





SCIENTIFIC RESEARCH DATA

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Bond of Resin Cements to Tooth Substrates in Self-cure Mode

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Objectives: Dual cured resin cements are frequently used to cement restorations that block light transmission in these situations the cement is polymerized using self-cure mode. The objective of this study was to measure the bond strength of 4 dual cure resin cements to dentin and enamel in a self-cure mode.

Methods: 160 extracted human molars were ground to mid-coronal dentin (N=80) or superficial enamel (N=80), finished to 320 grit SiC disks and sonicated. Four adhesive/cement combinations were applied to enamel and dentin using a self-etch, self-cure mode: Multilink Primer A & B/Multilink Automix (MA, Ivoclar), Panavia V5 Tooth Primer/Panavia V5 Paste(PanV5, Kuraray Noritake Dental), Optibond XTR/NX3 (NX3, Kerr), and Scotchbond Universal/Rely X Ultimate (RXU, 3M). A 1mmx1mm block of e.max (Ivoclar) was etched with 5% HF (20sec), rinsed, dried, silanated, coated with each cement and seated onto enamel or dentin. 100 g of force was applied to the tooth/e.max assembly for 8 minutes. To more closely replicate the oral cavity, the experiment was repeated but specimens were fabricated by applying pressure to the e.max/tooth assembly for 10 minutes in an incubator (37°C). After 24 hrs storage (at 37°C), samples were debonded at 1mm/min crosshead speed in a universal testing device. A separate 1-way ANOVA and Tukey post-hoc analysis (when appropriate) was performed for each material (alpha=0.05).

Results: n = 10

1. Bond strength of resin cements at room temperature(25°C).

Dentin: PanV5 > MA > NX3 = RXU. Enamel: PanV5 > MA > RXU > NX3.

2. Bond strength of resin cements at elevated temperature(37°C).

Dentin: PanV5 > MA > NX3 > RXU. Enamel: PanV5 > MA > RXU > NX3.

 Table 1
 Shear bond strength of resin cements to enamel and dentin (MPa)

| | MA(MPa) | PanV5(MPa) | NX3(MPa) | RXU(MPa) |
|---------------|----------|------------|----------|---------------|
| Dentin (25°C) | 13.7 ± 7 | 17.1 ± 5 | 0 | 0 |
| Enamel (25°C) | 13.4 ± 3 | 21.4 ± 7 | 6.6 ± 2 | 9.4 ± 4 |
| Dentin (37°C) | 11.7 ± 7 | 20.0 ± 5 | 5.6 ± 4 | 3.1 ± 0.3 |
| Enamel (37°C) | 13.7 ± 3 | 15.2 ± 8 | 9.6 ± 6 | 11.3 ± 6 |

Conclusions: Panavia V5 produced significantly greater shear bond strength compared to Multilink Automix, RelyX Ultimate and NX 3 in the self-cure mode both at room temperature and at elevated temperature (37°C)(p<.05)

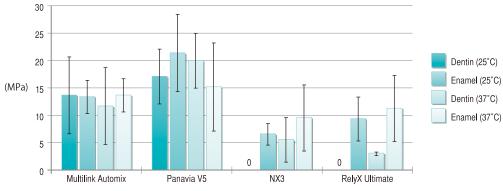


Fig. 1 Shear bond strength of resin cements to enamel and dentin

Bonding Ability to Dentin of Resin-cement: Dual-cure vs. Self-cure

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Objectives: One of the major concerns with resin-cements is their ability to achieve effective initial bonding to tooth substrate (Dent Mater 2010; 26: 608-615). The purpose of this study was to evaluate dual-cure mode *versus* self-cure mode of resin-cements on the shear bond-strength to dentin substrate (SBS). Five cement+adhesive types [Experimental Cement R1096/Prime & Bond elect: Dentsply/Caulk (DC), RelyX Ultimate/Scotchbond Universal: 3M ESPE (3E), Variolink Esthetic DC/AdheSE Universal: Ivoclar Vivadent (IV), ESTECEM/ESTELINK: Tokuyama Dental (TD), HPC-100/HPC-100 Primer: Kuraray Noritake (KN)], and three self-adhesive types [PermaCem 2.0: DMG (DM), G-Cem LinkAce: GC (GC), BeautiCem SA: Shofu (SF)] were used.

