

Shear bond strength of three self-adhesive resin cements to dentin

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ABSTRACT

Context: The result of the studies concerning the bonding of self-adhesive resin cements to dentin is controversial.

Aims: To assess *in vitro* shear bond strength (SBS) of three self-adhesive dual-cured resin cements to dentin compared to a currently used dual-cured resin cement, using SBS test.

Settings and Design: The extant study is an experimental *in vitro* one on extracted human third molars dentin.

Material and Methods: 40 intact human third molars were selected and randomly divided into 4 groups of 10. Buccal dentin surfaces were exposed perpendicular to the long axis and prepared with SiC papers. A translucent plastic ring, was placed over the dentin surfaces. Group I (Control group): After 15 s of etching and application of Excite DSC Bond (Ex), Variolink II (Var II) resin cement was injected into the plastic ring and was light cured for 40 s. Group II: RelyX Unicem (RX) was injected into the plastic ring and after 30 s, was light cured for 40 s. Group III: Maxcem (Mc) was injected into the plastic ring and after 30 s, was light cured for 40 s. Group IV: Multilink Sprint (MS) was injected into the plastic ring and after 30 s, was light cured for 40 s. After thermal cycling, SBS were measured with a universal testing machine.

Statistical Analysis Used: Statistical computations were conducted according to Student's *t*-test.

Results: The mean SBS and standard deviations (in parentheses) for groups I, II, III, and IV were 12.95 (2.64), 6.73 (0.79), 3.01 (0.90), 4.60 (0.75) MPa, respectively. Statistical analysis, revealed that: (1) the mean SBS of Var II were significantly higher than the other groups ($P < 0.05$). (2) The mean SBS of Mc and MS were significantly lower than RX ($P < 0.05$). (3) The mean SBS of Mc and MS did not show significant difference ($P > 0.05$).

Conclusion: Bond strength of three self-adhesive resin cements was significantly lower than the conventional total-etch resin cement. RX significantly performed better SBS than Mc and MS to dentin.

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Resin-based adhesive luting materials are widely used for the cementation of inlays and onlays, crowns, posts and veneers. Currently, all resin cements are based upon the use of an etch-and-rinse or self-etch adhesive along with a low-viscosity resin composite. This multi-step application technique is complex and rather technique sensitive, and

consequently may compromise bonding effectiveness.^[1] Therefore, in order to simplify the cementation process, some brands of self-adhesive resin cements have been released to the market, consisting of monomers which are capable of etching and bonding to dental surface without the need for separate application of an adhesive system. The use of these materials not only simplifies the bonding procedure between the tooth structure and the indirect restoration, but also reduces cement film thickness and clinical time spent.^[2] The objective in developing these cements was to combine ease of handling (no pretreatment steps required) offered by glass ionomer cements with the favorable mechanical properties, esthetics, and desirable tooth adhesion of resin cements.^[2]

Among these relatively new materials are the three self-adhesive dual-cured resin cements, Rely X Unicem (3M, ESPE, St. Paul, USA), Maxcem (Kerr, Orange, USA) and

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Multilink Sprint (Ivoclar Vivadent, Schaan, Liechtenstein), used in the present study.

The results of the studies accomplished on the bonding of Rely X Unicem (RX) to dentin are controversial. Piwowarczyk *et al.*,^[3] found that RX shear bond strength to dentin was significantly lower than that of Variolink II in two conditions: With a 150-day storage in water and storage in water plus 37 000 thermal cycles.

