# **ORIGINAL ARTICLE**

# Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin in vitro

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Received: 15 August 2008 / Accepted: 8 April 2009 / Published online: 9 May 2009 © Springer-Verlag 2009

Abstract Self-adhesive resin cements should ease the placement of dental restorations. The purpose of this study was to evaluate their shear bond strength to enamel and dentin. Sixty molars were randomly assigned to 12 test groups (each n=10), and the approximal surfaces were ground flat to get an enamel and dentin surface with a diameter of at least 4 mm. Ceramic specimens were bonded to the surfaces with either Variolink/Syntac Classic (VSC), Panavia F2.0 (PAF), RelyX Unicem (RLX), Maxcem Elite (MCE), iCem (IC), or an experimental self-adhesive resin cement (EXP). The shear bond strength (crosshead speed: 1 mm/min) was measured after 24-h storage in NaCl (37°C). The fracture modes were determined with a stereomicroscope (magnification, 8-50-fold). VSC had the highest shear bond strength within the enamel groups (42.9 $\pm$ 9 MPa) and IC the lowest (10.5 $\pm$ 4.2 MPa, p<0.001). The highest dentin shear bond strength was determined for VSC (39.2± 8.9 MPa, p < 0.001) and the lowest for EXP (7.8± 3.9 MPa, p<0.001). Self-adhesive resin cements fractured mainly between resin and enamel or dentin. The shear bond strength of self-adhesive resin cements was inferior compared to conventional composite resin cements.

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**Keywords** Self-adhesive resin cements · Shear bond strength · Adhesion · Enamel · Dentin

#### Introduction

Improved material properties of ceramic restorations and a reliable adhesive bond between ceramic and dental tissues lead to a growing interest and increased use of ceramic restorations compared to cast restorations [5, 23]. Clinical reviews and meta-analyses showed that there were no significant differences between ceramic and cast partial crowns regarding the survival rates [15, 24, 31] with stable clinical results after appropriate cementation even for large cavities with cusp replacement or dentin margins [22]. The adhesive procedure with a composite resin is mandatory for the insertion of ceramic restorations [7]. Because of their water uptake and swelling, hydrophilic cements, like componer cements, will induce fracture of the ceramic restoration material and therefore are inappropriate for the cementation of these restorations [27].

Compomer and composite cements used for indirect restorations can be classified as "active" materials because of an adhesive interaction with the dentin under formation of a hybrid layer and their bond to dental materials. Materials that achieve the retention of the restoration by mechanical interlocking between rough surfaces and the cement are called "passive" [8]. Conventional materials such as zinc phosphate, zinc polycarboxylate, and glass ionomer cements are "passive" materials because of their lack of adhesive bond to dental materials. Although for glass ionomer cements the formation of an intermediate layer was observed, which can be considered as an adhesive interaction with dentin [10, 33], these materials do not bond to dental materials mediated by etching with hydroflouric acid or



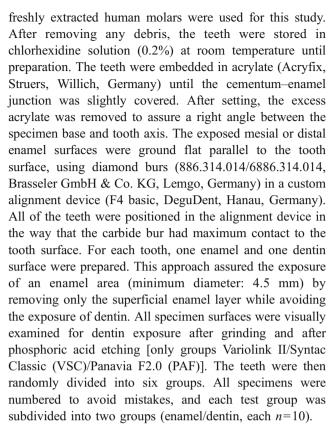
silication and silanization. "Active" cements can be cured by chemical or light polymerization; dual-curing materials combine both curing characteristics in one material. Despite their good mechanical properties, acrylic resins are used less frequently than conventional cements [6]. A possible explanation for instance can be the more time-consuming adhesive procedure, while using an adhesive system with multiple application steps, increased technique sensitivity and incompatibility between resin cements and one-step selfetch adhesives [9, 30]. Hence, resin cements based upon phosphoric-acid-modified acrylates, for example RelyX Unicem (RLX) as the first so-called self-adhesive material (3 M Espe, Seefeld), were developed. Due to their low pH value after mixing of two components, an adhesive bond to enamel and dentin is achieved in a self-adhesive procedure, e.g., without the use of an adhesive system. The enamel bond strength of self-adhesive resins is lower compared to conventional resin cements; in dentin, there are no significant differences between the materials [2, 11, 19]. The bond strength to enamel can be increased to the same level as that of a conventional adhesive resin cement combined with an adhesive system by additional phosphoric acid etching. This etching procedure should be limited to the enamel because it has adverse effects on the bond strength when extended to the dentin [11]. Compared to glass ionomers, the bond strength to enamel and dentin observed in vitro is higher after thermocycling for self-adhesive resin cements. Due to Abo-Hamar et al. [2], self-adhesive resin cements can be used for the cementation of high-strength ceramics or cast restorations as well as for ceramic restorations with extensive dentin exposure and lack of enamel after preparation. Research about marginal seal of Empress 1-veneers showed similar outcomes for adhesive resin cements compared to self-adhesive materials regarding dentin margins; the enamel margins revealed more marginal defects [21]. Another survey showed similar results; the marginal adaptation of self-adhesive materials was comparable to well-established conventional adhesive cements [3]. The marginal adaptation and the bond strength of self-adhesive materials to dentin were not improved by the additional use of an adhesive system [19, 21].

