Scanning Microscopy

Volume 3 | Number 4 Article 4

12-15-1989

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Scanning Microscopy, Vol. 3, No. 4, 1989 (Pages 1013-1022) 0891-7035/89$3.00+.00
Scanning Microscopy International, Chicago (AMF O'Hare), IL 60666 USA

EFFECTIVENESS OF DENTIN BONDING AGENTS

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(Received for publication April 29, 1989, and in revised form December 15, 1989)

Abstract

The interface between a composite resin restoration and dentin is still a problem with regard to secondary caries and esthetics. With conventional restorative techniques the absence of bonding between the restorative material and the dentin produces marginal gaps. These gaps are populated with microorganisms or trap pigments. Therefore, adhesive techniques are required for functional and esthetic restorations.

In extracted teeth stored in 0.1% thymol solution, 8 cylindrical cavities (diameter: 3 mm; depth: 2 mm) and 8 class V cavities (1/2 of the margin in dentin) were prepared in each group and filled with composite resin. Before actual placement the following dentin adhesives were used: *Scotchbond 2* (3M), *Gluma* (Bayer), *Scotchbond LC* (3M), *Dentin Adhesit* (Ivoclar), *Dentin Adhesive* (Kulzer) and *Durafill-Bond* (Kulzer) as a control. Before and after thermocycling (TC) (2000 cycles, 5° C to 55° C) replicas were taken and a quantitative margin analysis in the SEM was performed at 200 x magnification. To analyze the data, a rating scale (four criteria) was used to characterize the marginal configuration. Statistical analysis of the results showed that the materials *Gluma, Scotchbond LC* and *Scotchbond 2* yielded significantly ($p<0.05$) better margins than the other materials in both cylindrical and class V cavities. Before TC values > 90% were found with *Gluma, Scotchbond LC* and *Scotchbond 2* in Class V cavities. However, after TC, these values decreased significantly to 66% for *Gluma*, 42 % for *Scotchbond LC* and 81 % for *Scotchbond 2*. *Scotchbond 2* showed approximately 80% excellent margins in both cavity forms after TC.

A subsequent clinical study should be done to confirm these favourable results in vivo.

Key words: dentin bonding agents, quantitative margin analysis, dental materials, in vitro.

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Introduction

Dental restorations must seal the interface between the restorative material and tooth hard tissues. If this is not the case, functional and esthetical deficiencies will occur. The function is impaired, because microorganisms will colonize the gaps formed. Due to their toxins, pulpal disease will occur and due to their metabolic products, recurrent caries may develop. The esthetics are impaired, because gaps usually pick up stains and thus become visible even to the naked eye [24].

With the enamel etching techniques it is possible to obtain the above mentioned high requirements [24]. However, this technique does not work on dentin margins, which occur in many clinical situations, such as deep class II or III cavities or class V cavities. Dentin bonding agents are used to improve the marginal seal of composite resin restorations at the dentin/composite interface. Several dentin adhesives were developed in the last years and are now available.

These adhesives are built to obtain a chemical bond to dentin based on the following basic formula:

M-R-X

where M = a methacrylate group, R = a pendant chain to join M and X and X = a functional group, designed to react with the dentin [2]. The bonding "targets" on the dentin side are either the collagen or the calcium ions. A common substance for dentin adhesion are phosphate esters in combination with sulfonates (Scotchbond LC, Dentin adhesive). The phosphate bond is stabilized by halogens. As a bonding mechanism, chelation of Ca ions and C-C double bonds with the composite material are discussed [2]. This system is designed to incorporate the smear layer. Isocyanates (Dentin Adhesit) are also promising substances for dentin bonding, because they are able to react with the OH, NH_2, and COOH groups of the collagen. They are able to react in a hydrophilic environment, which is supposed to be a large advantage [2]. Another very promising approach is the combination of glutaraldehyde with 2-hydroxyethylmethacrylate (HEMA) (Gluma) [19]. Glutaraldehyde reacts with the collagen by forming N-(hydroxyalkylic) bonds with amino-, imino-, or amido- groups. This reaction product further reacts with HEMA by forming a methacrylic "film" bonded to the dentin. The methacrylic groups are copolymerized to the resins of the diacrylate based composite resins [19]. With Scotchbond 2 a similar mechanism was desired, by avoiding the use of glutaraldehyde. With this system a primer, containing a hydrophilic monomer (HEMA) and maleic acid, dissolves calcium out of the smear layer. The adhesive, containing a hydrophilic monomer (HEMA), Bis-GMA, and a photoinitiator, penetrates and interlocks with the pretreated surface.

Many investigations on the effectiveness of these dentin adhesives have been done by measuring the maximum marginal gap [1, 2, 11, 15, 16]. The margins are judged 0.1 mm below the original surface and only along a small part of the margin length - just where the gap occurs. However, it is also important to examine the entire margin of the restoration which lies in dentin. An effective method for this purpose is the quantitative margin analysis [24].

The purpose of this in vitro investigation was to determine the effectiveness of seven dentin bonding agents on the marginal adaptation of composite materials, placed in cylindrical and class V cavities, using morphological analyses of the margins.

