layers. The first layer was placed cervically extending up to one half of the cavity, and the second layer was placed on the occlusal side, thus filling the other half of the cavity. Each layer was light-cured for 40 seconds. Immediately after polymerization, the restorations were finished and polished with flexible aluminium oxide discs of different grain sizes (SofLex PopOn, 3M ESPE AG, Seefeld, Germany). The final polishing was controlled using a stereo microscope under 12x magnification.

Thermal and mechanical stressing

After storage in the dark in a 0.9% saline solution at 37° C for one week, the restored teeth were loaded into a computer-controlled chewing machine^{11, 26, 27}. Thermal and mechanical

TABLES 4A AND 4B.

Percentages of continuous margin at the **dentin** margins before and after thermal and mechanical loading. Significant differences between initial and terminal values for each group are represented with an * (Wilcoxon signed-rank test $p < 0.05$). Differences among groups were statistically evaluated with Kruskal-Wallis and Bonferroni's test ($p < 0.05$). Groups not connected by the same letter are significantly different.

| 4A. DENTIN MARGINS BEFORE LOADING. | | | | 4B. DENTIN MARGINS AFTER LOADING. | | | |
|---|---------|------------------|--|--|-------|------------------|---|
| GROUPS | | MEAN ± SD | | GROUPS | | MEAN ± SD | |
| ART Bond | A | 100.0 ± 0 | | ART Bond | A | 97.8 ± 3.1 | |
| One Coat SE Bond | A | 100.0 ± 0 | | iBond | A B | 92.2 ± 5.0 | |
| Hybrid Bond | A | 100.0 ± 0 | | Clearfil SE Bond | A B | 87.4 ± 14.2 | |
| FL-Bond | A | 99.6 ± 0.9 | | Nanobond | A B | 84.6 ± 11.3 | * |
| iBond | A | 98.2 ± 12.4 | | VoCo exp. | A B | 83.5 ± 12.4 | * |
| Nanobond | A | 96.7 ± 5.4 | | FL-Bond | A B | 81.8 ± 17.6 | |
| Clearfil SE Bond | A | 96.5 ± 3.7 | | One Coat SE Bond | A B | 79.2 ± 25.4 | |
| VoCo Exp. | A B | 91.9 ± 12.9 | | Hybrid Bond | A B | 77.2 ± 18.8 | * |
| Coltène Exp. | A B | 88.9 ± 8.7 | | Optibond Solo Plus | A B | 71.8 ± 24.1 | * |
| Scotchbond MP Plus | A B C | 84.2 ± 16.6 | | Coltène Exp. | A B C | 65.5 ± 11.0 | * |
| Admira Bond | A B C | 82.7 ± 13.0 | | Scotchbond MP Plus | B C D | 54.6 ± 29.9 | * |
| Optibond Solo Plus | A B C | 81.2 ± 21.9 | | Admira Bond | C D E | 27.9 ± 37.4 | * |
| Xeno III | A B C D | 69.2 ± 28.1 | | Xeno III | C D E | 27.5 ± 25.5 | * |
| James 2 | B C D | 63.9 ± 20.1 | | Contax | D E | 21.6 ± 18.5 | |
| Contax | C D | 56.4 ± 28.8 | | Tyrian SPE | D E | 16.2 ± 18.9 | * |
| Tyrian SPE | D | 49.0 ± 24.5 | | James 2 | E | 3.9 ± 7.7 | * |

loading were applied simultaneously²⁸. Thermal cycling was carried out in running water with temperatures changing 3,000 times and ranging from 5°C to 50°C with a cycle time of two minutes. The mechanical stress comprised a total of 1.2 millions load cycles transferred to the center of the occlusal surface with a frequency of 1.7 Hz and a maximal load of 49 N. The load was applied using a natural lingual cusp taken from an extracted human molar tooth. The pulpal pressure was maintained continuously throughout the loading procedure.