Holderegger *et al.*,^[4] reported that shear bond strength of RX cement to dentin was lower than conventional resin cements such as Multilink, Rely X ARC and Panavia-F, but technique-sensitivity of RX cement was lower due to its one-step application. In a study accomplished by Yang *et al.*,^[5] on microtensile bond strength of RX to dentin, they stated that RX bond strength to all three types of superficial, deep and cervical dentin was significantly lower than Panavia-F. But De-Munck *et al.*,^[6] found that microtensile bond strength of RX to dentin was similar to Panavia-F. Cantoro^[7] and Abo-Hamar^[8] *et al.* in two separate studies, found it to be similar to Panavia-F. According to Piwowarczyk *et al.*,^[3] the difference between RX and Panavia-F bond strength was not significant. Goracci *et al.*^[9] and Sarr *et al.*,^[10] showed the low interfacial strength and high amount of premature failures for Mc and MS bonded to dentin or enamel in comparison with Panavia F and Var II. Viotti *et al.*,^[11] found that the multistep etch-and-rinse system (RelyX ARC) and the two-step self-etch technique (Clearfil SE Bond with Panavia F) produced higher bond strength than the self-adhesive cements such as Mc and RX.

The current study was designed to compare shear bond strength of three self-adhesive resin cements. with the conventional dual-cured resin cement, VariolinkII (Ivoclar Vivadent, Schaan, Liechtenstein) used with the total-etch adhesive system Excite DSC (Ivoclar Vivadent, Schaan, Liechtenstein) in order to provide a conspectus for further investigation on these materials and practical guide for dentists, since the bond strength of resin cements is a fundamental factor in their clinical use.^[12]

MATERIALS AND METHODS

In this experimental *in vitro* study, 40 human third molars of 18–25-year-old patients were selected. They had no crack, caries or restorations, and were extracted not more than a month before the shear bond strength test. After removal of debris, they were stored in 0.01% thymol solution at 4°C temperature. The teeth were randomly divided into four groups of ten. Buccal enamel surfaces of all the teeth were totally removed perpendicular to the long axis of the tooth using water-cooled cylindrical diamond #837-016 (SSwhite, USA). Then the dentin surface was prepared with 240, 400, and 600 grit silicon carbide papers (Matador, Germany), respectively under running tap water to obtain a polished

surface. A translucent plastic ring, with 3.35 mm internal diameter and 2 mm of height, was placed over all the teeth, perpendicular to the polished buccal dentin surface, and fixed externally.

In group 1, after 20 s of rinsing, the surface was gently dried by clean air for 5 s. Then it was etched for 15 s by 37% phosphoric acid and rinsed for 20 s. The remaining moisture was removed using two sudden flashes of air. After mixing the base and activator, a thin layer of Excite DSC (EX) bonding was applied on the dentinal surface. The bonding layer was thinned by air and light cured for 20 s. Then the translucent plastic ring was filled with Variolink II (Var II) composite and light cured for 40 s.

In group 2, after 20 s of rinsing and removal of the remaining moisture by two sudden flashes of air, Rely X Unicem (RX) capsule, after being activated by pressing the side of the capsule, was inserted to Rotomix (3M, ESPE, St. Paul, USA) machine and the machine was activated for 15 s. The capsule was then removed from the Rotomix and cement was injected into the plastic ring by its special gun and after 30 s light cured for 40 s.

In group 3, after 20 s of rinsing and removal of the remaining moisture by two sudden flashes of air, Maxcem (Kerr, Orange, USA) was injected into the plastic ring and after 30 s light cured for 40 s.

In group 4, after 20 s of rinsing and removal of the remaining moisture by two sudden flashes of air, Multilink Sprint (Ivoclar, Vivadent, Schaan, Liechtenstein) was injected into the plastic ring, using mixing syringes and after 30 s light cured for 40 s.

All the curing process was done using Coltolux75 (Coltene/whaledent, Konstanz, Germany) light curing unit at 700 mW/cm². The output power from light-curing unit was monitored during the preparation process by a light meter device (APOZA, China).

All the Specimens were stored in distilled water for 24 h. The specimens were then thermocycled for 1000 cycles from 5 to 55°C. Each thermal cycle lasted 75 s–30 s dwell time for each bath and 15 s delay time for transmission between them. The specimens were then embedded in self-cured acrylic resin in such a way that the composite cylinders on the teeth were oriented perpendicular to the dentinal surface and force applying chisel. SBS was measured for each sample with a universal testing machine (Zwick/Roell, Z020, A.S.T. GmbH, Dresden, Germany). The chisel speed was 0.5 mm/min until failure. The results were reported in MPa. The samples were stored in distilled water at room temperature during the entire process of the study except for the period of force application. In order to evaluate the data distribution, Kolmogorov–Smirnov test was used.