Methods: Dentin surfaces for adhesive types alone were pretreated by each primer. Stock stainless steel rods (3.5 mm diameter and 2 mm height) were positioned by a Teflon mold (3.6 mm diameter and 2.0 mm height) on dentin surfaces, and luted with each cement. SBS were measured immediately after setting (Dual-cure: at 3 min after the start of light irradiation, Self-cure: 10 min storage in 37°C and 100 %RH) and after 24-h storage in water (37°C). Statistical analyses were performed by *t*-Test (Dual-cure *vs.* Self-cure).

Results: Summary of SBS: Mean (S.D.), MPa, N=10, S: significant difference (p<0.05), NS: Not significant difference (p>0.05).

| | | | | Adhesive type | type Self-adhesive type | | | ре | |
|------------------|---------------|---------------------|--------------------|--------------------|-------------------------|---------------------|--------------------|---------------------|---------------------|
| | | DC | 3M | IV | TD | KN | DM | GC | SF |
| Immediately | Dual- cure | 5.4 (1.5) S | 15.3 (1.7) S | 9.9 (2.9) S | 17.9 (4.2) S | 12.2 (3.2) NS | 5.5 (1.9) S | 5.4 (1.6) NS | 8.4 (2.3) S |
| | Self- cure | 3.7 (1.1) | 6.0 (1.6) | 3.9 (1.2) | 8.4 (2.6) | 13.5 (3.5) | 1.4 (0.6) | 6.7 (1.8) | 1.2 (0.3) |
| After one-day | Dual- cure | 15.8 (3.1) NS | 19.5 (4.2) S | 16.8 (2.8) S | 19.0 (3.1) NS | 23.3 (3.6) NS | 13.9 (3.1) S | 13.0 (2.3) NS | 12.8 (2.5) NS |
| | Self- cure | 15.7 (3.0) | 14.0 (3.5) | 10.5 (2.1) | 16.4 (3.3) | 24.0 (3.4) | 6.9 (1.2) | 10.6 (2.8) | 11.7 (3.1) |

Conclusions: Light irradiation increased the SBS over self-cure alone immediately after setting, but one-day storage brought the same SBS without depending on light irradiation.

Adhesive Property of a Newly Developed Resin Cement System "HPC-100"

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Objectives: Kuraray Noritake Dental Inc. has developed a new resin cement system "HPC-100" consisted of a dual-curing composite resin cement and a one-bottle MDP-based self-etching primer. The cement is a two paste formula composed of methacrylate monomers, new redox initiators and fillers in automix delivery system. The primer contains a new accelerator for promoting polymerization of the cement at adhesion interface. The purpose of this study was to compare the shear bond strength of HPC-100 with other two commercial resin cement systems; RelyX Ultimate (3M ESPE), and Multilink Automix (Ivoclar Vivadent), to human dentin.

Methods: Shear bond strength to human dentin in self-curing mode was measured after storage in 37°C water for 24 hours and after subsequent thremocycling (4°C-60°C, 1min. each, 3,000cycles: TC3000). The bonding procedure for each material was performed according to the manufacturer's instruction.

Results: The results were summarized in Table 1.

Table 1 The Shear Bond Strength to Human Dentin (MPa (S.D.))

| | HPC-100 | Rely X Ultimate /Scotchbond Universal | Multilink Automix /Multilink primer A+B |
|----------------|------------|--|--|
| 37°C, 24 hours | 24.2 (3.2) | 12.8 (5.0) | 12.4 (3.3) |
| TC3000 | 25.3 (2.1) | 10.9 (3.7) | 12.8 (3.4) |

Conclusions: The new resin cement system "HPC-100" showed the highest bond strengths to human dentin after 24 hours and TC3000 in comparison with the other commercialized two resin cement systems. This study indicates that HPC-100 would exhibit reliable clinical performance superior to the resin cement systems used in this study.

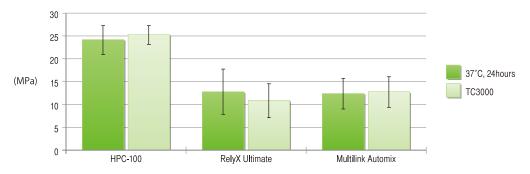


Fig. 1 The Shear Bond Strength to Human Dentin

Color Stability of Amine-free Dental Cement

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Objectives: Chemical and dual cured cements contain an amine-based initiator. Amine-based initiators may oxidize leading to color change in the cement. The purpose of this study was to measure the color change of a novel amine-free cement compared to two clinical successful resin cements.

