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# **Evaluating the Effectiveness of 8th Generation Universal Bonding Agents, Universal Primers, and Traditional Silane on Shear Bond Strength to Lithium Disilicate**

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## **ABSTRACT**

**Purpose:** The purpose of this study was to compare the shear bond strength amongst a new universal 8<sup>th</sup> generation bonding agent, universal primers, and traditional silane on lithium disilicate.

**Materials and Methods:** Ninety-six lithium disilicate (IPS e.max CAD) ingots were cut into 15mm x 15mm x 2.9 mm (+/- .8 mm) thick blocks and invested in Type 3 Stone (buff stone). The lithium disilicate blocks were crystallized according to manufacturer's instructions in an Ivoclar Vivadent Programat P510 dental furnace. Next, the specimens were sanded with 400/600/800 grit emery paper under running water for one minute with light pressure. The surfaces were rinsed with distilled water and were prepared with 5% hydrofluoric acid (Vita Ceramic Etch) for 30 seconds. These specimens were then divided into 4 groups with 24 specimens in each group (n=24 per DBA): Group A – Scotchbond Universal with Ultradent Silane and RelyX Ultimate cement ; Group B – Scotchbond Universal Plus and Relyx Ultimate cement ; Group C – Monobond Plus and Multilink Hybrid Abutment cement ; Group D – Clearfil Ceramic Primer Plus and Panavia V5 resin cement . Each primer or bonding agent was applied to the prepared surface according to the manufacturer's instructions and the cement transferred onto the surface via gelatin pill capsules. Each surface received two cement capsules: totaling 24 samples per bond group on twelve vested lithium disilicate specimens. The specimens were then mounted onto the Alliance RT/5 universal testing machine for notch-edge shear bond strength testing and the values were recorded on the MTS TestSuite TW elite software. After recording, the values were compared using a one-way ANOVA.

**Results:** The mean shear bond strength (MPa) of all groups was 7.89 MPa. The means and standard deviation of each group were: Group A:  $\mu$  - 8.85  $\sigma$  - 4.76, Group B:  $\mu$  - 7.64  $\sigma$  - 2.90,

Group C:  $\mu - 7.52$   $\sigma - 2.04$ , Group D:  $\mu - 7.48$   $\sigma - 1.80$ . The testing revealed no significant difference in shear bond strength between the four groups. Group A showed a larger standard deviation than the other three groups.

**Conclusion:** This in vitro study found: 1) No statistical difference between the groups could be found. 2) New universal bonding adhesive performed as well as traditional primers to lithium disilicate. The mode of failure was mainly due to failure of the adhesive from the lithium disilicate.

**Key Words:** 8<sup>th</sup> generation bonding agent, silane, lithium disilicate, shear bond strength, primer

## **ARTICLE – RESEARCH**

### **Introduction**

Advances in materials and technology in dentistry has brought about the utilization of different adhesives to bond to glass ceramic restorations. Glass-ceramic materials have been used for crowns, fixed dental prostheses, and veneers to replace lost tooth structure. With the desire and development for conservative preparations and replacement of missing tooth structure, the technology of resin-ceramic bonding has come to the forefront of these advances. Bonding is the attachment of one substrate to another. The dental bonding agent's role is to be applied to the surface of these substrates and create a uniform seal.<sup>1</sup> These advancements have revolved around increased bond strength and ease of use, particularly the development of eighth generation one-step universal dental adhesive systems.<sup>2</sup>

The eighth-generation bonding agents feature the addition of a bifunctional molecule, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) that allows for a bond to zirconia-based restorations as well as all metal substrates. With this addition there is still the presence of the previous generational molecules such as silane for silica-based glass ceramics and resins, making these new adhesives truly 'all-in-one' for any dental substrate. Two eighth-generation adhesives in the market are Scotchbond Universal (3M ESPE, St. Paul, MN) and the newly formulated Scotchbond Universal Plus (3M ESPE, St. Paul, MN). The new formulation claims to revolve around three main changes from the original adhesive: 1) optimized mixture of silanes to equal the bond strength of the gold standard separate traditional silane primers; 2) dual-cure accelerator including the ability to prime without curing when used with RelyX Ultimate resin cement; and 3) addition of a bisphenol A (BPA) derivative-free, crosslinking radiopaque monomer for

radiographic identification.<sup>5</sup> The claims from 3M ESPE are promising, but primary investigation into these claims are needed from non-propriety researchers.