Objectives were to determine the effectiveness of different self-adhesive cements in bonding a lithium disilicate ceramic to enamel and dentin.

The null hypothesis set forth was that there is no difference between the materials regarding the shear bond strength.

# Materials and methods

In order to obtain enamel and dentin surfaces for cementation of the ceramic specimens, 60 caries-free,



Afterwards, ceramic specimens were made of leucite reinforced ceramic (Cergo, DeguDent, Hanau, Germany, Color B2). In order to simulate the roughness of the lower surface of a ceramic restoration after embedding, the surface of the ceramic specimen that was assigned for adhesive cementation was sandblasted (Al<sub>2</sub>O<sub>3</sub>; particle size: 50 µm) and pre-treated for cementation according to the manufacturer's instructions [etching with hydrofluoric acid 5% for 4 min (Vita Ceramics Etch, Vita, Bad Säckingen, Germany) and silane treatment with Monobond S (Ivoclar Vivadent, FL-Schaan)/Clearfil Ceramic Primer (Kuraray, Okayama, Japan)]. Then, the specimens were attached to the tooth surfaces using "calibrated fingerpressure" (about 20 g/mm<sup>2</sup>) as determined by Goracci et al. [18]. Therefore, specimens were loaded with a small weight (250 g) adjusted to the specimens surface (12.56 mm<sup>2</sup>) after the cement excess was removed with foam pellets (Pele Tim, Voco, Cuxhaven, Gemany). The specifications of all materials and their application steps within the adhesive procedure are listed in Table 1. For the group VSC, the adhesive system was not polymerized until the resin cement was applied. In order to avoid adverse effects on bond strength caused by the application of "Primer" and "Adhesive," only two application steps (phosphoric acid etching and Heliobond) were used for bonding to the enamel surface. This procedure was performed in order preserve the micro-retentive surface and to eliminate any effects that could be detrimental for the adhesive bond. It



Table 1 Cements, manufacturers, and their application

Cement/adhesive system	Code	Group	Application	Manufacturer
Variolink II/Syntac Classic	VSC	Conventional with adhesive system	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, silanization (60 s), Heliobond (without polymerization), tooth: enamel conditioning 30 s (H <sub>3</sub> PO <sub>4</sub> 37%), rinsing, drying, Primer (15 s), Adhesive (10 s), Heliobond (without polymerization), cement: mixing of base/catalyst 1:1, applying on ceramic, removing of cement excess, polymerization	Ivoclar FL-Schaan,
RelyX Unicem	RLX	Self-adhesive	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, silanization (60 s), cement: mixing, applying on ceramic, removing of cement excess, polymerization	3 M Espe, Seefeld, Germany
Maxcem elite	MCE	Self-adhesive	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, silanization (60 s), Tooth: drying (no excessive drying) cement: mixing, applying on ceramic, removing of cement excess, polymerization	Kerrhawe, CH- Bioggio
V 35973	EXP	Self-adhesive	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, silanization (60 s), tooth: drying (no excessive drying), no water layer on surface, cement: mixing, applying on ceramic, removing of cement excess, polymerization	Voco, Cuxhaven, Germany
Panavia F2.0	PAF	Conventional with adhesive system	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, appliance of Clearfil Ceramic Primer, tooth: enamel conditioning 10 s (K Etchant Gel), mixing of ED Primer II 1:1, leave for 30 s, cement: mixing 1:1, applying on ceramic, removing of cement excess, polymerization	Kuraray, Okayama, Japan
iCem	IC	Self-adhesive	Ceramic: conditioning (HF 5%, 4 min), rinsing (1 min), drying, silanization (60 s), tooth: drying (no excessive drying), surface has to be slightly moisturized, cement: mixing, applying on ceramic, removing of cement excess, polymerization	Heraeus, Hanau, Germany