Considering the normal distribution of the data in all four groups, the data derived from the universal testing machine were analyzed with one-way analysis of variance (ANOVA) by SPSS software (Chicago, USA) version 15. Scheffe post-hoc test was used for pair comparison to detect any significant ($P < 0.5$) differences in SBS among four groups. After SBS test, the fractured surfaces were evaluated at $\times 20$ magnification under a light stereomicroscope (Olympus Corp., Tokyo, Japan) to carefully examine the failure mode.

RESULTS

The minimum, maximum, mean and standard deviations of all four groups are available in Table 1. Graph 1 shows the 95% confidence interval of mean SBS in four groups in MPa. According to analysis of variance, there was a statistically significant difference among four groups ($P < 0.001$).

$P < 0.05$ shows that at least one group had a significant difference with the other three groups. In order to find the liable group, Scheffe post-hoc test was used. Scheffe test showed that SBS in the first group (control group) was significantly higher than the second ($P < 0.001$), third ($P < 0.001$), and fourth group ($P < 0.001$). SBS in group 2 was significantly lower than the first group ($P < 0.001$) and higher than the third ($P < 0.001$) and fourth group ($P = 0.029$). SBS in group 3 was significantly lower than the first and second group ($P < 0.001$), but there was no statistically significant difference between group 3 and 4 ($P = 0.151$). SBS in group 4 was significantly lower than first ($P < 0.001$) and second group

($P = 0.029$), but had no significant difference compared to the third group ($P = 0.151$).

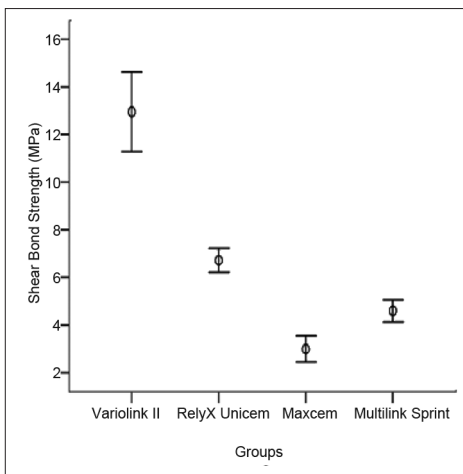
Two by two comparison of SBS between the four groups of the study is demonstrated in Table 2. The outcome of observing the shear surface under the stereomicroscope was the following: Fractured surfaces in all samples of groups 3 and 4 were adhesive-type at the interference of cement and dentin. In group 2, in two samples the fracture was cohesive-type in the resin cement and in the rest of the samples of this group an adhesive-type of fracture was observed. In the first group, all the fractures were cohesive-type inside the composite or bonding layer.

DISCUSSION

Bonding to dentin has been the target of numerous studies, in an attempt to attain adhesive systems capable of efficiently interacting with this delicate substrate. Compared with enamel, bonding to dentin is harder to achieve, because of its morphologic characteristics, high organic content and tubular structure partially filled with the odontoblastic process.^[13,14] The formation of smear layer that occludes the tubules and reduces dentinal permeability on instrumented dentin is another reason that the interaction between the adhesive system and dentin is difficult.^[15]

Shear bond strength is the common laboratory parameter most often used to evaluate bond strength of restorative materials to dental structures.^[16] A major disadvantage of SBS test is that it does not consider the three-dimensional geometry of tooth preparation and consequent variations in polymerization shrinkage vectors. Therefore, data deriving from SBS tests should be evaluated along with clinical assessment results. However, this test is an excellent tool for screening new materials and comparing different adhesive systems.