This study investigated the claim that when using these products together a clinician no longer needs to light-cure the dental bonding agent before application of the cement, which if true would further streamline the application process. This study also attempted to verify the claims that this new formulation can achieve similar bond as to when a separate pure silane is used prior to bonding. Other silane primers and the traditional use of pure silane require an extra step and extend application time which can allow for contamination or other complications.<sup>6</sup> Traditionally, the intaglio surface of lithium disilicate is treated with hydrofluoric acid (HF) and then silanated or primed. Previously silane utilized  $\gamma$ MPTS and were used as the gold standard prior to inclusion in the adhesive.<sup>7</sup> In the case of Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN), the adhesive was cured before placement of cement. The whole process included preparation of the tooth, selective etching the tooth (optional), and application of 3M Scotchbond Universal to the intaglio of the crown with a light cure, followed by 3M RelyX Ultimate resin cement application before seating. The new process would avoid the need for a separate cure of the intaglio surface, streamlining the process and preventing risk of contamination.<sup>5</sup> Comparison of the shear bond strength of this new adhesive formula to other established universal bonding adhesives and its predecessor with a traditional gold-standard pure silane treatment is needed to verify claims by the manufacturer. In previous studies, treatment with a pure silane prior to use of an adhesive agent has shown to achieve the same, if not higher, shear bond strength values.<sup>8</sup> In addition, verification of this adjusted streamlined instruction for use protocol needs verification that no strength is lost. If claims are substantiated, streamlining supply and procedural steps for providers could be crucial when determining which bonding system to use.

The **objectives** of this study were: 1) Evaluate and compare the shear bond strength of resin cements to lithium disilicate comparing new formulation of eight generation adhesives to verify manufacturer claims on these new adhesive systems; 2) Evaluate failure mode of adhesive interface under SEM microscope. The **null hypotheses were** that 1) There will be no significant difference between the shear bond strengths of the different adhesive systems on lithium disilicate substrate, and 2) The mode of failure for these adhesive systems will be at the adhesive to lithium disilicate interface and not within the material.

## **Materials and Methods**

Forty-eight lithium disilicate IPS e.max CAD ingots (Ivoclar Vivadent, Buffalo, NY) were cut to form 15mm x 15mm x 2.9 mm ( $\pm$ .8mm) blocks using a low-speed precision cutter (Isomet Saw, Buehler, Lake Bluff, IL). The IPS e.max CAD blocks (Ivoclar Vivadent, Buffalo, NY) were crystallized in groups according to manufacturer's instructions in a ceramic furnace (Programat P310, Vivadent, Buffalo, NY) and invested in Type 3 Stone (Microstone, Whip Mix, Louisville, KY) cylinders. These samples were then sanded with 400/600/800 grit SiC abrasive paper (3M ESPE, St. Paul, MN) under running water for one minute with light pressure for each grit. The surfaces were rinsed with distilled water and were etched with 5% Hydrofluoric Acid (Vita Ceramic Etch, Dentsply Sirona, Charlotte, NC) for 30 seconds. These specimens were then divided into 4 groups with 12 cylindrical samples in each group and two substrates bonded to each sample (n=24 per DBA). The four test groups were formed based on four dental bonding adhesives paired with their associated resin cement and treatment modality. Group A – Scotchbond Universal (3M ESPE, St. Paul, MN) with Ultradent Silane (Ultradent, South Jordan, UT) and RelyX Ultimate cement (3M ESPE, St. Paul, MN); Group B – Scotchbond Universal Plus and Relyx Ultimate cement (3M ESPE, St. Paul, MN); Group C – Monobond Plus and