Materials and Methods

Cylindrical cavities

Extracted teeth, stored in a 0.1% thymol solution, were randomly assigned to the different groups listed in Table 1. The crowns were cut off and the roots were ground flat on their proximal surfaces. Eight cylindrical cavities were prepared into the flat surface with a diamond bur (*1 Table 2) at high speed using water as a coolant. The preparation was 3 mm in diameter and approximately 2 mm deep.

The dentin was treated and the adhesives applied according to the manufacturers' instructions. The composite resins were then applied in one increment.

Table 1. Dentinadhesive/Composite systems (n=8 for each group).

Group	Dentin adhesive	Composite
1	Durafill-Bond	Durafill
2	Dentin Adhesive	Durafill
3	Dentin Adhesit	Heliosit
4	Gluma	Lumifor
5	Gluma	Durafill
6	Scotchbond LC	Silux
7	Scotchbond 2	Silux

Table 2. Materials and Devices used.

* 1	Diamond bur No. 838/314/014; Gebr. Brasseler GmbH D-4920 Lemgo, FRG
* 2	Finishing diamond bur No. 8838/314/012; Gebr. Brasseler GmbH D-4920 Lemgo, FRG
* 3	Sof-Lex Pop-on Nr. 1981 SF/F/M/C; 3M Deutschland GmbH D-4040 Neuss, FRG
* 4	President light body; Coltene AG CH-9450 Altstätten
* 5	Stycast 1266 Part A + B; Emerson and Cumming B-2431 Westerlo-Oevel
* 6	Stereoscan 100; Cambridge Instruments D-4600 Dortmund, FRG

Class V cavities

Eight extracted teeth, stored in a 0.1% thymol solution, were randomly assigned to the different groups listed in Table 2. The class V cavities were prepared with a diamond bur (*1) at high speed using water as a coolant. The oval preparation was approximately 1.5 mm deep, 3 mm wide, and 4 mm high (2 mm were apical to the cementoenamel junction). The enamel

Table 3. Criteria for the marginal examination in the SEM at a magnification of 200.

Margin Quality	Definition
Margin Quality 1 (Fig.1)	Margin not or hardly visible No or slight marginal irregularities, no gap
Margin Quality 2 (Fig.2)	No gap but severe marginal irregularities
Margin Quality 3 (Fig.3)	Gap visible (hairline crack up to 2 µm) No marginal irregularities
Margin Quality 4 (Fig.4)	Severe gap (more than 2 µm) Slight and severe marginal irregularities
	the term "marginal irregularities" means - porosities - marginal restoration fracture - bulge in the restoration

Fig. 1. Example of margin quality 1 in the SEM.

Fig. 2. Example of margin quality 2 in the SEM.

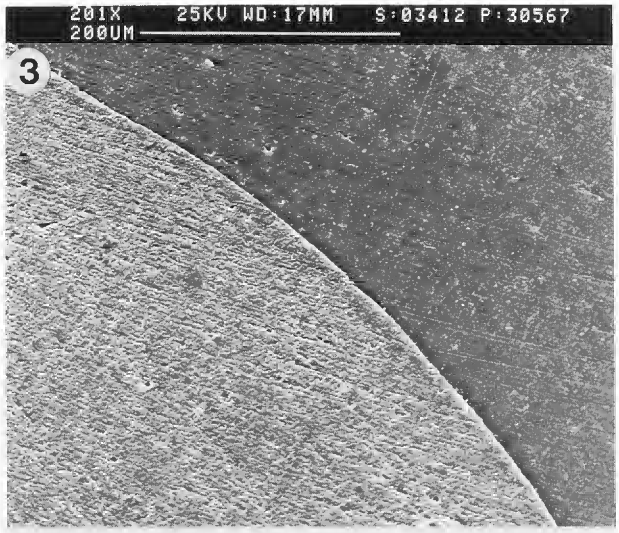

Fig. 3. Example of margin quality 3 in the SEM.

Fig. 4. Example of margin quality 4 in the SEM.

portions were beveled with a finishing diamond bur (*2) and the cavosurface margins in dentin finished to a 90 degree angle. The enamel portions of the cavity margins were etched for 60 s.

The dentin was treated and the adhesives applied according to the manufacturers' instructions. The composite resins were then applied in three increments starting at the cervical margin.

<u>Experimental parameters and analytical techniques</u>

After polishing (*3), the teeth were stored for 21 days in water and then thermocycled for 2000 cycles between +5 °C and +55 °C. The immersion time was 30 s and the transition time 12 s. Before and after the thermocycling procedure, impressions were taken with a polyvinylsiloxane impression material (*4) and replicas were produced by casting the impressions with an epoxy resin (*5).