was stated by Woronko et al. [32] that the application of a primer showed a tendency to decrease enamel bond strengths. The rubbing application of a primer to etched enamel surfaces could decrease enamel bond strength up to 40% because the microretentive pattern was affected [16].

The resin cements were light cured overlapping with a light-emitting diode (LED) unit (Bluephase, Ivoclar Vivadent, FL-Schaan) for 20 s at 1,100 mW/cm² from the top and three lateral surfaces. After each curing cycle, the power output of the LED unit was tested with the instrument immanent testing device to assure identical conditions for each all samples. The weight was removed after the three lateral curing cycles were completed.

The samples were then stored in distilled water for 24 h at 37°C before the determination of the shear bond strength. Afterwards, all specimens were transferred from the water to a computer-controlled universal testing machine (Type 20K, Firma UTS, Ulm, Germany) and mounted in the testing device. The distance between the crosshead and the tooth surface was 0.1 mm at the resin–ceramic interface. All speci-

mens were loaded at a crosshead speed of 1 mm/min until fracture occurred. The shear bond strengths were determined by dividing the maximum load by the covered enamel area (diameter: 4 mm). The fractured surfaces of the specimens were examined using a light-optical microscope (Stemi SV 6, Firma Zeiss, Jena, Germany) at 8- and 50-fold magnification.

For statistical analysis, the mean values for each group were calculated. For comparison within the groups regarding enamel and dentin surfaces, t test with Levene's test for equality of variances was used (p<0.05). Furthermore, the one-way analysis of variance and Scheffé test at a level of significance of p<0.05 was performed for each enamel and dentin group.

## Results

The highest shear bond strength regarding the enamel surfaces was measured for VSC (42.9±9 MPa), the lowest for iCem (IC; 10.5±4.2 MPa). Regarding dentin, VSC had



the highest  $(39.2\pm8.9 \text{ MPa})$  and the experimental selfadhesive resin cement (EXP) the lowest shear bond strength (7.8±3.9 MPa). Mean values and standard deviations are shown in Tables 2 and 3. For PAF, EXP and Maxcem Elite (MCE), there were significant differences between the materials for enamel and dentin surfaces (p < 0.05/p <0.001). Regarding the enamel surfaces, VSC was superior to all other test groups (p < 0.05/p < 0.001). Among the selfadhesive resin cements, RLX (23.0±6.3 MPa) and MCE (22.3±3.3 MPa) had the highest shear bond strength values but only being significant compared to IC (p<0.005). For dentin, the highest values were recorded for RLX regarding the self-adhesive resin cements (18.4±5.0 MPa) with significant differences only between RLX and EXP (<0.05). When comparing shear bond strength values dependent on the tooth surfaces (enamel vs. dentin), the bond strength for all materials except IC was higher in enamel than in dentin. All results are shown in Fig. 1.

The results of the fracture analysis are presented in Fig. 2. For the self-adhesive resin cements (RLX, EXP, IC, and MCE), nearly all fractures were adhesive and occurred between cement and tooth surface. For VSC and PAF, the distribution of the fracture modes was heterogeneous with no preference for one fracture type, and no adhesive fracture between cement and tooth surface was observed.