Multilink Hybrid Abutment cement (Ivoclar Vivadent, Buffalo, NY); Group D – Clearfil Ceramic Primer Plus and Panavia V5 resin cement (Kuraray, Okayama, Japan). (Table 4) Each bonding agent was applied to the prepared surface according to the manufacturer’s instructions and the cement was injected into gelatin capsules (Torpac, Fairfield, NJ) and transferred onto the surface. Once placed, the samples were light cured with an Ivoclar Vivadent Bluephase Style rechargeable curing light (Ivoclar Vivadent, Buffalo, NY) according to the manufacturer’s instructions for use either 20 or 10 seconds. The samples were then maintained for 24 hours. The samples were mounted on an alignment jig (Ultradent, South Jordan, UT) and loaded into a universal testing machine (Alliance RT/5 MTS, MTS Corp, Eden Prairie, MN) for notch-edge shear bond strength testing and the values were recorded on the MTS TestSuite system (MTS Systems Corp, Eden Prairie, MN). The force was applied by a knife-edge wedge with 1 mm/min crosshead speed and stopped at point of failure. The maximum load of failure was found in Newtons (N). The shear bond strength (MPa) was then calculated by dividing the maximum value of load failure (N) by the bonding area (mm<sup>2</sup>). These values were recorded and compared using One-way Analysis of Variance (ANOVA).

After testing, each specimen was viewed by a single researcher under a microscope to determine the mode of failure, whether cohesive, adhesive, or both. This was determined by an abundant threshold—with greater than 70% of cement remaining on lithium disilicate sample indicating a cohesive failure, less than 30% of cement remaining on the lithium disilicate sample indicating an adhesive failure and samples determined to be between these two thresholds indicating an undescriptive or dual-mode failure. These non-parametric results determined modality of failure and assisted in discussion of the weak point in the system.

### **Specimen Preparation**

*Group A:*

Applied Scotchbond Universal (3M ESPE, St. Paul, MN) for 20 seconds, dried for 5 seconds, and light cured with a curing light (Bluephase Style Rechargeable, Ivoclar Vivadent, Buffalo, NY) for 10 seconds. Filled gelatin capsules (Torpac, Fairfield, NJ) with RelyX Ultimate Cement (3M ESPE, St. Paul, MN) and transferred onto the surface, removing excess prior to light curing for 20 seconds.

*Group B:*

Applied Scotchbond Universal Plus (3M ESPE, St. Paul, MN) for 20 seconds and dried for 5 seconds. Filled gelatin capsules (Torpac, Fairfield, NJ) with RelyX Ultimate Cement (3M ESPE, St. Paul, MN) and transferred onto the surface, removing excess prior to light curing for 20 seconds.

*Group C:*

Applied Monobond Plus (Ivoclar Vivadent, Buffalo, NY) for 60 seconds and dried for 5 seconds. Filled gelatin capsules (Torpac, Fairfield, NJ) with Multilink Automix cement (Ivoclar Vivadent, Buffalo, NY) and transferred onto the surface, removing excess prior to light curing for 10 seconds.

*Group D:*

Applied Clearfil Ceramic Primer Plus (Kuraray, Okayama, Japan) and dried for 5 seconds. Filled gelatin capsules (Torpac, Fairfield, NJ) with Panavia V5 (Kuraray, Okayama, Japan) and transferred onto the surface, removing excess prior to light curing for 10 seconds.

**Results**

A total of 96 samples (24 per group of dental adhesive systems) were included in this study. Raw data of shear bond strength is presented in Table 1. There were no differences in shear bond strength for any of the groups. The mean shear bond strength of all groups was 7.89 MPa. The means and standard deviation of each group were: Group A:  $\mu - 8.85$   $\sigma - 4.76$ , Group B:  $\mu - 7.64$   $\sigma - 2.90$ , Group C:  $\mu - 7.52$   $\sigma - 2.04$ , Group D:  $\mu - 7.48$   $\sigma - 1.80$ . (Table 2) One button from Group D debonded prematurely in the testing machine, so no shear bond strength was reported for that specimen. The statistical difference between the groups was further confirmed by the p-value being .3987. The non-parametric results of failure modality were determined to be overwhelming with the adhesive failure between the adhesive agent and the lithium disilicate found in 90/96 samples (Group A – 23/24, Group B – 22/24, Group C – 21/24, Group D – 24/24). None of the groups had significantly more cohesive failures than the other groups. (Table 5)

## **Discussion**

In this experiment the null hypothesis was accepted. The experiment set up the evaluation of the interface between the tested adhesive systems and lithium disilicate with each systems correlating cement. The selected systems are the latest developments for each respective company for glass-ceramic bonding and priming. Specifically, from 3M ESPE, research on Scotchbond Universal Plus made claims of similar shear bond strengths to these leading brands with additional benefits. The benefits included a streamlined application procedure, radiopacity for seamless radiographic interpretation, and the ability to bond to ceramic in addition to tooth structure; these claims support a true ‘all-in-one’ eighth generation bonding agent. No other study outside of the 3M-supported models has shown this type of comparison looking directly at this interface.