The margins of the restorations at the dentin/composite interface were examined and quantified with a scanning electron microscope (SEM) (*6) at a magnification of 200X using defined criteria (Table 3, Figs. 1 - 4) to assess the margin qualities. The entire perimeter of the restoration is assessed in multiple steps. Using a digitizer or a mouse, the beginning and endpoint of a specific margin quality are marked. If the perimeter is not linear in the assessed area, intermediate points are marked to account for the curvature. Then the length of the marked margin portion is automatically measured and stored in the file corresponding to the assigned margin quality. After completion of the assessment of the complete restoration margin, the margin quality is expressed as a percent distribution of the different criteria (1-4) [24]. The configuration of the system

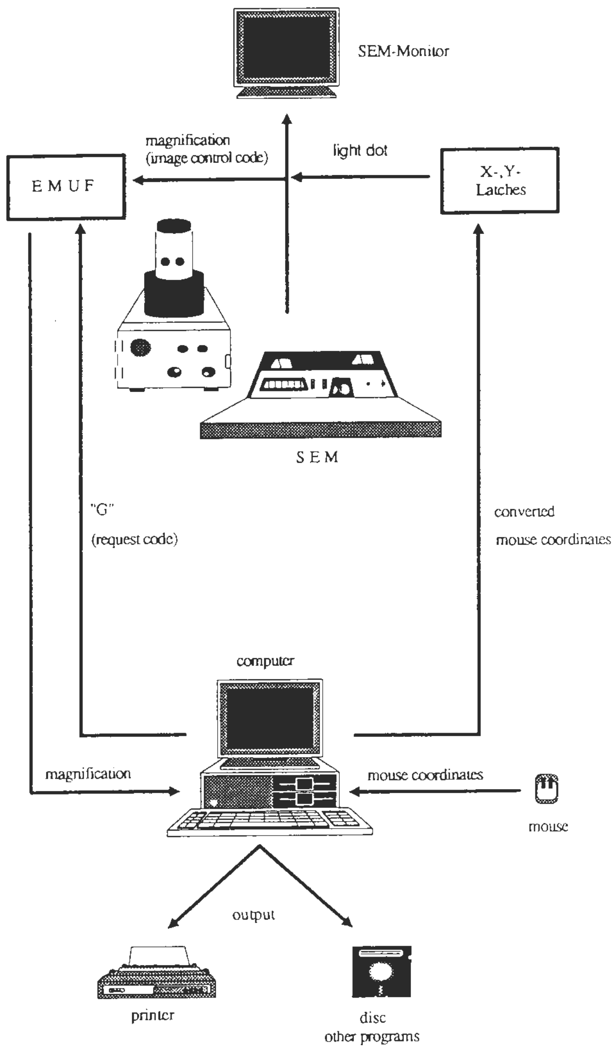

Fig. 5. Block diagram of the SEM - computer system combination used for the evaluation.

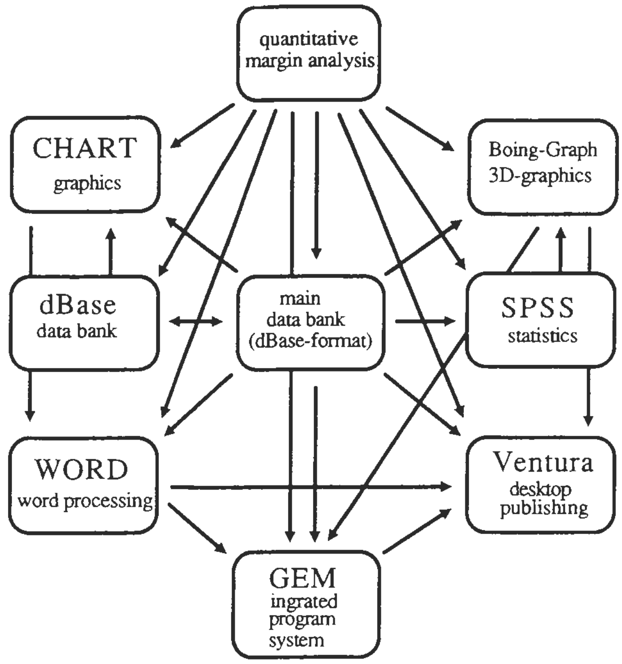

Fig. 6. The software used allows easy data transfer into many commercial programs.

published earlier [24] was improved by (1) using a more powerful computer and (2) building a ROM-programmed single board microcomputer (EMUF) interface, which makes it possible to also give the computer the magnification factor of the SEM image (Fig. 5). Additionally, by using an AT-computer, a convenient data format could be used so that the raw data were directly processed (Fig. 6).

Results

The results of the experiments are summarized in Tables 4 to 7 and Figures 7 to 10.

The statistical evaluation was done by using the SPSS statistical software [20]. With the WILCOXON test significant differences ($p<0.05$) were found between the dependent groups before and after thermocycling. As an analysis of variance by ranks, the KRUSKAL-WALLIS test, was performed for the data both before and after TC and showed main effects for the materials. The NEMENYI test then was done to analyse significant differences ($p<0.05$) in the amount of the defined margin qualities between the different dentin bonding agents.

Cylindrical cavities (Fig. 7 and 8)

For the adhesives *GLUMA*, *SCOTCHBOND LC* and *SCOTCHBOND 2* margin quality 1 was found on approximately 80% - 90% of the margin length before thermocycling (TC). These results are significantly better ($p<0.05$) than those of *DENTIN ADHESIVE* and *DENTIN ADHESIT*. After thermocycling *SCOTCHBOND 2* showed still approximately 75% margin quality 1, while the amount for this quality for *GLUMA* and *SCOTCHBOND LC* decreased to approximately 40% and 60%, respectively. The results of *GLUMA*, *SCOTCHBOND LC* and *SCOTCHBOND 2* are significantly better ($p<0.05$) than those of *DENTIN ADHESIVE*, *DENTIN ADHESIT* and *DURAFILL-BOND*.