Assessment of marginal adaptation

Immediately following completion of the polishing procedure and after loading respectively, impressions were made of each restoration with a polyvinylsiloxane material (President light body, Coltène-Whaledent, Altstätten, Switzerland). Subsequently, gold coated epoxy replicas were prepared for the computer assisted quantitative margin analysis in a scanning electron microscope (XL20, Philips, Eindhoven, Netherlands) at 200x magnification¹¹. The marginal quality, expressed in percentages of “continuous margins”, was reported for the total marginal length, as well as for enamel and dentin margins separately.

Statistical analysis

The statistical analysis was performed using NCSS-PASS (Kaysville, Utah, USA) statistical software. According to the Shapiro-Wilk W-test the scores of marginal adaptation were non-normally distributed. For this reason differences in the percentages of “continuous margins” were tested for significance at the 95% level using the Kruskal-Wallis and Bonferroni tests for the initial and terminal values and the Wilcoxon signed-rank test for the comparison of initial/terminal values within a group.

Results

The total percentages of “continuous margins” (CM) before and after thermal and mechanical loading for the 16 original groups are listed in Tables 2a and b. Before loading, a cluster of eleven materials had high percentages of CM ranging from 97.5 ± 2.9% to 78.2 ± 16.7%, with no significant differences among these groups. After loading, the self-etching material ART Bond showed the best marginal adaptation among all the groups, attaining 91.2 ± 3.7 % of CM. The worst adaptation after loading was observed for the two-component etch & rinse adhesive James 2 (31.4 ± 16.6%).

Tables 3a and b detail the percentages of “continuous margins” (CM) before and after loading at the enamel part

TABLE 5.

Comparison between a three-step etch & rinse (Scotchbond Multipurpose Plus / SMPP) and a single-step self-etch (iBond / iB) bonding agent. Marginal adaptation was evaluated at the total marginal length and at enamel and dentin margins as well, when the products were applied either following the manufacturers instructions (original = o) or when enamel conditioning was performed with 37% phosphoric acid for 60 s (modified = m). Mean ± SD values of continuous margin and are reported. Groups not connected by the same letter are significantly different.

TABLE 5A. TOTAL MARGINAL LENGTH

| Groups | Initial (Mean±SD) |
|--------------|--------------------|
| iB (m) A | 93.3 ± 3.3 |
| SMPP (m) A B | 82.0 ± 11.9 |
| SMPP (o) B C | 73.6 ± 13.9 |
| iB (o) C | 64.8 ± 8.1 |
| Groups | Terminal (Mean±SD) |
| SMPP (m) A | 74.8 ± 11.9 |
| iB (m) A | 56.5 ± 24.2 |
| SMPP (o) A | 55.9 ± 12.1 |
| iB (o) A | 55.2 ± 11.0 |

TABLE 5B. ENAMEL MARGINS

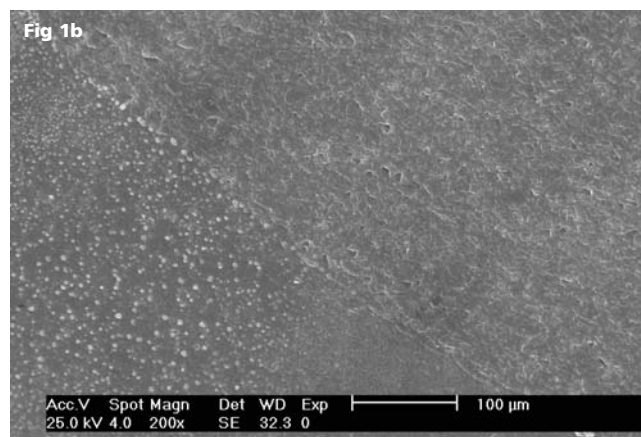
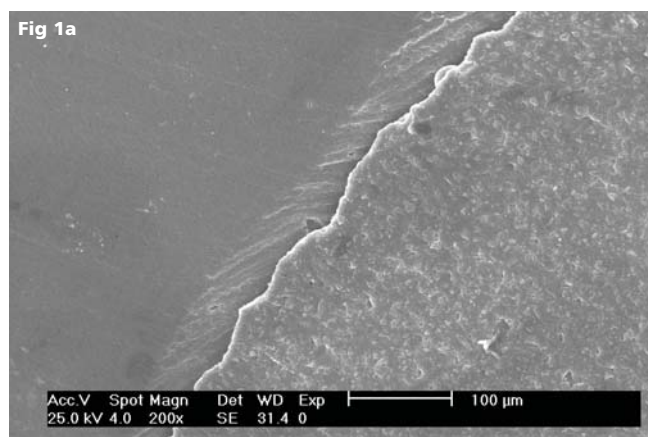
| Groups | Initial (Mean±SD) |
|--------------|--------------------|
| iB (m) A | 88.2 ± 6.9 |
| SMPP (m) A B | 84.6 ± 10.3 |
| SMPP (o) B C | 64.0 ± 19.5 |
| iB (o) C | 44.8 ± 16.2 |
| Groups | Terminal (Mean±SD) |
| SMPP (m) A | 71.3 ± 13.1 |
| SMPP (o) A B | 55.3 ± 21.6 |
| iB (m) A B | 44.7 ± 26.5 |
| iB (o) B | 24.6 ± 17.0 |

