

In vitro quality testing of dentin adhesives

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A tensile and a shear bond strength test are compared, using two well-known and two more recently marketed dentin adhesive systems: Gluma (Bayer), Scotchbond 2 (3M), Scotchbond Multi-Purpose (3M), and Syntac (Vivadent). The adhesives were used to bond composite resins to the buccal surface of human third molars. Specimens stored for 24 h before testing did not show significant differences between bond strength values obtained by either the tensile or the shear test method for any of the materials. The bond strength of thermocycled specimens did not differ for Gluma, was significantly lower for Scotchbond 2, and significantly higher for Scotchbond Multi-Purpose and Syntac. SEM observations showed increasing amounts of cohesive failure in resin with increasing bond strength values. Cohesive fractures in dentin were also observed. It is concluded that either the tensile or the shear test method can be used for quality testing of dentin adhesives. Thermocycling may provide interesting information about the quality of a bond. □ *Composite resins; dental bonding; dental materials; dentin*

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New and improved versions of dentin adhesives are constantly being marketed. It is difficult to assess quality and usefulness of the products, as in vitro data vary considerably and have an uncertain clinical relevance. When clinical long-term data are available, the product is often outdated. At present no pre-marketing approval of such products is obtained in Scandinavia.

Bond strength testing is often used to demonstrate the quality of dentin adhesives. This is, however, only one of several ways to assess the quality of such materials and can only be used with certain limitations (1). A group of experts working for The International Organization for Standardization, ISO/TC 106 Dentistry, has for some years dealt with the subject of developing standardized tests for quality assessment of adhesive materials, including dentin adhesives. A document, TR 11405 Dental Materials—Guidance on Testing of Adhesion to Tooth Structure (2), has recently been approved for publication. In this document several methods for quality testing of adhesive materials are described, including both a tensile bond strength test and a shear bond strength test. Such tests may be per-

formed after fulfilling various storage conditions, such as 24 h in water or a specified thermocycling of specimens.

It was the purpose of the present study to compare the effect of the two bond strength methods and the storage conditions on the measured bond strength of two well-known dentin adhesive systems and two recently marketed systems.

Materials and methods

Materials used in this study are shown in Table 1.

Gluma Bonding System is based on pretreatment of the dentin with an ethylenediaminetetraacetic acid (EDTA) solution. The adhesive is an aqueous solution of glutaraldehyde and 2-hydroxyethyl methacrylate (HEMA), and a bonding resin based on bisphenol-A-glycidyl dimethacrylate (BISGMA) is used as intermediate layer to a composite.

Scotchbond 2 consists of a primer based on HEMA and maleic acid and a light-cured adhesive based on a mixture of HEMA and BISGMA.

Table 1. Materials used in this study

Adhesive	Composite	Producer	Curing lamp used
Gluma Bonding System	Pekafill	Bayer Dental, Leverkusen, Germany	Visilux 2
Scotchbond 2	Silux Plus	3M Dental Products Division, St. Paul, Minn., USA	Visilux 2
Syntac	Heliomolar	Vivadent, Schaan/Liechtenstein	Heliolux II
Scotchbond Multi-Purpose Dental Adhesive System	Silux Plus	3M Dental Products Division, St. Paul, Minn., USA	Visilux 2

The Scotchbond Multi-Purpose system consists of an etchant containing maleic acid and poly(vinyl alcohol), a primer containing HEMA and polycarboxylic acid, and a light-

cured adhesive containing BISGMA and HEMA.

Syntac has a primer based on triethylenglycol dimethacrylate (TEGDMA) and maleic acid in acetone and water. The adhesive is an aqueous solution of polyethylene glycol dimethacrylate (PEGDMA) and glutaraldehyde, and the bonding resin contains BISGMA and TEGDMA (2, 3).

