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Resin composites: strength of the bond to dentin versus mechanical properties

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Abstract This study (1) investigated whether the combination of an adhesive system from one manufacturer with a resin composite from the same manufacturer provides superior bonding of the resin composite to dentin compared with the combination of an adhesive system from one manufacturer with a resin composite from another manufacturer, and (2) tested for a possible influence on bond strength of mechanical properties of the resin composite. After application of an adhesive system, a resin composite was bonded to flattened human dentin and tested in shear after 1 week. Five adhesive systems (AdheSE, Adper Prompt L-Pop, Clearfil SE Bond, Optibond Solo Plus, and Xeno III) were tested with each of five resin composites (Tetric Ceram, Filtek Supreme, Clearfil AP-X, Premise, and EsthetX). The mechanical properties flexural strength and flexural modulus were determined by three-point loading. Bond strengths were influenced by the brand of adhesive system (P < 0.0001) and by the brand of resin composite (P < 0.0001), but the combination of an adhesive system from one manufacturer with a resin composite from the same manufacturer did not provide bond strengths that were superior to those obtained when an adhesive system from one manufacturer was combined with a resin composite from another manufacturer. Independent of the brand of resin composite, the adhesive system Clearfil SE Bond mediated the highest bond strength to dentin. For each adhesive system, the resin composite Clearfil AP-X resulted in the highest bond strength to dentin. Significant positive correlations were found between bond strength and flexural

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strength (P<0.0026, r=0.21) and between bond strength and flexural modulus (P<0.0017, r=0.22).

Keywords Adhesion · Flexural strength · Modulus

Introduction

The bonding of resin composite to enamel and dentin is obtained through the use of an adhesive system. Adhesive systems have undergone tremendous development since the launching of the first systems in the late 1970s, and development continues with the aims of simplification and/or improvement. In vitro and in vivo studies have found the efficacy of adhesive systems to differ greatly between the various types and brands of systems [13, 21]. The efficacy has been shown to depend on numerous factors, such as infiltration of adhesive into the demineralized tissue [2, 3, 7, 9, 11, 20], degree of conversion and strength of the adhesive [8, 9, 12, 16, 23], mechanical properties of the resin composite [6, 24], and compatibility between the resin composite and the adhesive-treated dentin surface as regards sensitivity of the resin composite initiator system to an acidic environment [19] and as regards surface energy parameters [1].

The abundance of resin composites and adhesive systems on the market makes heavy demands on the clinician when he has to decide for specific types and brands of materials. Manufacturers tend to recommend that an adhesive system from one manufacturer be used with a resin composite from the same manufacturer. However, clinicians may have several reasons for wanting to combine an adhesive system from one manufacturer with a resin composite from another manufacturer. Thus, it is of relevance to know whether the combination of an adhesive system and a resin composite from the same manufacturer does indeed result in superior performance.

A recent study investigated the effect on bond strength of mixing adhesive systems and resin composites from different manufacturers [15]. The combinations of materials from one and the same manufacturer failed to show superiority to other combinations, but the study did not include analysis of explanatory factors. Likewise, another study found the strength of the bond to dentin promoted by two adhesive systems to vary with the brand of resin composite [14]. The influence of the brand of resin composite was explained in part by the base component of the surface energy of the resin composites. It seems warranted to search for determining factors that have even stronger influence and that may prove practicable as a selecting criteria by virtue of information being readily available to the clinician.

This study tested the null hypothesis that the brand of resin composite does not influence the strength of the bond to dentin mediated by an adhesive system. The aims were (1) to investigate whether the combination of an adhesive system from one manufacturer with a resin composite from the same manufacturer provides superior bonding of the resin composite to dentin compared with the combination of an adhesive system from one manufacturer with a resin composite from another manufacturer, and (2) to test for a possible influence on bond strength of the mechanical properties flexural strength and modulus of the resin composite.

Materials and methods

Table 1 lists the materials used and their manufacturers. To make the study useful to a large number of dentists, some of the most popular adhesive systems and resin composites were chosen. Further, adhesive systems that use fundamentally and clinically different approaches were chosen. Thus, the adhesive systems were of the following types: one twostep etch-and-rinse system (Optibond Solo Plus), two twostep self-etch systems (AdheSE and Clearfil SE Bond), and two one-step self-etch systems (Adper Prompt L-Pop and Xeno III).