TABLE 5C. DENTINAL MARGINS

| Groups | Initial (Mean±SD) |
|------------|--------------------|
| iB (m) A | 98.5 ± 1.5 |
| iB (o) A | 98.2 ± 3.7 |
| SMPP (m) A | 91.6 ± 7.9 |
| SMPP (o) A | 84.1 ± 16.6 |
| Groups | Terminal (Mean±SD) |
| iB (o) A | 92.2 ± 12.0 |
| iB (m) A | 68.2 ± 37.8 |
| SMPP (m) A | 66.1 ± 22.7 |
| SMPP (o) A | 54.6 ± 29.9 |

and Tables 4a and b represent the situation at the dentin part of the margins.

Tables 5a to c represent the comparison of original (o) and modified (m) application techniques for Scotchbond Multipurpose Plus (SMPP) and iBond (iB). Although the modified procedures improved the marginal adaptation of SMPP and iBond in enamel before loading (Table 5b), these improvements were not maintained significantly after loading (p > 0.05). No significant differences (p > 0.05) between the original and the modified procedures were detected for the two materials in dentin, both before and after loading (Table 5c).



Figures 1a and 1b: 1a is a SEM photomicrograph of a “non continuous margin” at the enamel/composite interface after loading (200x magnification). The material shown is iBond applied following manufacturer’s instructions. The defect is located at the enamel/restorative composite interface. 1b is a SEM photomicrograph of a “continuous margin” at the dentin/composite interface after loading (200x magnification). The quality of the marginal adaptation is excellent.

Figures 1 and 2 show some representative SEM micrographs of the marginal qualities observed in the investigation.