Class V cavities (Figs. 9 and 10)

Before thermocycling (TC) for the adhesives *GLUMA*, *SCOTCHBOND LC* and *SCOTCHBOND 2* acceptable marginal adaptation (quality 1) was observed on more than 90% of the margin length. With *SCOTCHBOND 2* 100% margin quality 1 was obtained. Examining the margins of the class V cavities, the interface dentin/composite was hardly recognizable. The percentages of quality 1 for the above mentioned materials are significantly better than those obtained with the other materials. After TC, the amount of margin quality 1 decreased with the adhesive/composite system *GLUMA/DURAFILL* to approximately 60% of the margin length, with the *GLUMA/LUMIFOR* system and with *SCOTCHBOND LC* to approximately 40%, while approximately 80% of the margin length of the composite fillings applied with *SCOTCHBOND 2* still showed best marginal adaptation. The results for *GLUMA/DURAFILL*, *GLUMA/LUMIFOR* and for *SCOTCHBOND LC* and *SCOTCHBOND 2* are significantly better ($p<0.05$) than the amount for margin quality 1 after TC of *DURAFILL BOND*, *DENTIN ADHESIVE*, and *DENTIN ADHESIT*.

Discussion

Since the replica technique is non-destructive the quality of filling margins can be assessed and marginal defects are easily detected. The high sensitivity of this method, due to the SEM's excellent detail reproduction, is a great advantage for the evaluation of the bonding of dentin adhesives [24]. With the quantitative margin analysis, minute changes at the dentin/composite interface can be recognized and the ability for a marginal sealing of the tested adhesive can be assessed for the entire margin length.

The different margin qualities were chosen to characterize the different consequences for the clinical situation. While margin quality 1 is what we expect as a marginal sealing, all kinds of gaps (margin quality 3 and 4) are a sign of failure of adhesion. Margin quality 2 may not be a complete failure of adhesion but it doesn't show the marginal adaptation required for a composite resin restoration. Therefore the statistical comparision for the conclusions was done especially for margin quality 1.

The occuring gaps at the dentin/composite resin interface can be related to the material, because the application techniques were similar in all cavities. With these techniques some materials (Figure 7 and 9) showed 90 to nearly 100 % perfect marginal adaptation before TC. Higher amounts of gaps were found only after TC. These gaps appeared in areas which were rated with margin quality 1 before TC as well as in marginal segments with margin quality 2.

The adhesives *SCOTCHBOND 2*, *GLUMA* and *SCOTCHBOND LC* produced significantly better marginal adaptation than the other tested materials. The statistical evaluation was done by the comparison of ranks for nonparametrically distributed data in the seven groups as described by NEMENYI. This test shows less differentiation with increasing numbers of groups. Statistical comparison between the four most effective dentin adhesive groups, using a pairwise Mann-Whitney test, shows significant differences ($p<0.05$) between *GLUMA/LUMIFOR* and *GLUMA/DURAFILL* for cylindrical cavities before TC and between *SCOTCHBOND 2* and *SCOTCHBOND LC* for class V cavities after TC.

When these results are ranked there is good correlation between the results of this study and those of shear bond tests. The 3M company reported bond strength values of approximately 18 MPa for *SCOTCHBOND 2* [14], while bond strength values for *GLUMA* were reported to be approximately 13 MPa [5], 15 MPa [3, 18] and 17.5 MPa [19]. The direct comparison of shear bond strength is given in a study by Retief et al. [22] and showed mean bond strength values of 6.9 to 8.5 MPa for *SCOTCHBOND 2* and of 8.9 to 10.8 MPa for *GLUMA* [21]. The large discrepancy in the measurements of bond strength tests signifies the problems connected with in vitro studies [7].

The results of the present investigation are in accordance with those of other studies dealing with the effect of dentin adhesives on margin qualities. Some authors used *GLUMA* and observed smaller contraction gaps or better margin qualities [10, 13, 15, 17, 23, 25]. Some researchers found *SCOTCHBOND LC* to be an effective adhesive [4, 8, 11, 23], while other authors did not obtain acceptable results [6, 9, 10, 12]. With *SCOTCHBOND 2* comparisons were not possible since further data on marginal behaviour were not available.

Contrary to the results of Robinson et al. [23], there was no significant decrease in margin quality when a different composite resin other than that recommended by the manufacturer was used.

Conclusion

The morphological analytical techniques show large differences in the effect of different dentin bonding agents. In both cavity types tested, stressing the restorations by thermocycling significantly decreased the performance of all dentin bonding agents. Some dentin adhesives (*Gluma, Scotchbond LC and Scotchbond 2*) were effective, showing 40 % - 80 % excellent margin quality even after TC in cylindrical and class V cavities. Other dentin bonding agents (*Dentin Adhesive, Dentin Adhesit*) did not show sufficient bonding capabilities, since after TC the percentage of excellent margin (= no gap formation) dropped below 10 %.

A clinical study must be carried out in order to show whether the promising materials are the solution to the problems at the dentin/composite interface.