Table 1 Adhesive systems and resin composites tested

Adhesive system	Composite	Manufacturer
AdheSE	Tetric Ceram	Ivoclar Vivadent
Adper Prompt L-Pop	Filtek Supreme	3M ESPE
Clearfil SE Bond	Clearfil AP-X	Kuraray Dental
Optibond Solo Plus	Premise	sds Kerr
Xeno III	EsthetX	Dentsply DeTrey

Bond strength

Non-carious human permanent molars that had been stored in 0.5% chloramine at room temperature since extraction were embedded in an auto-curing resin (Epofix; Struers, Copenhagen, Denmark). After setting, the mesial or distal surface was wet-ground on silicon carbide paper #1000 to produce a flat dentin surface. The 200 prepared teeth were randomly divided into 25 groups of 8 teeth. Immediately after grinding, the dentin surfaces were treated with an adhesive system according to the manufacturer's instructions. A split Teflon mold (diameter=3.6 mm, height= 2.5 mm) was clamped to the adhesive-treated surface and filled with a resin composite. The resin composite was lightcured for 40 s with an ESPE Elipar Highlight unit (power density=650 mW/cm² as determined by an LED Radiometer, Demetron, sds Kerr). Ten minutes after the completion of the light-curing, the bonded specimens were freed from the mold and stored in water at 37°C for 1 week. The strength of the bonds was determined in shear by the use of a universal testing machine (model 5566, Instron, High Wycombe, UK) operating at a crosshead speed of 1 mm/min.

Flexural strength and modulus

The resin composite was inserted into a split brass mold $(25 \times 2 \times 2 \text{ mm})$, covered with transparent matrix strips and a glass microscope slide and light-cured (Elipar Highlight) from one side with five overlapping footprints of 20 s each. The light-curing procedure was repeated on the opposite surface of the specimen. Fifteen minutes after start of cure, the resin composite specimen was freed from the mold, gently wet-ground on silicon carbide paper #320 to remove any flash, and transferred to a water bath at 37°C. After 24 h, the height (a, mm) and width (b, mm) of each specimen were measured, and the specimen was then subjected to three-point loading with l=20 mm between the supports. The crosshead speed of the universal testing machine (model 5566, Instron) was 0.75 mm/min. Flexural strength (S, MPa) was calculated as $S=3Fl/(2a^2b)$ where F (in N) is the force at fracture. Flexural modulus (E, MPa) was calculated as $E=\alpha l^3/(4a^3b)$ where α is the slope (in N/mm) of the straight-line relationship between force and deflection of the resulting flexural curve. Eight specimens were produced from each resin composite, and a mean value and standard deviation were computed for each resin composite.

Statistics

Statistical evaluation of the bond strength data was performed using two-factorial analysis of variance (ANOVA; SAS 9.1 software, SAS Institute, Cary, NC, USA) to identify effects of adhesive system and resin composite. Flexural strength and modulus data were analysed by one-way ANOVAs. All three ANOVAs were followed by Newman–Keuls' multiple range tests. Regression analyses were performed between bond strength and flexural strength data and between bond strength and flexural modulus data (SAS 9.1 software). Statistical significance was considered as P<0.05.

Results

The results of the bond strength and flexural tests are shown in Table 2, and the results of the two-factorial ANOVA are shown in Table 3. The factor adhesive system as well as the factor resin composite had significant effect on bond strength (P < 0.0001). There was no significant interaction between the two factors. Highest bond strengths were obtained with Clearfil SE Bond followed by AdheSE. Adper Prompt L-Pop, Xeno III, and Optibond Solo Plus yielded lower and almost similar bond strengths. With respect to resin composite, Clearfil AP-X constantly resulted in the highest bond strengths. Flexural strength values varied with statistical significance as did the flexural modulus values (P < 0.0005). Clearfil AP-X yielded the highest flexural strength and flexural modulus, while Premise yielded the lowest flexural strength and flexural modulus. A statistically significant positive correlation was found between bond strength (single values) and flexural strength (mean values; P < 0.0026, r =0.21). Likewise, a statistically significant positive correlation was found between bond strength (single values) and flexural modulus (mean values; P < 0.0017, r = 0.22).

Discussion

This study found the strength of the bond between resin composite and dentin to be influenced by the adhesive system used to mediate the bond and by the resin composite.