Of note in the results is the differences present between 3M's traditional formula and that of the new Scotchbond Universal Plus. From the collected data, even though not significant, there appears to be a more predictable bond when evaluating the standard deviation of the data achieved for these subgroups. The standard deviation between the two groups was almost double as Group A's standard deviation was 4.76 MPA while Group B's was only 2.89 MPA (Table 3) This could correlate clinically to the ability of the additional mixture of silane ingredients that are in the new formula contributing to a more predictable bond than its predecessor. This marked improvement could be a key indicator for providers that utilize the traditional formula to make an upgrade to the new product.

The results also showed that the interface between lithium disilicate and resin cement may be the weakest when compared to that of the resin cement to tooth structure as none of the shear bond strengths exceeded the acceptable value of 17 MPa; mean shear bond strength across all groups was 7.89 MPa. Proprietary data from each company shows that their products achieve and exceed this acceptable value when testing this resin cement to tooth interface. Microscopic investigation of the specimens after testing showed the overwhelming failure modality was adhesive failure. An additional finding during the microscopic investigations showed bubbles present in some of the samples. When classified by system, Group 3 – Monobond/Multilink had 4/24 samples with bubbles present, while the other three groups combined presented with that amount. This brings into question the wettability of the Multilink cement and if increased viscosity would improve the adhesive result for this product.

This research and others like it continue to benefit the overall library of products and their application. Continued investigation in the dental materials and their company claims furthers the understanding of these products and their usage. As new products are developed

comparison should be made to ensure that improvements or adjustments in formula do not hinder the achieved attributes from the predecessors. More research on Scotchbond Universal Plus could further verify other claims made by 3M ESPE. Ideally this data set could have been larger to afford a more concise distribution giving a higher confidence level of the results. In addition, another limitation to this research study was the uniformity of the cement-to-sample interface. Further investigation could be conducted with a different substrate like zirconia to test the abilities of 10-MDP in these products as a bifunctional molecule for the zirconia-to-resin cement interface.

Another result to note is that when utilizing the original 3M bonding agent formula there did seem to be higher variance in the data even though not significant. With more samples, it would be interesting to see if this trend continues to a statistically significant result. On a clinical level this would suggest that the original formula of Scotchbond Universal (3M ESPE, St. Paul, MN) may have a more unpredictable bond than the other three adhesive systems. Clinically, this could result in unreliable bonding and possible debonding more often with this adhesive.

Finally, the results suggest that the failure mode between all of these bonding systems is at the interface between the lithium disilicate and cement suggesting that the cement is strong enough to withstand the shear force. This could be a factor clinically because when a debond does occur and the cement remains on the tooth this would require re-preparation or pumicing to ensure all cement has been removed. Another finding when viewing the adhesive surfaces under a microscope was the presence of bubbles in the MB group. This finding could suggest that this cement is slightly less viscous and could result in air bubbles being present when applied on the intaglio surface of a crown. This could affect the bonding surface area and may suggest the possibility of a weaker bond when using this system.

## Conclusion

The resulting data confirmed that these four groups had similar shear bonding strength when used on lithium disilicate. It also showed that an addition of pure silane does not improve the bond to lithium disilicate, even when utilizing the older formula from 3M compared to using the new formula independently. These results suggest that clinicians utilizing any of these bonding agents can expect similar success when bonding to lithium disilicate. More studies are needed with larger samples sizes to statistically confirm these results.