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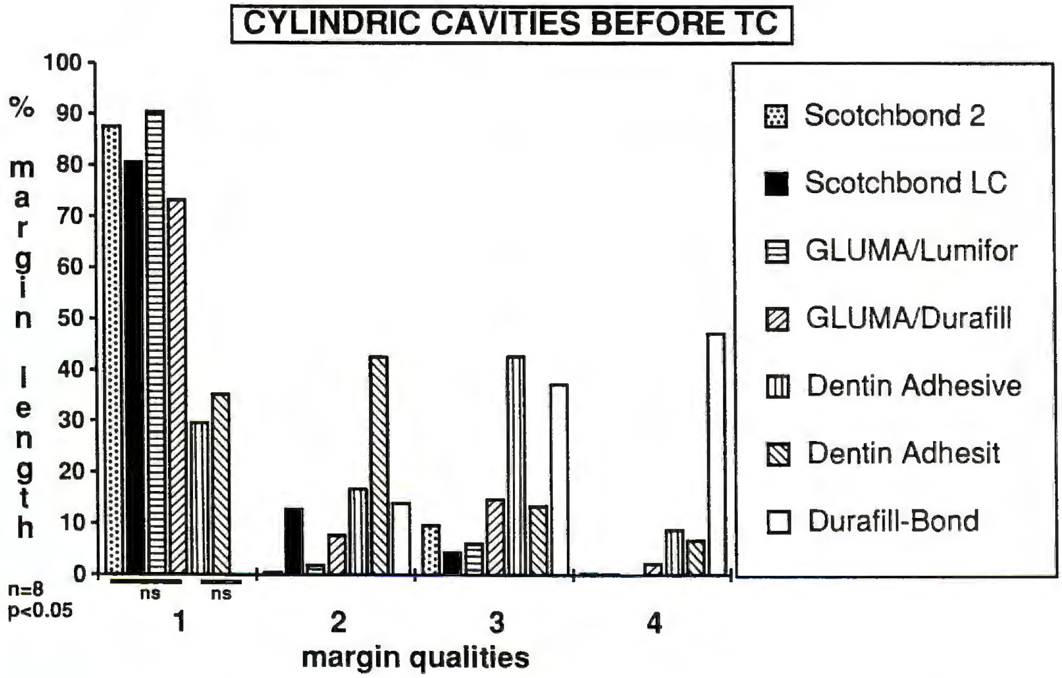

Fig. 7. Results for the cylindrical cavities before thermocycling.

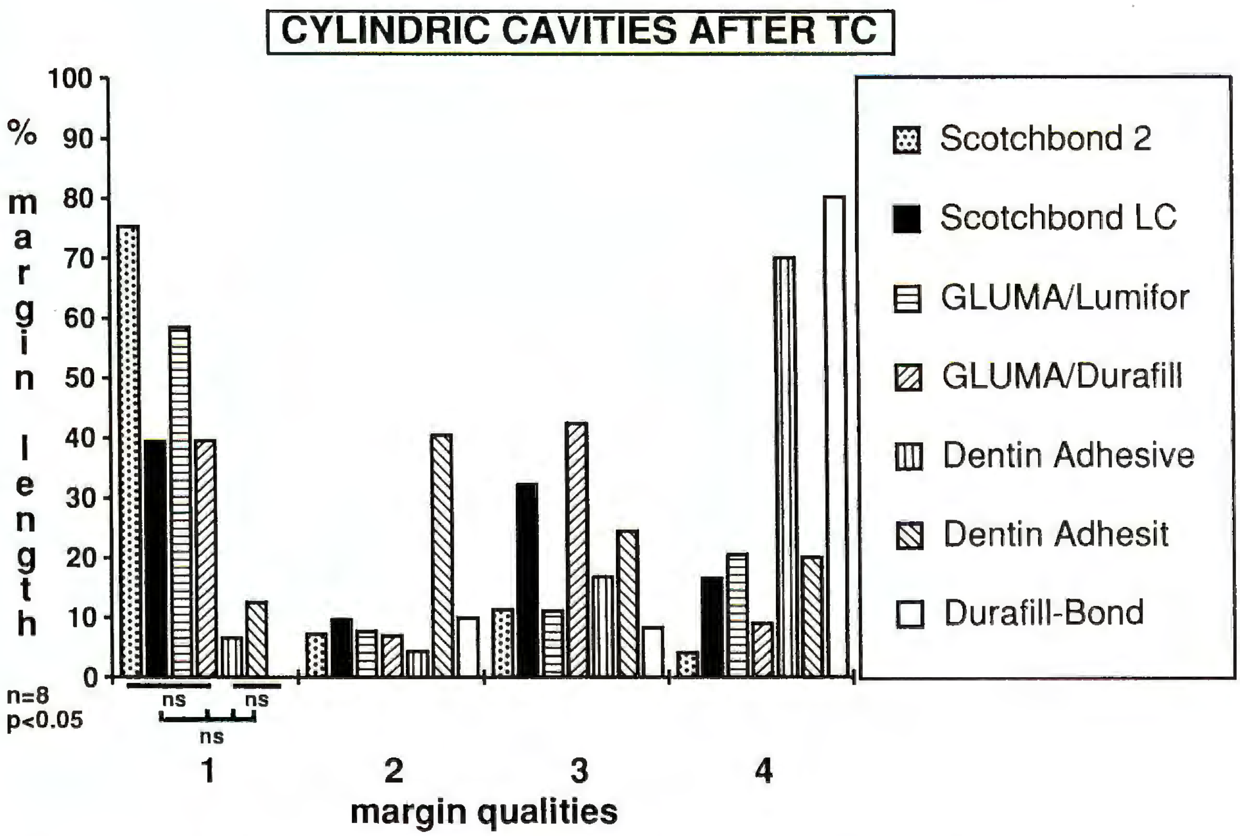

Fig. 8. Results for the cylindrical cavities after thermocycling. Means connected with a horizontal bar are not statistically different. With the lower bar, the means depicted with vertical marks are not statistically different (ns).