 Table 3 Results of the two-factorial ANOVA, dependent variable:

 bond strength

Effect	SS	df	ms	F	Р
Adhesive system	14,406.46	4	3,601.62	121.33	< 0.0001
Resin composite	1,123.66	4	280.91	9.46	< 0.0001
Adhesive system x resin composite	537.16	16	33.57	1.13	0.3299
Error	5,194.62	175	29.68	_	_

This implies that the null hypothesis, stating that brand of resin composite does not influence the strength of the bond to dentin mediated by a given adhesive system, has to be rejected. As no interaction between adhesive system and resin composite was found, it also implies that the combination of an adhesive system from one manufacturer with a resin composite from the same manufacturer did not consistently show superior bond strengths, e.g. as a result of unique chemical compatibility between the two materials. This non-superiority of "one-manufacturer combinations" is in line with the findings of previous studies [14, 15], and it indicates that the characteristics of adhesive systems and resin composites per se are more important for bond formation than the issues of compatibility between the two materials.

The two-step self-etch systems showed higher bond strengths than did the one-step self-etch and the two-step etch-and-rinse adhesive systems. The superior bond-mediating capacity to dentin of the two-step self-etch systems corroborates findings of previous in vitro and in vivo studies [10, 13, 15, 17, 21] and is believed to result from a number of factors, one of the most important being the simultaneous demineralization and infiltration of dentin. These two simultaneous actions lead to a shallow but uniform resin-infiltrated dentin

Table 2 Strength of the bond to dentin obtained with the five respective adhesive systems (MPa), flexural strength (MPa), and flexural modulus(GPa) of the five respective resin composites (means \pm SD)

Property	Adhesive system	Resin composite				
		Tetric Ceram	Filtek Supreme	Clearfil AP-X	Premise	EsthetX
Bond strength	AdheSE	27±6 c	29±6 cd	32±7 cde	20±5 b	27±6 c
	Adper Prompt L-Pop	15±5 ab	19±6 b	21±8 b	15±5 ab	17±5 ab
	Clearfil SE Bond	36±6 de	36±3 de	39±7 e	33±4 cde	32±9 cde
	Optibond Solo Plus	12±2b ab	12±4 ab	18±6 ab	12±3 ab	13±3 ab
	Xeno III	14±4 ab	10±6 a	19±5 b	14±5 ab	13±4 ab
Flexural strength		111±4 b	137±6 c	161±12 d	89±11 a	117±13 b
Flexural modulus		8.3±0.8 b	10.6±0.7 c	15.3±1.1 d	$7.3 {\pm} 0.8$ a	10.0±0.5 c

The combinations marked in bold used an adhesive system and a resin composite from the same manufacturer. For each of the three properties, identical letters indicate that mean values did not differ with statistical significance.

layer throughout which the residual hydroxyapatite remains available for chemical interaction [18, 21].

Within the group of the two-step self-etch adhesive systems, Clearfil SE Bond showed higher bond strengths than did AdheSE. This finding is in harmony with that of other studies [4, 21]. Based on pH measurements, these studies have categorized Clearfil SE Bond as a "mild" selfetch adhesive and AdheSE as an "intermediary strong" adhesive, the latter displaying a thicker hybrid layer and more pronounced resin tags instead of hybridized smear plugs as typically observed with Clearfil SE Bond.

Several explanations have been offered for the general finding that one-step self-etch systems show poorer performance than two-step self-etch systems despite that both types of adhesive systems simultaneously demineralize and infiltrate the dentin [4, 10, 13, 15, 21]. These explanations include inhibition of polymerization in the subsequently applied resin composite due to high acidity of the adhesive, too thin an adhesive layer resulting in incomplete cure and in varying thickness or even in local absence of the adhesive layer, and phase separation between hydrophilic and hydrophobic adhesive ingredients resulting in increased susceptibility to hydrolysis.

The five resin composites varied significantly as regards flexural strength and flexural modulus, with Clearfil AP-X being the strongest and stiffest. The main explanatory factor for the significant differences in mechanical properties is filler load: Properties such as flexural strength and modulus are strongly influenced by the filler concentration of the resin composite [22]. In support of the present finding, other studies have also found Clearfil AP-X to display very high strength and stiffness [5, 6, 15], first and foremost, as a result of an exceptionally high filler load.

A positive correlation was found between the strengths of the bond to dentin mediated by the adhesive systems and the flexural strength or flexural modulus, respectively, of the resin composites tested. This finding supports that of previous investigations [6, 24]. Furthermore, as information about strength and modulus of resin composites is readily accessible, the finding also supports the capacity of strength and stiffness to serve as selecting criteria in search for high bonding potential.