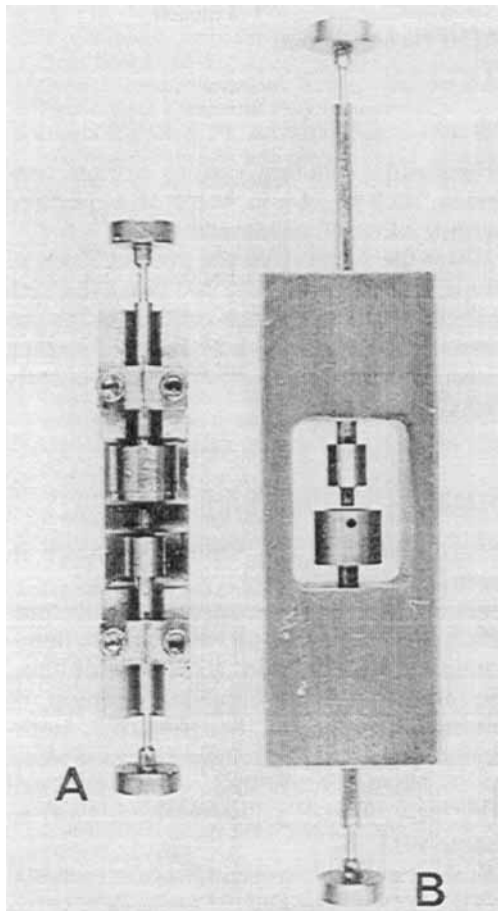


Fig. 1. Tensile test apparatus. 1A. Bonding alignment block. 1B. Measurement alignment block.

Substrate

Recently extracted, caries-free third molars were used in this study. They were stored for a maximum of 1 week in the dental surgery in a 1% chloramine solution and thereafter cleaned and stored in distilled water in the refrigerator.

Before application of the adhesive, the buccal surface of the teeth was ground and polished on wet silicon carbide paper grade 1000 to expose a dentin surface at least 4 mm in diameter.

Tensile bond strength test

The tensile test used was originally proposed by Kemper & Killian (5). It is described in detail in ISO TR 11405 (2). The tooth part of the specimen and the material holder are fixed to metal rods to ensure alignment during both bonding and tensile loading (Fig. 1). A special material holder (cup) of transparent polystyrene resin for fixation to the metal alignment rods was developed to enable the use of light-curing materials.

Pre-treatment of the dentin surface and application of the adhesive were performed

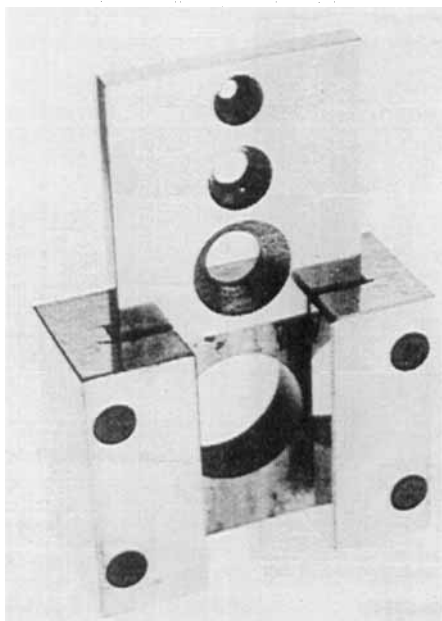


Fig. 2. Shear-testing apparatus.

in accordance with the manufacturers' instructions. When appropriate, the adhesive was cured with a curing lamp from the same producer (Table 1). The material cup was thereafter filled with a light-curing composite from the same manufacturer (Table 1), placed in correct position on the dentin surface, and light-cured from three different directions, each direction for 30 sec with the corresponding curing lamp (Table 1).

Shear bond strength test

The shear test used was originally proposed by Noguchi et al. (6). It is described in detail in ISO TR 11405. It consists of a solid block for fixation of the specimen and the shearing blade (Fig. 2).

Pre-treatment of dentin and application of the different adhesives were done in accordance with the manufacturers' instructions. Circular specimens 3 mm in diameter were made in a Teflon mold fixed to the dentin surface in a special jig. The mold was successively filled with a light-curing composite from the same manufacturer and cured in accordance with the manufacturer's instructions with the corresponding curing lamp.

The cylindrical adhesive/composite specimen is pushed through a hole in the shearing blade, which has a 0.5-mm blunt edge.

Storage of specimens

Two groups of specimens, one for tensile testing and one for shear testing, were stored for 24 h in water at 37°C. A third group of specimens (shear testing) was stored for 24 h in water at 37°C and thereafter thermocycled from 5°C to 55°C and back to 5°C 500 times. The exposure to each bath was 20 sec, and the transfer time between baths was 5 sec.

Statistics

A multiple-range test, the LSD procedure (SPSS/PC+, SPSS Inc.), was used to calculate differences between means (0.05 significance level).