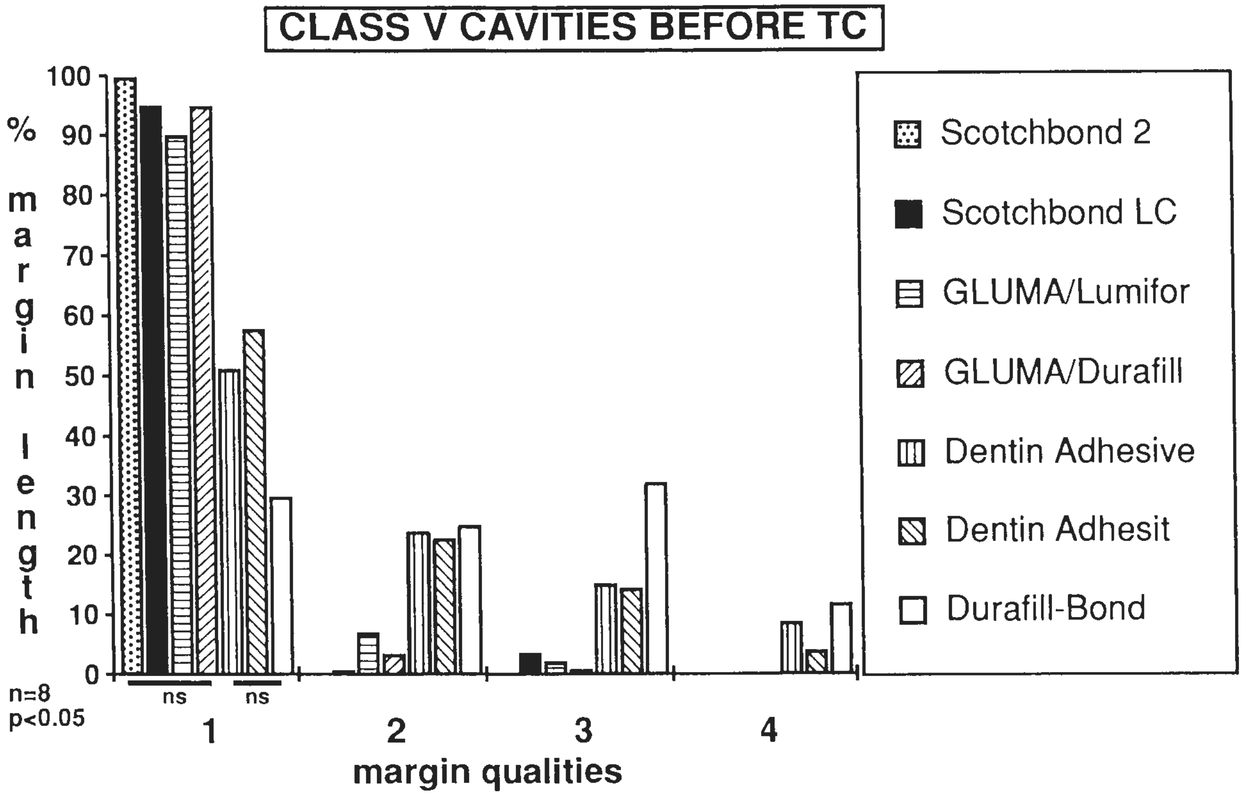

Fig. 9. Results for the class V cavities before thermocycling.