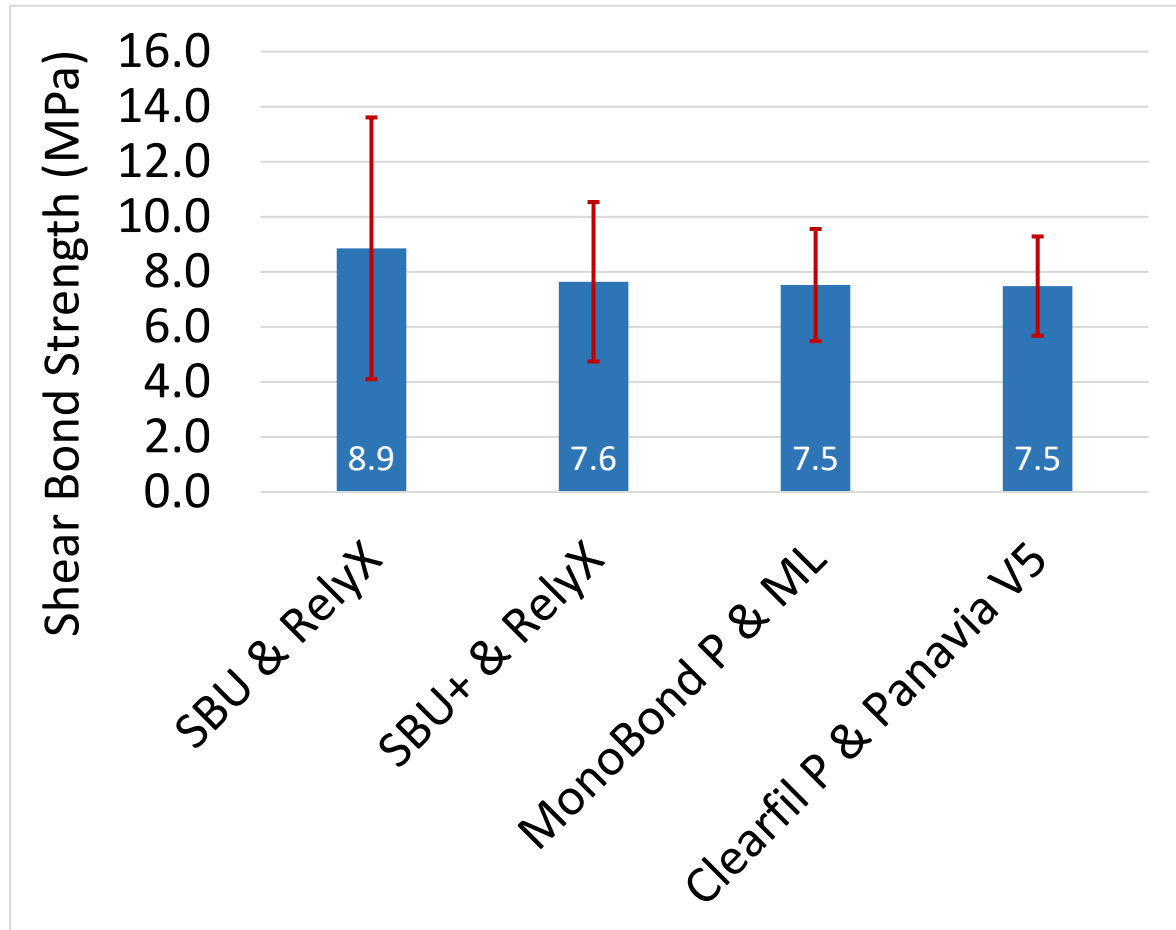
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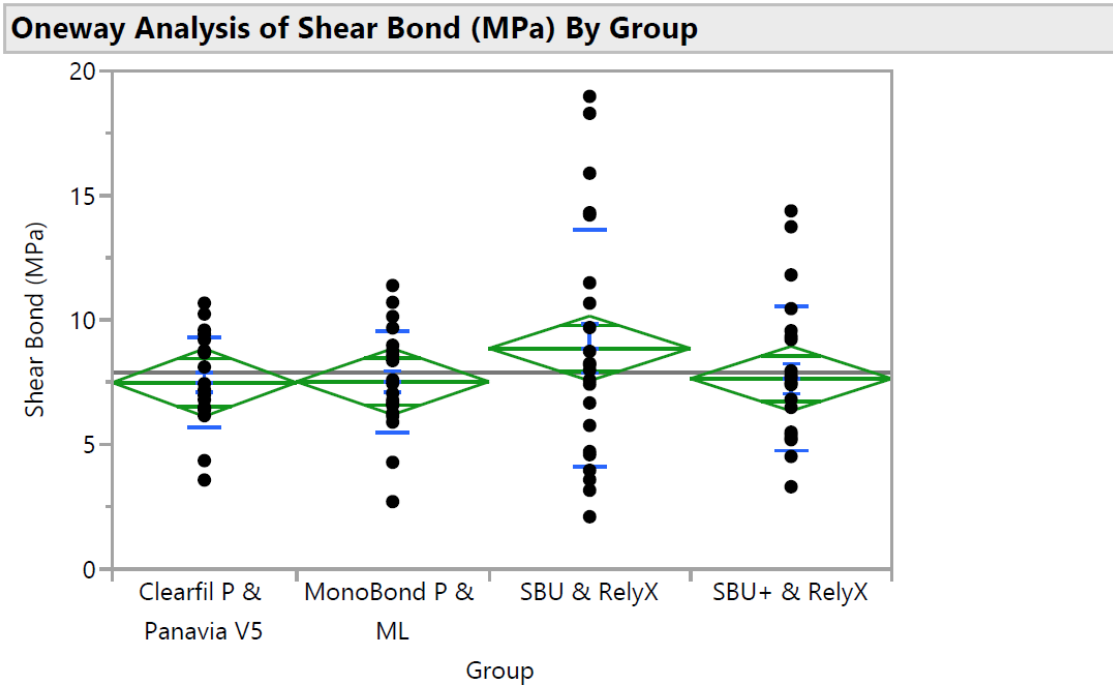
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## Appendix

