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# Shear bond strength of resin cements to human dentin

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ABSTRACT

*Objectives.* The objectives of this in vitro study were (1) to assess the bond strength of the universal cement RelyX Unicem to dentin and to compare it with three conventional resin cements, (2) to test the influence of aging on their bonding capacity and (3) to test the influence of the operator on bonding quality by performing the same test in two different centers.

*Methods.* 160 third molars, divided into 80 for tests at the University of Zurich (Z) and 80 for tests at the University of Berne (B), were assigned to $2 \times 8$ subgroups of 10 teeth each. The specimens were prepared with the corresponding bonding agents and acrylic rods were luted either with RelyX Unicem (U), RelyX ARC (A), Multilink (M) or Panavia 21 (P). All specimens were stored in water for 24 h (W) and half of the specimens were subjected to 1500 cycles of thermocycling (5 °C and 55 °C) (T). Bond strength was measured by means of a shear test.

*Results.* After water storage RelyX Unicem exhibited lowest bond strength (UWZ: $9.2 \pm 1.6$ MPa, UWB: $9.9 \pm 1.2$ MPa, AWZ: $15.3 \pm 6.0$ MPa, AWB: $12.2 \pm 4.3$ MPa, MWZ: $15.6 \pm 3.3$ MPa, MWB: 12.4 MPa $\pm$ 2.4, PWZ: $13.4 \pm 2.9$ MPa, PWB: $14.9 \pm 2.6$ MPa).

Thermocycling affected the bonding performance of all four cements. However, bond strength of RelyX Unicem was least influenced by thermocycling (UTZ: $9.4 \pm 2.9$ MPa, UTB: $8.6 \pm 1.3$ MPa, ATZ: $11.4 \pm 6.3$ MPa, ATB: $13.3 \pm 3.7$ MPa, MTZ: $15.4 \pm 3.1$ MPa, MTB: $10.3 \pm 2.4$ MPa, PTZ: $11.1 \pm 2.8$ MPa, PTB: $11.3 \pm 2.8$ MPa).

*Significance.* Although the bond strength of RelyX Unicem to dentin was lower in comparison to RelyX ARC, Multilink and Panavia 21, its bond strength was less sensitive to variations in handling and aging.



## 1. Introduction

Dental luting cements form the link between a fixed restoration and the supporting tooth structure. Especially for weak and brittle restorative materials like, e.g. silica-based ceramics the cement plays a key role for the clinical performance [1]. Several in vitro and in vivo studies suggested that all-ceramic restorations should be bonded to tooth structure with resin cements in order to increase their fracture resistance [1–4]. Moreover, compared to other traditional luting materials such as glass ionomer, zinc phosphate, and polycarboxylate cements, resin cements have several additional advantages. They improve retention [5,6] and exhibit reduced dissolution in the oral environment [6]. Furthermore, they lead to less micro-leakage and have excellent esthetic shade-matching potential [6–10]. However, resin cements require skilful han-

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dling: the preparation of the hydrophilic dentin surface for the application of the hydrophobic resin cement is a technique sensitive procedure and is time consuming [11].

A durable bond depends on the chemical composition of the adhesive agent and the surfaces that are connected. In order to establish a successful bonding to tooth substance or materials like ceramics, appropriate pre-treatment methods have to be applied [12].

An organic silica-based ceramics need to be etched with hydrofluoric acid and silanized to achieve a chemical bonding to organic adhesive cements [13–15]. Newly utilized high-strength ceramics like alumina or zirconia, however, have a different chemical composition and structure. As they do not contain silica, conditioning procedures as known for silica-based ceramics are not indicated [16]. In order to establish a chemical bond to these ceramics 10-methacryloyloxydecyl hydrogen phosphate (MDP) is necessary [17].

The surface conditioning of various all-ceramic systems requires a fundamental knowledge of ceramic compositions. The practitioner faces the problem to choose the adequate cement to get an optimal bond both to the tooth substrate and the ceramic reconstruction. The challenging part of the luting procedure is the bonding to dentin. Dentin is hydrophilic while conventional adhesive cements are hydrophobic. Therefore, the bond has to be established stepwise by means of etching and by applying an amphiphilic primer and the corresponding hydrophobic bonding agent [18]. Furthermore, during the pre-treatment of the dentin, the oral environment should be free of moisture in order to obtain a good bond [19].