Table 2. Number of specimens and range of bond strength values observed under the various testing conditions

Adhesive	24 h, tensile		24 h, shear		Thermocycled, shear	
	No.	Range	No.	Range	No.	Range
Gluma Bonding System	10	1.1-8.3	9	2.7-10.0	10	1.1-14.1
Scotchbond 2	9	1.8-23.5	9	4.7-16.7	9	1.8-7.4
Syntac	10	8.1-25.0	10	5.1-27.6	10	12.0-31.1
Scotchbond Multi-Purpose Dental Adhesive System	10	9.8-21.9	10	4.4-25.0	10	14.1-27.9

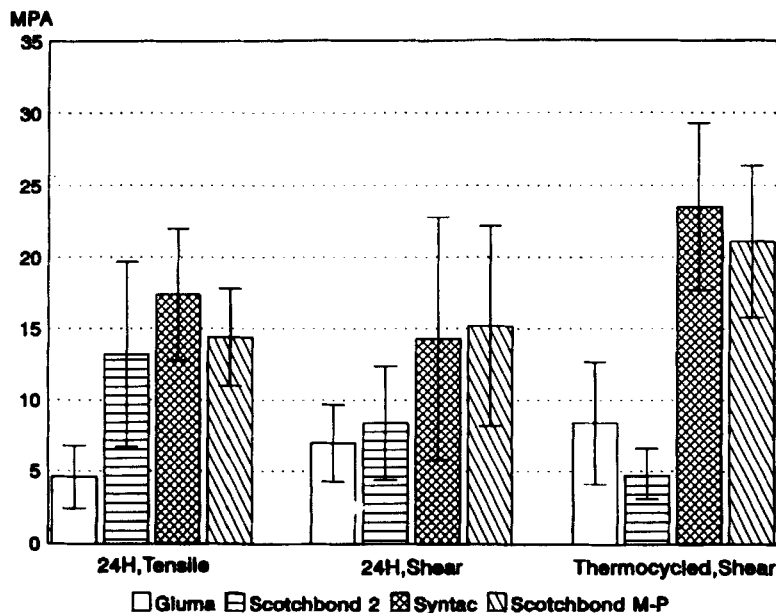


Fig. 3. Mean bond strength values and standard deviation.

Scanning electron microscope

The dentin part of some fractured specimens with the highest bond strength values from each group was inspected in a scanning electron microscope (Philips XL 30, Philips, Eindhoven, The Netherlands) after the fractured surface had been coated with a thin layer of gold/palladium.

Results

Bond strength measurements

The number of specimens and the range of measured bond strength values for different adhesive systems are given in Table 2. The mean bond strength values and standard deviations are given in Fig. 3.

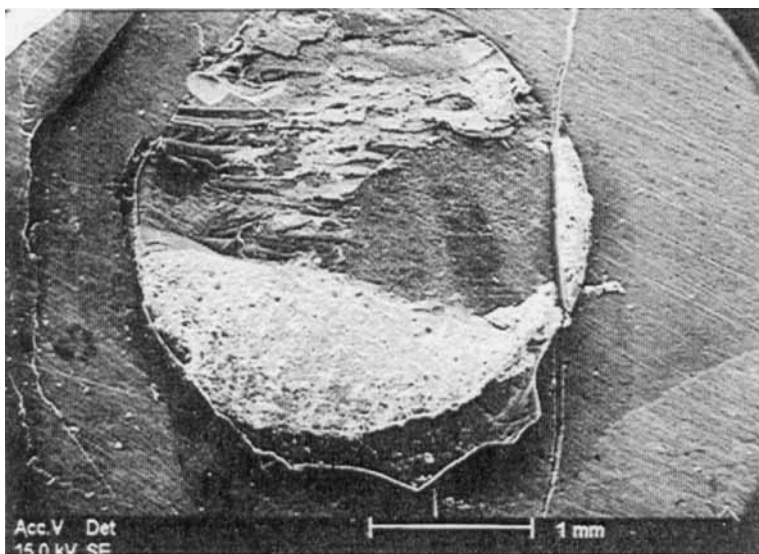


Fig. 4. Dentin part of a Scotchbond Multi-Purpose specimen fractured in shear. High bond strength value.