Discussion

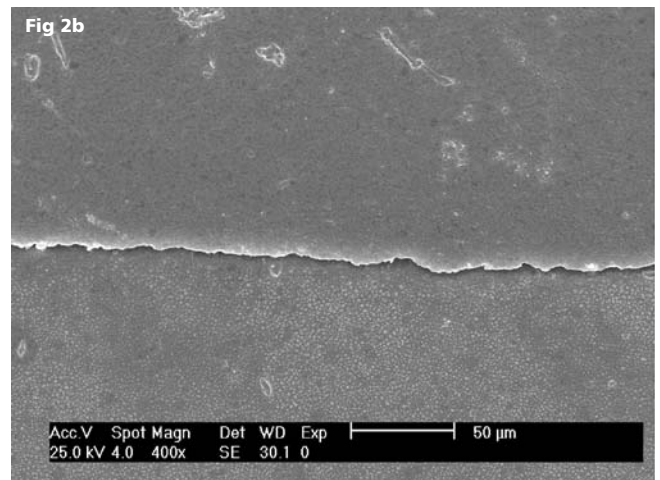
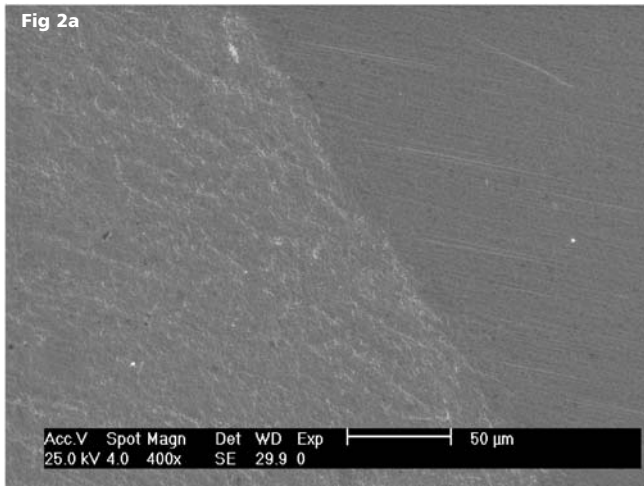
This *in vitro* study evaluated the marginal adaptation of 16 contemporary adhesive systems under the effect of artificial aging as the simulation of oral conditions might be crucial to a better evaluation and understanding of the performance of adhesive materials²⁸. A chewing machine comprising thermocycling and cyclic occlusal mechanical loading together with the simulation of dentinal fluid was used for this purpose^{11, 23, 26}. The rationale for this protocol was based on the work of Arola and Huang²⁹ who found that the combination of thermal changes and occlusal forces comprised almost 95% of the stresses applied to restorations. Moreover, dentinal fluid is an important component to consider in the evaluation of adhesive systems³⁰. *In vitro* studies may reproduce the clinical situation to a certain extent, as a correlation has been demonstrated between *in vitro* and *in vivo* ultrastructural findings and marginal adaptation^{31, 32}.

For the evaluation of the marginal adaptation of the restorations, a computer assisted quantitative SEM margin analysis on the replicas of these restorations was used before and after thermo-mechanical stressing. SEM evaluation based on replicas has several advantages^{33, 34, 35, 36}. These methods may be used for evaluating cavities prepared *in vitro* and *in vivo* as well. The method is also quantitative³⁷, as the presence or absence of leakage may be expressed as percentages of “continuous margin” along the entire tooth/restoration interface. Furthermore, the method is non-destructive, allowing marginal qualities to be assessed before and after exposure to stressing, and it is highly discriminative, allowing the potential of different operative techniques to be quantified in terms of the percentages of “continuous margin”. Therefore, thermo-mechanical fatigue tests and SEM marginal assessments used together could provide relevant information

when *in vivo* behavior of dentin bonding agents is to be predicted on the basis of an accelerated *in vitro* test^{38, 39, 40, 41, 42}.

The majority of the adhesives and the restorative systems evaluated in this study are commercially available. In order to ensure that fresh products were available for testing, the adhesive systems were provided directly by the corresponding manufacturers. Each manufacturer suggested that their own composite resin should be used with the corresponding dentin bonding agent, claiming that specific interactions might occur between the adhesive and the restorative composite as the marginal sealing potential of a restoration is attained by their combining⁴³. The sealing ability of the adhesive system itself relies on several factors such as bond strength, hydrophilicity, chemical stability and the nature of the solvent⁴⁴. The elastic modulus of the composite, its shrinkage, water uptake and the coefficient of thermal expansion, among other factors, are important determinants that could influence the final performance of the restoration⁴⁵. Therefore, it would be reasonable to assume that if adhesive systems are to be compared, one type of composite should be used. However, many reports provide confusing information on the choice of either one restorative composite or the combination composite/adhesive system provided by the same manufacturer for the comparison of adhesive systems^{25, 43, 46, 47, 48}. Considering that the testing conditions of this study were intended to mimic the clinical conditions as closely as possible, a decision was ultimately made to employ the adhesive/composite combination from the same manufacturer^{31, 49}.

As shown in Table 2a, the percentages of continuous margins before loading were high for most of the products tested. However, this changed considerably after loading due to marginal degradation (Table 2b), resulting in up to 68.6 % of marginal defects. As significant differences in marginal adaptation were identified among the groups, the first null hypothesis tested in this study had to be rejected.