To simplify these technically sensitive procedures a new type of adhesive cement has been developed (RelyX Unicem; 3M ESPE, Seefeld, Germany). During the setting of the cement its properties change from hydrophilic to hydrophobic. Therefore, it is claimed that this cement should adhesively bond to dentin without any pre-treatment. Furthermore, as it contains MDP, it is expected to bond to a wide range of ceramic or metal-based restorative materials [20].

Several studies indicated that RelyX Unicem exhibited similar bonding performance to various prosthodontic materials like other resin cements [21–23]. Furthermore, good marginal integrity of reconstructions luted with RelyX Unicem was shown in microscopic investigations [24,25].

Studies showed promising results concerning its bond strength to dentin [26–28]. However, differences in test design lead to difficulties in the interpretation and comparability of the results.

The objectives of this in vitro study were (1) to assess the bond strength of the universal cement RelyX Unicem to dentin and to compare it with conventional resin cements, (2) to test the influence of aging on the bonding quality and (3) to test the influence of the operator on the bonding quality of the cements.

## 2. Material and methods

The dental schools of the Universities of Zurich (Z) and Berne (B) participated in this investigation.

The following four adhesive cements and their corresponding bonding agents were included in this study:

- (U) RelyX Unicem (3M ESPE, Seefeld, Germany)
- (A) RelyX ARC (3M ESPE, Seefeld, Germany)
- (M) Multilink (Ivoclar Vivadent, Schaan, Liechtenstein)
- (P) Panavia 21 (Kuraray, Osaka, Japan).

The compositions of these cements as indicated by the manufacturers are listed in Table 1.

A total of 160 non-carious human third molars were divided into two main groups of 80. In each center, Zurich and Berne, the teeth were furthermore divided into 8 subgroups of 10 teeth each.

The preparation and the analysis of the specimens followed the same study-protocol.

An overview of the subgroups is presented in Table 2.

### 2.1. *Preparation of the specimens*

Teeth were stored in 0.1% thymol solution for a maximum of 6 months until bond strength testing. They were cleaned, embedded in epoxy resin (Specific Resin 20, Struers, Switzerland) and hardened under reduced pressure of 100–200 mbar (cold mounting). The embedded teeth were ground flat on the buccal side to obtain a dentin surface of at least 10 $mm^2$ for the bonding procedure. This was achieved by means of a polisher (Reco GMT 5350, Le Leux, Switzerland) with a series of SiC-papers of different grit sizes ending with 320 grit.

### 2.2. *Bonding procedure*

For each cement a specific bonding procedure was applied (Table 3).

#### 2.2.1. *RelyX Unicem*

The dentin surfaces of the specimens were cleaned with water and dried with a short air blast (oil and water-free air) in order not to overdry the dentin surface. RelyX Unicem capsules were inserted in the Aplicap activator (3M ESPE) and activated for 2 s. The capsules were then mixed in a high-frequency rotary mixer (Rotomix, 3M ESPE) for 10 s and the cement applied on the dentin surfaces as described below.

#### 2.2.2. *RelyX ARC*

The dentin surfaces of the specimens were cleaned and etched with Scotchbond etchant for 15 s, rinsed for 10 s with water and softly dried with oil and water-free air leaving the dentin surface visibly moist. Adper Scotchbond 1XT was applied for 5 s and light-cured for 10 s. RelyX ARC was dispensed on a mixing pad and was mixed for 10 s. Mixed RelyX ARC cement was then applied onto dentin surfaces as described below.

#### 2.2.3. *Multilink*

The dentin surfaces of the specimens were cleaned with water and dried with oil and water-free air. Multilink Primers A and B were mixed in a 1:1 ratio and applied on dentin surfaces for 15 s with slight pressure. The applied primer was subsequently dried with air. Multilink cement catalyst and base were dispensed from the double-push syringe and mixed in a 1:1 ratio.