Methods: 1mm thick discs of 2 amine based resin cements (Multilink Automix; Ivoclar, Rely X Ultimate; 3M) and an amine-free resin cement (Panavia V5; Kuraray Noritake Dental) were fabricated (n=10) in a polyvinyl siloxane mold covered by a mylar sheet and glass slide. The cements were allowed to polymerize for 1hr in the dark. Baseline color values (Lab) were then measured with a spectrophotometer against white and black backgrounds. Specimens were then placed in distilled water (37°C) for 1 day, 1 week, 2 weeks, 3 weeks, and 4 weeks. After each storage time, the color measurements were taken. Change in color from baseline was reported as ΔE based on the formula: $\Delta E = (\Delta^2 + \Delta a^2 + \Delta b^2)^{0.5}$.

Results: Two separate repeated measures ANOVAs found factors material and time and their interaction significant (p<.01) for both white and black backgrounds. Tukey post-hoc test determined that color change of materials ranked: Panavia V5 < Multilink Automix < RelyX Ultimate (for white background) and Panavia V5 and Multilink Automix < RelyX Ultimate (for black background). Separate post-hoc tests for each material grouped statistically similar time points with similar superscripts.

Table 1 Color change of cements

| | Multilink Automix (⊿E) | | Rely X Ultimate (⊿E) | | Panavia V5 (⊿E) | |
|--------|------------------------|---------------|----------------------|-----------------|-----------------|---------------|
| | white | black | white | black | white | black |
| 1 day | 2.25 ± 0.73 a | 1.59 ± 0.64 a | 3.90 ± 2.05 a | 5.00 ± 2.22 b | 1.55 ± 0.75 a | 1.60 ± 0.94 a |
| 1 week | 1.53 ± 0.57 a | 1.92 ± 0.68 a | 3.55 ± 2.32 a | 5.21 ± 2.64 b | 1.37 ± 0.59 a | 2.12 ± 1.30 a |
| 2 week | 1.61 ± 0.43 a | 2.06 ± 0.49 a | 3.11 ± 2.33 a | 4.74 ± 2.64 a,b | 1.73 ± 0.63 a | 1.55 ± 0.75 a |
| 3 week | 4.37 ± 0.72 ° | 1.20 ± 0.41 a | 4.45 ± 0.72 a | 2.74 ± 0.86 a | 2.10 ± 0.96 a | 1.12 ± 0.53 a |
| 4 week | 3.22 ± 1.19 b | 4.01 ± 1.65 b | 3.76 ± 1.55 a | 3.49 ± 2.23 a,b | 2.09 ± 1.14 a | 1.57 ± 0.65 a |

Time points in the same column with similar superscripts have statistically similar delta E.

Conclusions: The amine-free resin cement showed less color variation with time than two amine-based cements. Project supported by Kuraray Noritake Dental Inc.

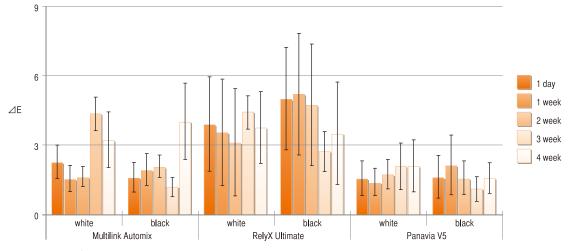


Fig. 1 Color change of cements

Microtensile Bond Strength Between Four Resin Cements and Dentin

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Objectives: The purpose of this study was to evaluate the microtensile bond strength of a newly developed resin cement (HPC-100, Kuraray Noritake Dental) and three controls (RelyX Ultimate®, 3M ESPE; Panavia F2.0®, Kuraray Noritake Dental; Multilink® Automix, Ivoclar Vivadent) when used with human dentin.

Methods: Extracted non-carious human molars were sectioned at the dentin-enamel junction to expose the dentin, and were ground with #600 silicon carbide paper. Two types of bonded specimen were prepared. One was the specimen that the flat dentin surface was covered with resin cement to form resin cement crown (Filling). In the other specimen, a piece of masking tape was attached to the dentin surface, and a prefabricated resin block was luted with resin cement (Luting). After 30 min, all of the bonded specimens were immersed in water at 37°C for 48 h. They were then sectioned to obtain sticks with dimensions of 0.9 × 0.9 mm, and subjected to a microtensile bond strength test at a crosshead speed of 0.5 mm/min. The mean bond strength and standard deviation of 15 specimens were calculated. The data were analyzed by ANOVA and a Tukey compromise test at a statistical significance of 0.05.