It may be concluded that combining an adhesive system from one manufacturer with a resin composite from the same manufacturer did not guarantee optimal performance with respect to bond strength. Instead, one specific resin composite, viz., the strongest and stiffest, and one specific adhesive system, a two-step self-etch system, consistently provided the highest bond strengths to dentin.

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References

- Asmussen E, Peutzfeldt A (2005) Resin composites: strength of the bond to dentin versus surface energy parameters. Dent Mater 21:1039–1043
- Carvalho RM, Mendonca JL, Santiago SL, Silveira RR, Garcia FCP, Tay FR, Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin. J Dent Res 82:597–601
- De Munck J, van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P, Vanherle G (2003) Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res 82:136–140
- De Munck J, Vargas M, Iracki J, van Landuyt K, Poitevin A, Lambrechts P, van Meerbeek B (2005) One-day bonding effectiveness of new self-etch adhesives to bur-cut enamel and dentin. Oper Dent 30:39–49
- Ferracane JL, Ferracane LL, Musanje L (2003) Effect of light activation method on flexural properties of dental composites. Am J Dent 16:318–322
- Hasegawa T, Itoh K, Koike T, Yukitani W, Hisamitsu H, Wakumoto S, Fujishima A (1999) Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system. Oper Dent 24:323–330
- Hashimoto M, Ohno H, Endo K, Kaga M, Sano H, Oguchi H (2000) The effect of hybrid layer thickness on bond strength: demineralized dentin zone of the hybrid layer. Dent Mater 16:406–411
- Ito S, Tay FR, Hashimoto M, Yoshiyama M, Saito T, Brackett WW, Waller JL, Pashley DH (2005) Effects of multiple coating of two all-in-one adhesives on dentin bonding. J Adhes Dent 7:133– 141
- Jacobsen T, Söderholm KJ (1995) Some effects of water on dentin bonding. Dent Mater 11:132–136
- Kaaden C, Powers JM, Friedl KH, Schmalz G (2002) Bond strength of self-etching adhesives to dental hard tissues. Clin Oral Investig 6:155–160
- 11. Pashley DH, Ciucchi B, Sano H, Homer JA (1993) Permeability of dentin to adhesive agents. Quintessence Int 24:618–631
- Pashley DH, Tay FR (2001) Aggressiveness of contemporary selfetching adhesives. Part II: etching effects on unground enamel. Dent Mater 17:430–444
- Peumans M, Kanumilli P, de Munck J, van Landuyt K, Lambrechts P, van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater 21:864–881
- 14. Peutzfeldt A, Asmussen E (2004) Determinants of in vitro gap formation of resin composite. J Dent 32:109–115
- Roh BD, Chung JH (2005) Micro-shear bond strength of five resin-based composites to dentin with five different dentin adhesives. Am J Dent 18:333–337
- Takahashi A, Sato Y, Uno S, Pereira PN, Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin. Dent Mater 18:263–268
- Tanumiharja M, Burrow MF, Tyas MJ (2000) Microtensile bond strengths of seven adhesive systems. Dent Mater 16:180– 187
- Tay FR, Pashley DH (2001) Aggressiveness of contemporary selfetching systems. I. Depth of penetration beyond dentin smear layers. Dent Mater 17:296–308
- Tay FR, Pashley DH, Yiu CKY, Sanares AM, Wei SHY (2003) Factors contributing to the incompatibility between simplifiedstep adhesive and chemically cured or dual-cured composites. Part I. Single-step self-etching adhesive. J Adhes Dent 5:27–40
- 20. Van Meerbeek B, Yoshida Y, Snauwaert J, Hellemans L, Lambrechts P, Vanherle G, Wakasa K, Pashley DH (1999)

Hybridization effectiveness of a two-step versus a three-step smear layer removing adhesive system examined correlatively by TEM and AFM. J Adhes Dent 1:7-23

- 21. Van Meerbeek B, de Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, van Landuyt K, Lambrechts P, Vanherle G (2003) Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent 28: 215–235
- 22. Willems G, Lambrechts P, Braem M, Vanherle G (1993) Composite resins in the 21st century. Quintessence Int 24:641–658
- Zheng L, Pereira PNR, Nakajima M, Sano H, Tagami J (2001) Relationship between adhesive thickness and microtensile bond strength. Oper Dent 26:97–104
- Zidan O, Asmussen E, Jörgensen KD (1980) Correlation between tensile and bond strength of composite resin. Scand J Res 88: 348–351

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