In the control group of this study, Ex dual cured bonding system along with Var II composite was used, because it has been used as control group in many of bond strength studies and also as a well-known commercial product, has a wide clinical use in dentistry.^[3] This system is among the fifth generation of bonding systems, known as total-etch adhesives. These adhesive systems have many advantages, including ease of working process and the ability to produce



Graph 1: Mean of shear bond strength (MPa)±2 standard deviation in four groups

Table 1: The minimum, maximum, mean and standard deviations of all four groups

Standard deviation	Mean	Maximum	Minimum	Group
2.64	12.95	16.22	9.00	Group 1 (VariolinkII)
0.79	6.73	8.05	5.55	Group 2 (RelyX Unicem)
0.90	3.01	4.54	2.00	Group 3 (Maxcem)
0.75	4.60	5.60	3.38	Group 4 (MultilinkSprint)

Table 2: Two by two comparison of SBS between the four groups

P value	SBS difference (MPa)	Groups
0.00*	6.22	1 to 2
0.00*	9.94	1 to 3
0.00*	8.35	1 to 4
0.00*	3.72	2 to 3
0.029*	2.13	2 to 4
0.151†	0.41	3 to 4

[†NS - Non-significant ($P > 0.05$), *S - Significant ($P < 0.05$)]

filled adhesive as shock absorbers. These characteristics make these systems not only capable of achieving favorable bond strength, but also highly acceptable.^[17] Another reason for choosing this system was that we wanted a material as the control group to be dual-cured, just like the three self-adhesive resin cements used in this study, to minimize the effect of polymerization pattern on its bond strength to dentin. It has been proven that dual-cured and light-cured resin cements exhibit higher bond strength than the self-cured cements.^[2,18,19]

Since RX was the first resin cement introduced to the market,^[3] most of the studies about self-adhesive resin cements, concern this product. We found that shear bond strength of self-adhesive resin cements were significantly lower than Var II combined with a total-etch system. This is in accordance with the study of Piwowarczyk *et al.*,^[3] on RX.

Vrochari *et al.*,^[20] also found that the degree of cure for RX, MS, Mc in dual-curing mode were low (26.40-41.52%) in comparison with Multilink Automix self-etching resin cement system (61.36%). This may be one of the reasons why self-adhesive resin cements exhibit decreased bond strengths.

In another study, Vrochari *et al.*,^[21] found that Mc exhibited very high water sorption, whereas RX and MS exhibited water sorption only slightly higher than Multilink Automix. Regarding water solubility, they found that the greater mass loss was found for Mc, MS exhibited no mass change whereas RX and Multilink Automix exhibited negative solubility. Higher water sorption and solubility in Mc may explain lower bond strength of this cement after water storage and thermocycling in this study.

In our study, SBS of RX to dentin was significantly higher than the two other self-adhesive resin cements, but there was no significant difference between SBS of Mc and MS. Gerth *et al.*,^[22] in their study showed an increased chemical interaction of RX with Calcium from hydroxyapatite, which may explain the higher bond strength of this cement in this study compared to the other self-adhesive resin cements.

The fracture pattern of these three resin cements showed that the main pitfall of them is incomplete adhesion to the instrumented dentin. Remaining the smear layer may account for the decreased bond strength they exhibit, since it has been clearly proven that to achieve a favorable bond to dentin, the smear layer must be removed and collagen fibers exposed to let the adhesive materials enter this network.^[7] De Munch *et al.*,^[6] also showed the remnant of smear layer and insufficient penetration of RX into the dentinal tubules under electronic microscope and stated that no hybrid layer is evident at the interference of dentin to RX. This is also a reason for reduced shear strength and development of an adhesive-type fracture pattern in this cement. Inspecting

the electronic microscope section of RX, Yang *et al.*,^[5] found a similar outcome. They also inferred that insufficient penetration of this cement into the dentinal tubules, lack of ability to remove the smear layer and lack of penetration between the collagen fibers decreased the bond strength of RX to dentin. They also concluded that the reason for this reduced bond strength is the high level of fillers and subsequent high viscosity of this cement in comparison with adhesive materials. It is worthy of mention that Mc and MS cements are now discontinued and their companies have currently released new products of this type, such as Maxcem Elite, Mutilink Speed and Speed CEM.

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