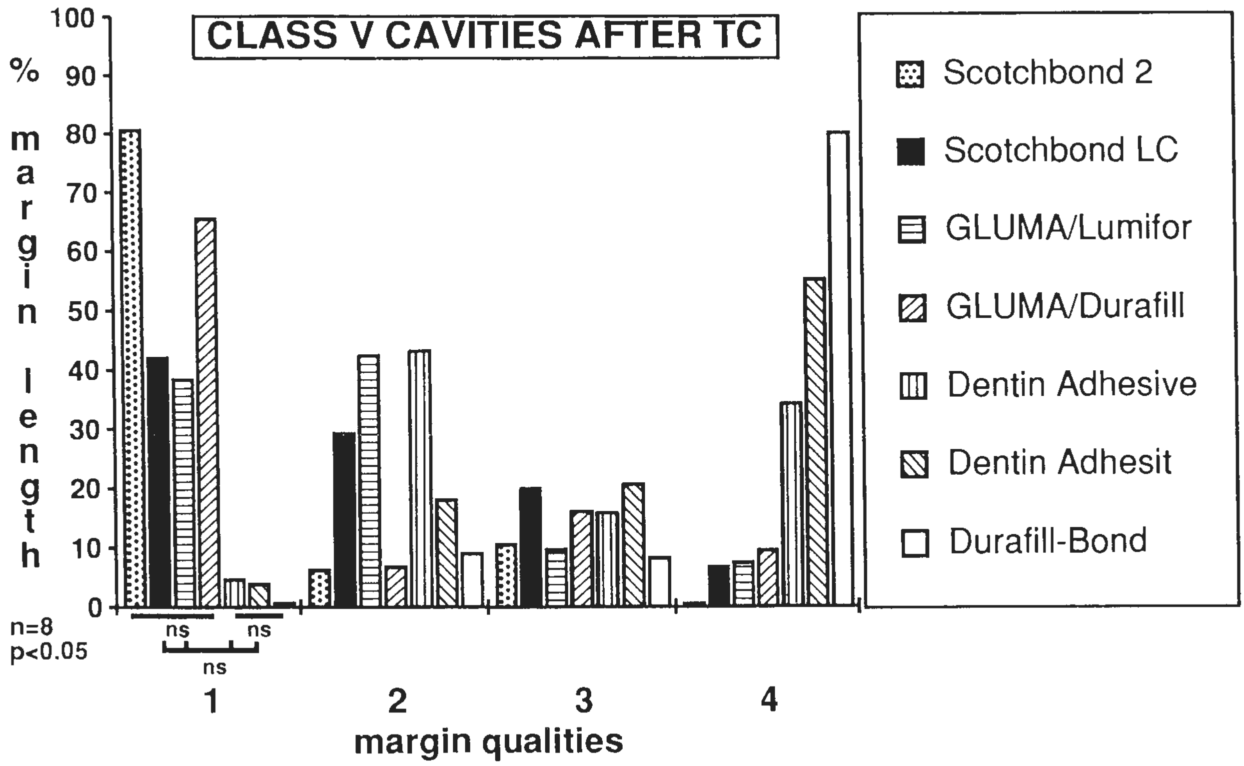

Fig.10. Results for the class V cavities after thermocycling. Means connected with a horizontal bar are not statistically different (ns). With the lower bar, the means depicted with vertical marks are not statistically different.

Table 4. Average margin length before TC in cylindrical cavities n=8 for each group M = mean SD = standard deviation.

MATERIALS		MARGIN QUALITIES 1	2	3	4
Durafill (Kulzer)	M	0.000	14.500	37.813	47.688
	SD	0.000	14.702	15.515	17.179
Dentin Adhesive (Kulzer)	M	30.113	17.263	43.325	9.262
	SD	26.314	12.373	20.815	14.850
Dentin Adhesit (Vivadent)	M	35.700	43.175	13.912	7.212
	SD	21.075	12.039	12.606	9.451
GLUMA/LUMIFOR (Bayer)	M	90.975	2.413	6.600	0.000
	SD	15.839	6.824	9.498	0.000
GLUMA/DURAFILL (Bayer)	M	73.763	8.200	15.338	2.725
	SD	20.559	6.713	16.621	3.246
Scotchbond LC (3M)	M	81.050	13.225	4.937	0.775
	SD	16.835	10.149	7.812	2.192
Scotchbond 2 (3M)	M	87.963	0.950	10.262	0.825
	SD	18.182	2.687	15.279	2.334

Table 5. Average margin length after TC in cylindrical cavities n=8 for each group M = mean SD = standard deviation.

MATERIALS		MARGIN QUALITIES 1	2	3	4
Durafill (Kulzer)	M	0.000	10.388	8.800	80.813
	SD	0.000	15.253	10.472	19.997
Dentin Adhesive (Kulzer)	M	7.050	4.800	17.362	70.762
	SD	12.251	8.065	17.136	29.150
Dentin Adhesit (Vivadent)	M	13.175	41.100	25.075	20.625
	SD	8.494	25.551	19.108	14.332
GLUMA/LUMIFOR (Bayer)	M	59.137	8.225	11.550	21.075
	SD	32.0917	8.278	11.045	28.630
GLUMA/DURAFILL (Bayer)	M	40.025	7.350	43.050	9.550
	SD	11.490	3.926	11.369	10.074
Scotchbond LC (3M)	M	40.000	10.112	32.838	17.062
	SD	29.492	11.271	23.621	9.709
Scotchbond 2 (3M)	M	75.800	7.750	11.875	4.537
	SD	29.405	14.188	11.593	8.965

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Table 6. Average margin length before TC in class V cavities n=8 for each group M = mean SD = standard deviation.

MATERIALS		MARGIN QUALITIES 1	2	3	4
Durafill (Kulzer)	M	30.100	25.300	32.462	12.125
	SD	30.447	18.327	27.394	15.499
Dentin Adhesive (Kulzer)	M	51.487	24.187	15.375	8.925
	SD	33.752	24.850	15.702	15.123
Dentin Adhesit (Vivadent)	M	58.050	23.075	14.712	4.187
	SD	31.435	19.960	11.897	6.824
GLUMA/LUMIFOR (Bayer)	M	90.300	7.275	2.425	0.000
	SD	18.622	14.119	4.511	0.000
GLUMA/DURAFILL (Bayer)	M	95.237	3.662	1.100	0.000
	SD	13.470	10.359	3.111	0.000
Scotchbond LC (3M)	M	95.175	1.037	3.800	0.000
	SD	6.987	2.934	7.036	0.000
Scotchbond 2 (3M)	M	100.000	0.000	0.000	0.000
	SD	0.000	0.000	0.000	0.000

Table 7. Average margin length after TC in class V cavities n=8 for each group M = mean SD = standard deviation.