Figures 2a and 2b. 2a is a SEM photomicrograph of a “continuous margin” at the enamel/composite interface after loading (400x magnification) (modified iBond material, with phosphoric acid enamel etching carried out for 60 seconds). The composite material exhibits excellent continuity with the enamel surface. In 2b the margin of the same cavity in dentin is completely open (400x magnification).

Of all the groups tested, ART Bond showed the best marginal adaptation for the total marginal length after loading. Although this material is in the self-etching group with comparatively low acid aggressiveness, its percentage of “continuous margin” in enamel was among the highest (Table 3b). It must be stressed that the margins of all restorations were located in non-sclerotic dentin and in mechanically cut enamel. In sclerotic dentin and on uncut enamel the results might have been different⁵⁰.

Separate evaluation of enamel and dentinal margins as a complement to the assessment of total marginal length proved useful, as illustrated in the findings with Scotchbond Multipurpose Plus, iBond and Xeno III after loading. Although the total values for these three materials were similar, their behavior in enamel and dentin were different. iBond performed well in dentin but poorly in enamel. Xeno III had high values of “continuous margin” in enamel but low values in dentin, while Scotchbond Multipurpose Plus showed a similar performance both in enamel and in dentin. These observations may provide important information to the manufacturers for further optimization of their products and also to define the precise nature of a given adhesive system. Based on these observations, the second null hypothesis has also to be rejected.

Independent of the type of the adhesive system (etch & rinse or self-etch), a tendency towards an inverse relationship of the percentage of “continuous margin” in enamel and in dentin after loading was observed in several groups. This implies that when there was a high percentage of “continuous margin” in enamel, a lower percentage of CM in dentin was observed and vice versa. A logical explanation for these results is that there are different adhesive strengths with enamel and dentin: The polymerized composite resin shrinks toward the superior bond at one margin and at the same time away from the weaker bond at the other margin⁵¹ (Figures 3 to 5). The results show that dentinal adhesion is not always the weakest link, as some

of the products tested, e.g. iBond, had higher values of “continuous margin” in dentin than in enamel. Because iBond conditions the tooth substrate with a weak acid, it was speculated that its marginal adaptation on enamel could be improved by combining it with more aggressive etching. As 37% phosphoric acid is frequently used when effective adhesion to enamel is desired³, it was decided to selectively condition enamel for 45 seconds in an additional group described as iBond modified (iB (m)) in addition to the recommended standard application of this product. It is evident (see Table 5b) that for iB (m) the marginal adaptation in enamel improved compared with the finding for iBond original (iB (o)). Such an improvement in enamel marginal integrity, however, was achieved at the expense of a decline of the marginal integrity in dentin, with the latter decreasing from $92.2 \pm 12.0\%$ to $68.2 \pm 37.8\%$ after thermomechanical loading (Table 5c). Apparently, the good marginal adaptation attained in dentin was not only due to the excellent adhesion of iBond to this substrate, but also due to a weak adhesion to enamel, which allowed for shrinkage compensation (as schematically represented in Figs. 4 and 5). Figures 1 and 2 illustrate some of the most representative SEM observations of this phenomenon. This suggests that a single component system like iBond is not able to condition enamel and dentin simultaneously and equally efficiently for bonding. However, it is interesting to note that its overall performance did not differ from a multi-step etch & rinse adhesive such as Scotchbond Multi Purpose Plus. With Scotchbond Multipurpose Plus, enamel was etched for 15 seconds only in its original application protocol. It was speculated that a more aggressive etching by extending etching time to 60 seconds could improve its adaptation to enamel. The findings for this modified application showed that prolonged enamel etching for 60 seconds resulted in an insignificant improvement of marginal adaptation in enamel before and after loading when compared with the original application method.