**Table 1:** Comparison of shear bond strength (MPa) between lithium disilicate and various bonding agents with their respective cements. No group is significantly different.



**Table 2:** One-way ANOVA test for comparison of shear bond strength (MPa) between lithium disilicate and various bonding agents with their respective cements including standard deviation and mean with distribution values



**Table 3:** Specific values for shear bond strength for each group of adhesive system and their distribution values including standard deviation.

Means and Std Deviations						
Level	Number	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
				Mean		
Clearfil P & Panavia V5	21	7.4816656	1.8030154	0.3934502	6.6609428	8.3023883
MonoBond P & ML	22	7.5192295	2.0369882	0.4342873	6.6160796	8.4223795
SBU & RelyX	23	8.8540684	4.7564543	0.9917893	6.7972233	10.910913
SBU+ & RelyX	23	7.6396707	2.8977289	0.6042182	6.3865987	8.8927426

**Table 4:** Adhesive systems and their corresponding cements, composition, indications and curing requirements.

Brand	Composition	Indications	Light Cure	Correlating Cement
Scotchbond Universal	MDP Phosphate Monomer, Dimethacrylate resins (containing BisGMA), HEMA, Vitrebond Copolymer, filler, ethanol, water, initiator, silane.	Glass ceramics, zirconia, alumina, metals, composites	10 seconds	RelyX Ultimate resin cement-light or self-cure (20 seconds if light cured)
Scotchbond Universal Plus	MDP Phosphate Monomer, BPA-Free Dimethacrylate resins, HEMA, Vitrebond Copolymer, Radio-opaque filler, ethanol, water, dual-cure accelerator, optimized mixtures of silanes.	Glass ceramics, zirconia, alumina, metals, composites	None	RelyX Ultimate resin cement-light or self-cure (20 seconds if light cured)
Monobond Plus	Silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate	Glass and oxide ceramics, metal, composites, fiber-reinforced composite	Apply to surface, air dry 5 seconds	Multilink Hybrid Abutment cement optional light cure for 10 seconds
Clearfil Ceramic Primer Plus	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate	Ceramic, hybrid ceramics, composite resin and metal	Apply to surface, air dry 5 seconds	Panavia V5-light cured for 10 seconds

**Table 5:** Mode of Failure for all four adhesive systems.

	Adhesive	Cohesive	Both
Clearfil P & Panvia V5	24	0	0
Monobond P & Multilink	21	1	2
SBU & RelyX	23	1	0
SBU+ & RelyX	22	2	0