MATERIALS		MARGIN QUALITIES 1	2	3	4
Durafill (Kulzer)	M	1.225	9.525	8.725	80.525
	SD	3.465	9.351	8.483	16.331
Dentin Adhesive (Kulzer)	M	5.225	43.750	16.375	34.650
	SD	9.825	21.802	10.623	26.566
Dentin Adhesit (Vivadent)	M	4.412	18.637	21.200	55.737
	SD	12.480	11.811	15.468	22.901
GLUMA/LUMIFOR (Bayer)	M	38.925	43.025	10.162	7.850
	SD	26.256	20.942	13.548	12.698
GLUMA/DURAFILL (Bayer)	M	66.150	7.262	16.575	10.013
	SD	39.039	8.966	23.347	19.299
Scotchbond LC (3M)	M	42.587	29.762	20.512	7.150
	SD	29.470	18.201	16.525	9.677
Scotchbond 2 (3M)	M	81.075	6.787	11.063	1.100
	SD	14.847	8.013	14.933	3.111

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Discussion with Reviewers

H.J. Mueller: Since Scotchbond 2 and Dentin Adhesive are both based upon phosphate esters in combination with sulfonates, how can the large differences in adhesion between the two materials be explained if not by chemistry? Or are there subtle differences in chemistries between the materials that may explain the differences in behavior ?
Authors: We assume that you mean Scotchbond LC, since Scotchbond 2 has a completely different chemistry. The true bonding mechanisms of these so-called dentin bonding agents are still not completely understood and are often based on hypotheses. We agree that both materials are based on phosphate esters. However, phosphate esters are a large class of compounds and the two manufacturers have selected different phosphate esters. Without going into chemical details, we will explain the differences. Based on the difference, Scotchbond LC is more active and more acidic than Dentin Adhesive. Additionally, with Scotchbond 2 ethanol is used as a solvent, while Dentin Adhesive is dissolved in acetone. With these differences we have to expect a different clinical behavior for both materials: the interaction with the smear layer is changed by the pH; the hydrophilic/hydrophobic properties are different and thus the wettability is changed; and probably the bonding mechanism is influenced by all chemical properties. It seems that the interaction with the smear layer, which is partially removed due to the acidic pH of these materials, is crucial, thus supporting the suspicion that dentin bonding is more a micromechanical phenomenon than a true chemical bonding.

G.W. Marshall: What was the rational for establishing the marginal index characteristics ? Would a material with a combined percentage of type 1 and 2 characteristics be clinically inferior to a different material with a similar level of type 1 margins and essentially zero type 2 margins ?
Authors: The quantitative margin analysis has the advantage that the assessment criteria can be easily adapted to the different experiments. In this study the primary question is whether bonding has occurred or not. Therefore we have distinguished between gaps (quality 3 and 4) and no gaps (quality 1 and 2). If your hypothetical material should show virtually no qualities 3 and 4 and we should evaluate bonding, our answer is no. However, especially in the cervical area, we must demand restorative material - dentin transitions which are as smooth as possible, to prevent plaque accumulation. Thus, seeing the problem as clinicians, we would prefer a material showing as much as possible the margin quality one to prevent not only pulpal diseases (no gaps) but also plaque retention. Therefore, in the latter case, our answer is yes.

G.W. Marshall: What kind of defects demonstrated progression as a result of thermocycling; i.e. do gaps widen, do minor cracks become irregularities or do 'good' margins become gaps directly ?
Authors: The thermocycling process usually has two effects on the margin quality: (1) The stresses caused at the interface due to the difference of the coefficient of thermal expansion create a debonding process, visible as a gap formation. (2). Minute cracks (in the restorative material) and irregularities in the marginal area, which were induced during the trauma from the contouring, finishing or polishing process, are "demasked" after thermocycling, e.g. are only visible after thermocycling. If you look at the Figures 7 - 10 it is clearly visible that the largest increase was observed with the margin quality 4, e.g. in the combination of the above-mentioned effects, which makes sense to us. The increase in quality 2 indicates that, despite some trauma from contouring/finishing or polishing, the dentin bond did not suffer, whereas the increase noticed in qualities 3 and 4 is a sign of debonding. With "good bonding" material we see an increase in quality 2 (if ever) and with "poor bonding materials" usually quality 3 shifts to quality 4 after TC or we can see a shift from quality 1 or 2 to quality 3 or 4.

G.W. Marshall: Could the authors speculate on how much margin quality index of 3 or 4 can be tolerated before or after thermocycling and still be considered an effective system?

Authors: You have precisely pinpointed the number one problem of dental materials and dental materials-related clinical research. There is no study with sound, hard data showing a direct correlation between the margin quality and the clinical occurrence of recurrent caries and/or pulpal diseases. In addition, we have to accept the fact that dentin as a living tissue is subjected to physiological changes, which makes this issue even more complicated. Therefore we must, as we have already stated several times, induce complex clinical studies which monitor as accurately as possible the margin quality and observe the clinical outcome over the years.