Results: HPC-100 and RelyX Ultimate exhibited higher bond strengths than Panavia F2.0 and Multilink Automix (Table). No significant difference was found between HPC-100 and RelyX Ultimate and between Panavia F2.0 and Multilink Automix.

Table 1 Mean and standard deviation of microtensile bond strength (MPa).

| | HPC-100 | Rely X Ultimate | Panavia F 2.0 | Multilink Automix |
|---------|----------------|-----------------|----------------|-------------------|
| Filling | 63.4 (22.1) A | 53.9 (14.0) A | 12.8 (5.2) B | 19.0 (9.5) B |
| Luting | 29.7 (13.2) ab | 33.3 (8.8) a | 22.9 (11.6) bc | 15.5 (8.1) c |

Groups with identical letters on each horizontal line indicate no significant difference (p>0.05).

Conclusions: The present findings suggest that HPC-100 and RelyX Ultimate are effective at bonding to dentin.

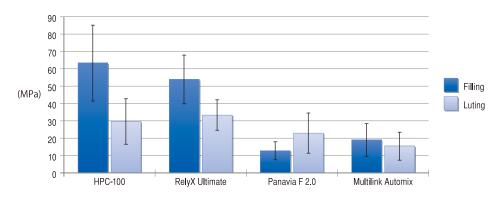


Fig. 1 Microtensile bond strength

Effects of Ultrasonic Cleaning and Etching on CAD/CAM Resin Bonding

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Objectives: Available information is limited about the bonding effectiveness between highly polymerized CAD/CAM resin blocks and adhesive resin cement. It is important, therefore, to establish the appropriate treatment method of CAD/CAM resin blocks. The purpose of this study was to assess the effects of ultrasonic cleaning and acid etching on CAD/CAM resin blocks with a micro tensile bond strength (μ TBS) test.

Methods: Twenty four experimental CAD/CAM resin blocks (KCB-100, Kuraray Noritake) were divided into 2 resin cement groups: HPC group (HPC-100, Kuraray Noritake) and CSA group (Clearfil SA Cement, Kuraray Noritake). Each group was divided into 4 subgroups depending on ultrasonic cleaning and/or acid etching methods: (1) neither cleaning nor etching (Co) subgroup, (2) ultrasonic cleaning (Uc) subgroup, (3) acid etching (Et) subgroup, and (4) Uc+Et subgroup. All specimens were first sandblasted using Al₂O₃ (Adabrader, Morita) and followed by ultrasonic cleaning and/or etching. Silanization was then performed with Clearfil ceramic primer (Kuraray Noritake). Resin cements were built up and stored in water for 24h. The samples were cut into beams (0.7mm×0.7mm, n=24) for each group and μTBSs were measured. Tukey and Student t-test were used for statistical analysis.

Results: μ TBS and failure analysis data are shown in a table. No pre-testing failure was observed. Regardless of subgroups, μ TBSs in the HPC group were significantly higher than those in the CSA group (P<0.001). Acid etching and/or ultrasonic cleaning on sandblasted CAD/CAM resin had no effect on increasing initial bond strengths in both adhesive resin cements.

| | Со | Uc | Et | Uc + Et |
|-----|--------------------------|------------------------|------------------------|-------------------------|
| HPC | 96.54 ± 10.30 (15/24) | 87.57 ± 8.93 (13/24) | 81.12 ± 7.56 (6/24) | 82.87 ± 11.72 (7/14) |
| CSA | 62.77 ± 10.43 (0/24) | 62.55 ± 9.90 (1/24) | 62.58 ± 9.86 (1/24) | 63.85 ± 10.92 (0/24) |

Table 1 μTBS (MPa, mean±standard deviation) and failure analysis data (interfacial failure/ number of specimens)

Conclusions: This study suggests that silanization following sandblasting is a sufficient bonding method to increase the initial bonding strength of CAD/CAM resin.

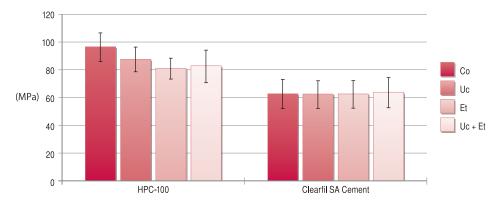


Fig. 1 Microtensile bond strength

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