### Discussion

Self-adhesive resin cements were developed in order to reduce the number of application steps and technique sensitivity compared to conventional resin cements while obtaining comparable results [29]. In contrast to this claim, our results indicate higher shear bond strength values for conventional resin cements independent of the tooth surface, i.e., dentin or enamel. Based on our data, the null hypothesis set forth that there is no difference between the materials regarding the shear bond strength has to be rejected. Regarding bond strength to enamel, which was inferior for the self-adhesive systems, Abo-Hamar et al. [2]

**Table 2** Test results for enamel surfaces; means with the same superscript are not significantly different (p<0.05, Scheffé multiple comparisons)

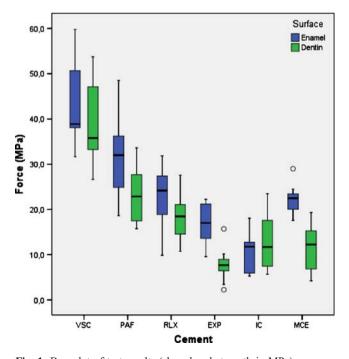
Cement	Mean	Standard deviation	N
VSC	42.9 <sup>a</sup>	9.0	9
PAF	31.2 <sup>b</sup>	8.5	10
RLX	23.0 <sup>b,c</sup>	6.3	10
MCE	22.3 <sup>b,c</sup>	3.3	10
EXP	16.9 <sup>c,d</sup>	4.5	10
IC	10.5 <sup>d</sup>	4.2	10

**Table 3** Test results for dentin surfaces; means with the same superscript are not significantly different (p<0.05, Scheffé multiple comparisons)

Cement	Mean	Standard deviation	N
VSC	39.2ª	8.9	10
PAF	23.4 <sup>b</sup>	6.5	10
RLX	18.4 <sup>b,c</sup>	5.0	10
MCE	11.4 <sup>c,d</sup>	5.8	9
EXP	$7.8^{\mathrm{d}}$	3.9	9
IC	12.8 <sup>c,d</sup>	5.8	10

found similar results with a conventional resin cement showing higher bond strength values than a self-adhesive resin cement after 24 h water storage. In another study, RLX also had lower bond strength values compared to Variolink/Syntac [19].

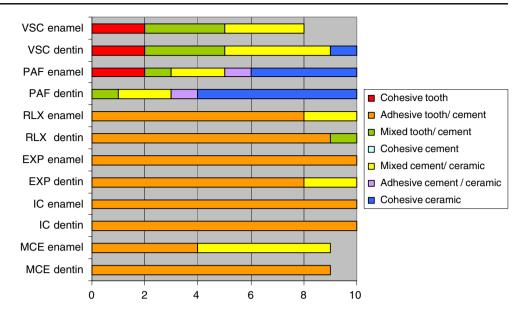
Our data indicated that the self-adhesive resin cements had significantly lower shear bond strengths when bonded to dentin surfaces. Other studies found no significant differences between self-adhesive resin cements and conventional resin cements [11, 19]. Escribano et al. reported results that are similar to our investigation, but unlike our findings, the lowest bond strength values were determined for RLX [12]. In contrast to our study, where the lowest dentin shear bond strength was determined for the experimental material (EXP), Holderegger et al. found the lowest bond strength values for RLX after 24 h water



 $\textbf{Fig. 1} \ \, \text{Box plot of test results (shear bond strength in MPa)}$ 



Fig. 2 Fracture modes of tested cements



storage when investigating one self-adhesive and three conventional resin cements [20]. The shear bond strength for RLX that was determined in our investigation was twice as high compared to [20], which could be explained by the type of polymerization (no light polymerization but only self curing was used).

For MCE, which is, according to the manufacturer, an improved version of MC, no significant differences in dentin were determined compared to RLX. In contrast to this finding, Goracci et al. showed that MC had a significantly lower microtensile bond strength (MTBS) than RelyX for enamel and dentin [18]. This was independent of the pressure that was applied on the specimens during cementation. There were no signs of micromechanical interlocking within enamel; in dentin, no hybrid layer formation was detected by scanning electron microscopy (SEM), but smear plugs were found. For adhesive cementation of orthodontic brackets, MC showed significant lower shear bond values than the control [4]. Furthermore, it was demonstrated that MC had lower retentive strength values after thermocycling than RLX when being autopolymerized [13]. The marginal integrity of ceramic restorations after luting showed a higher amount of gap free margins for etch & rinse systems than for resin cements combined with a self-etch adhesive or self-adhesive resin cements. Among the self adhesive resin cements, RLX performed better than MC in enamel and dentin [17].