Fig 3. Perfect Class V composite filling

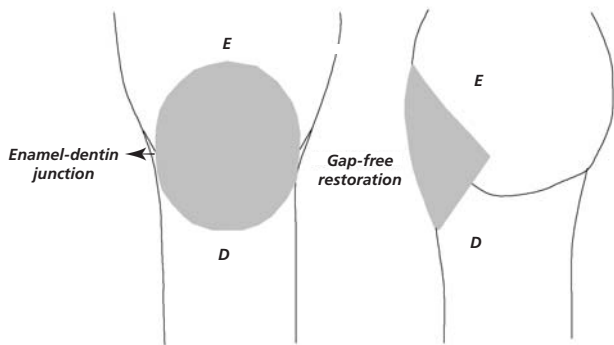


Figure 3. Schematic drawing of an ideal restoration where gap-free margins are present both in enamel (E) and dentin (D). In this case, the adhesion to enamel and dentin exceed the polymerisation contraction forces of the composite material as well as the deformation forces under load.

Recent studies have shown that improved adhesion may be obtained when formulations with separate primers and bonding agents are used⁴⁷. With enamel adhesion, etch & rinse systems are claimed to be the state of the art for reliable bonding to enamel. Moreover, simplified formulations of adhesive systems are presumed to result in lower performance in terms of bond strength and sealing abilities^{52, 53, 54, 55, 56}. Within the limits of this *in vitro* study, these findings could not be confirmed. On the contrary, our results substantiate previous reports^{8, 57}, that the establishment of a sufficient and stress resistant adhesion might be determined by factors other than the application steps and the adhesive strategy.

Conclusions

None of the systems tested in this study were able to completely eliminate the formation of marginal defects, either in enamel or in dentin. The best products with respect to the percentage of total "continuous margin" after loading were self-etching systems, closely followed by an etch & rinse system. It is suggested that the adhesion strategy (etch & rinse or self-etch) and the number of application steps may not be the only parameters to consider when evaluating the *in vitro* performance of dentin bonding agents. Other factors such as the compositions of the adhesive and composite may determine both the quality of initial marginal adaptation and the stability of the adhesion under load.

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Fig 4. Class V composite filling: Detachment from dentin margins

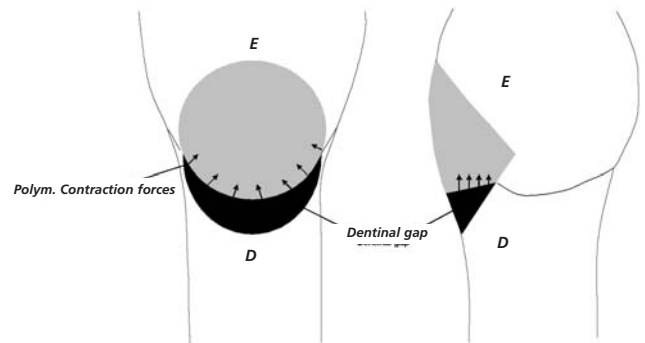


Figure 4. Schematic drawing of an excellent margin in dentin with corresponding detachment at enamel margins. This compensatory effect may occur due to two reasons: 1. Excellent adhesion to dentin & poor adhesion to enamel, or 2. Poor adhesion to dentin & non-existent adhesion to enamel.

Fig 5. Class V composite filling: Detachment from enamel margins

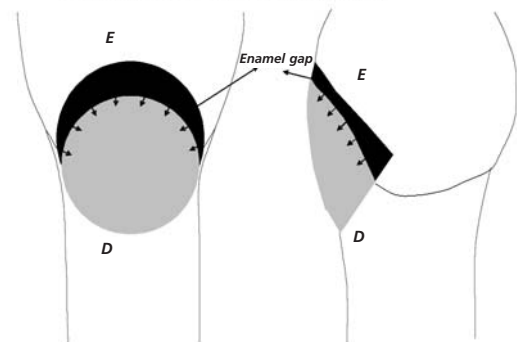


Figure 5. Schematic drawing of an excellent margin in enamel with corresponding detachment at dentin margins. The compensatory effect may occur due to: 1. Excellent adhesion to enamel & poor adhesion to dentin, or 2. Poor adhesion to enamel & non-existent adhesion to dentin.

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