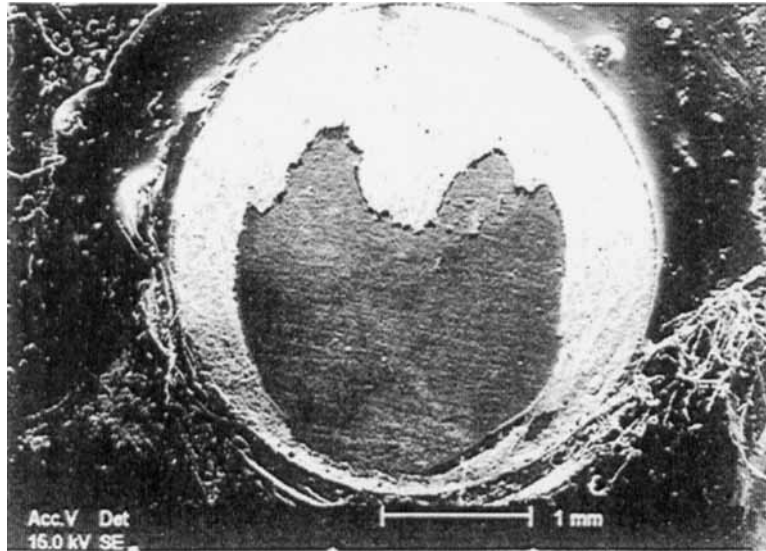


Fig. 5. Dentin part of a Syntac specimen fractured in tension. High bond strength value.

No significant differences were found for the values for Gluma under the three various testing conditions. Scotchbond 2 showed significantly lower bond strength values when tested with the shear test after thermocycling than when tested with the tensile test at 24 h.

For Scotchbond Multi-Purpose and Syntac, no significant differences were shown between values from tensile and shear tests after 24 h in water. For both materials, how-

ever, a significant increase in bond strength was observed after thermocycling.

Scanning electron microscope

Observations in the scanning electron microscope indicate that high bond strength values, both under tensile and shear load, were connected with an increasing amount of cohesive failure either in the composite

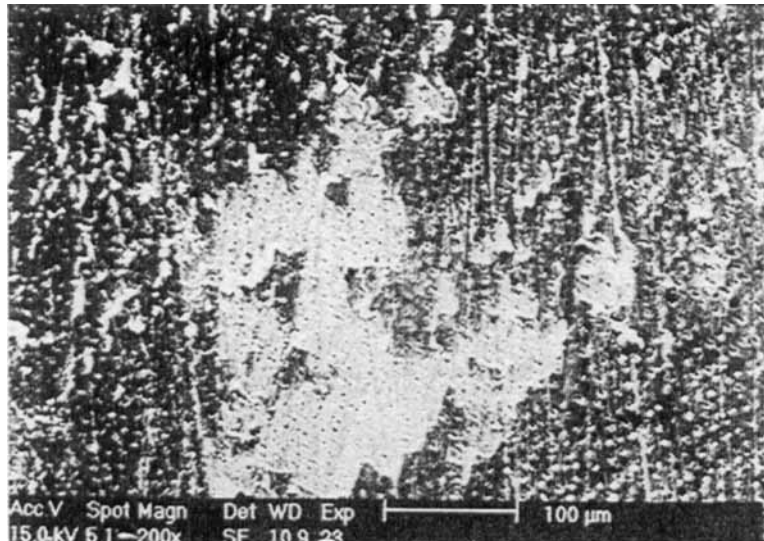


Fig. 6. Dentin surface of a Syntac specimen after fracturing in shear.

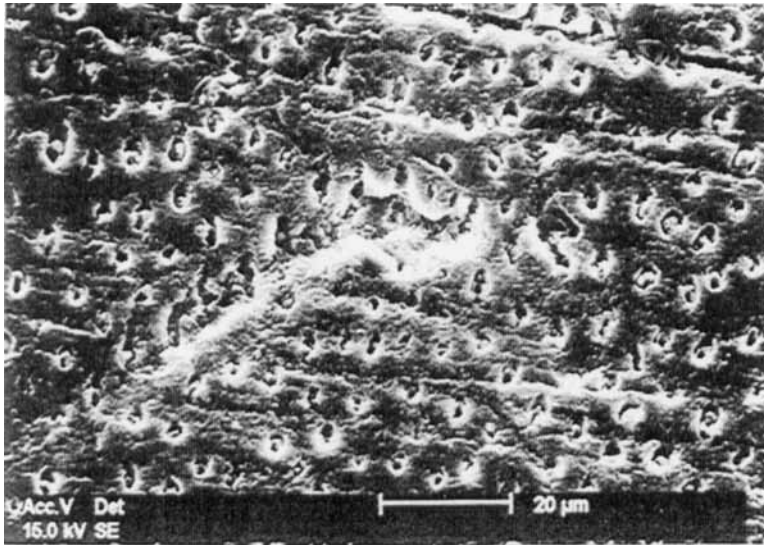


Fig. 7. Dentin surface of a Syntac specimen after fracturing in tension.

resin or the bonding resin layer (Figs. 4 and 5). In some specimens observations indicated either a rupture of a surface layer of the dentin under shear stress (Fig. 6) or a fracture pulling out pieces of the dentin under tensile stress (Figs. 7 and 8).