Other investigations analyzed the influence of a pretreatment in terms of phosphoric acid etching on the bond between tooth and resin cement in order to improve the clinical behaviour. Regarding enamel, an additional phosphoric acid etching could improve bond strength; in dentin, it is detrimental to the bond and should be avoided [11, 19]. This may be due to the high viscosity of the materials, which causes an incomplete resin infiltration of the demineralised collagen network. SEM showed that those areas were existent after phosphoric acid pre-treatment [11].

Regarding microleakage of ceramic restorations, the additional application of an adhesive system when using self-adhesive resins could improve marginal integrity in enamel, but dentin margins showed a higher permeability that was not existent without adhesive pre-treatment [21]. In class II cavities that were treated with gold or ceramic restorations, RLX had comparable or better results for both types of restorations with Variolink/Excite [11].

With respect to the MTBS, an additional surface treatment with adhesives leads to significant higher bond strength values in dentin compared to the self-adhesive resin alone. For enamel surfaces, no significant differences were found [19].

In contrast to some of the cited studies that used the MTBS test, in our study, the shear bond strength was determined. If the bond strength to tooth surfaces is analyzed in vitro, different methods are available. The surfaces in this study had to be >4 mm in diameter; the shear bond strength was measured because this method is appropriate for testing occlusal and approximal surfaces with such diameters [28]. The MTBS test has small bonding areas with a size 0.8–1.0 mm<sup>2</sup>, and there is also a linear correlation between bond strength and specimen location regarding tooth surface [25]. A possible advantage of the shear bond strength test is that, this method, compared to the MTBS, is easy to perform. Nevertheless, it has to be taken into account that with increasing bond strength values, the chance for cohesive failures with fractures in dentin increases due to a non-uniform load distribution [26]. It is therefore difficult to compare the outcomes of MTBS and shear bond strength studies because identical adhesive systems produce higher bond strength values when the MTBS test was used [1]. Therefore, absolute values determined with these different methods



cannot be compared, but conclusions concerning a ranking of each material can be drawn from both methods [29].

The fracture analysis of the self-adhesive resin cements showed mainly adhesive fractures between cement and tooth surface. Therefore, this area seems to be the weak link of the bond. Similar to our results, RLX showed mainly adhesive fractures between cement and enamel [19]. In contrast to our survey, where the results for dentin also showed mainly adhesive fractures between tooth and cement, Hikita et al. [19] found heterogeneous results. SEM studies showed only little and superficial interaction between resin and dentin without hybrid layer formation for the self-adhesive materials [11, 18]. Furthermore, our microscopic evaluation showed several voids within the cement for RLX, which was also confirmed by an SEM evaluation with RLX showing a porous structure with voids within the cement layer [14, 18].

The fracture modes in our study were heterogeneous for VSC/PAF regarding enamel and dentin. This finding is similar to the data generated by Hikita et al. [19] with enamel surfaces. However, dentin surfaces showed 50% adhesive fractures between cement and tooth and mixed fractures with cohesive failure in dentin, respectively.

#### **Conclusions**

Self-adhesive resin cements are promising materials for luting indirect restorations because of their simplified application and reduced technique sensitivity. Nevertheless, the available data shows the need for improvement regarding the bond strength compared to conventional resin cements. Some studies showed promising results regarding microleakage, but clinical long-term studies are necessary in order to evaluate the in vivo performance of self-adhesive resin cements.

**Acknowledgement** Our thanks are due to Dr L. Hoy, Department of Biometry, Hannover Medical School, for his help with the statistical analysis of our data and to Schneemann Zahntechnik, Hannover-Langenhagen, Germany for their help with the fabrication of the ceramic specimens.

**Conflict of interest** The authors declare that they have no conflict of interest.

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