Discussion

The methods used in the present study are

thoroughly described in the ISO document. The variation is still large, with a coefficient of variation exceeding 50% in one series of specimens. The main reason is possibly related to the fact that it is impossible to standardize the dentin substrate. Large variations of the dentin structure are observed both within the same bonding area and from tooth to tooth (7).

The lack of significant differences between values obtained with the tensile and the shear

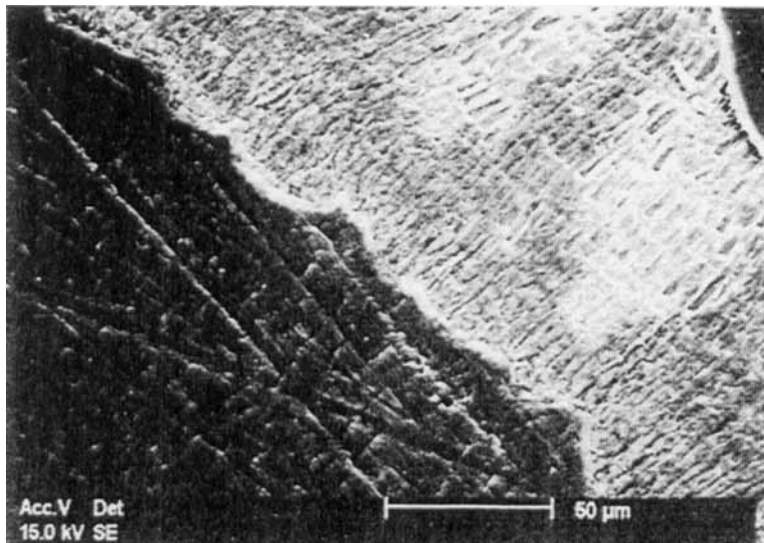


Fig. 8. Dentin surface of a Scotchbond Multi-Purpose specimen after fracturing in tension.

test methods for any of the materials indicates that the two methods are equally good for an in vitro test on the quality of a bond.

Both the mean and the range of measured bond strength values indicate that modern adhesives, in spite of a large variation, have improved bond strength properties. The studies of the fractured surfaces in the scanning electron microscope showing a mixture of cohesive and adhesive failures indicate, however, that the quality and probably also the type of bond vary across one and the same bonding area. The wetting and type or degree of reaction between the adhesive and dentin may vary with the quality of the substrate.

Several observations in the scanning electron microscope indicate a cohesive fracture in the dentin. In some specimens from the shear tests, a surface layer showing tubule openings and polishing grooves has obviously been ripped-off parts of the dentin surface (Fig. 6). It may be speculated that this layer is the decalcified surface layer of dentin infiltrated by resin, in which the so-called hybrid layer is formed (8). Observations in the tensile test specimens indicate a similar pattern. In specimens with high bond strength values and a fracture surface close to the interface, either a surface layer or pieces of dentin are pulled out from the dentin (Figs. 7 and 8). Similar fracture patterns have been reported previously (9). It may thus be concluded that when strong bonds are obtained, the solidity of the dentin or the connection between the hybrid layer and the underlying dentin may be the limiting factor for the strength of such bonds.

An interesting observation was the increase in the strength of the bond after thermocycling for two of the adhesives. It may be speculated that a relaxation of stresses from polymerization contraction due to thermal effects and water absorption is responsible for these observations. A swelling of the resin in the hybrid layer may also increase the strength of a mechanical bond

to the remaining dentin. The decrease in bond strength for Scotchbond 2 after thermocycling correlates with previous reports (10).

It may be concluded that the tensile and the shear test methods seem to give values of comparable magnitude and seem to be equally representative as an in vitro test for the quality of dentin adhesive systems.

Bond strength measurements indicate that modern adhesives have an increased bond strength value when compared with earlier products, and the bond is in certain areas of the dentin surface stronger than the dentin structure it adheres to. Microscopic inspections indicate, however, that the bond and its quality are unevenly distributed over the dentin surface.

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