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SHEAR BOND STRENGTH OF LOW-SHRINKAGE COMPOSITE TO DIFFERENT DENTAL SUBSTRATES

Hassan El-Shamy* and Hala Ragab**

ABSTRACT

Purpose: This study determined shear bond strength (SBS) of two composite restoratives bonded to enamel and dentin immediately (after one day) and after one month of water-storage. **Methods:** Eighty intact molars were sterilized with Gamma irradiation and mounted in acrylic resin bases. These were divided into two equal groups. In one group buccal enamel was ground flat while in the other buccal enamel was removed to expose flat dentin surface. Cylindrical specimens 2mm thick and 3mm diameter prepared from two composites: Filtek-Z250 (Z250) (3M/ESPE, USA) and Filtek-LS (LS) (3M/ESPE, USA) were bonded to enamel and dentin surfaces. Single Bond (3M/ESPE, USA) adhesive was used with Z250 while LS adhesive (3M/ESPE, USA) was used with LS. Each group was subdivided into 2 subgroups. One subgroup was tested after one-day storage in distilled water at 37°C while other was tested after one-month. An Instron testing machine was used and force at failure was recorded. Mean and SDs were calculated and data statistically-analyzed using ANOVA. **Results:** For enamel, mean and SD for SBS in MPa measured immediately were: 18.4(1.9) and 9.4(1.3) for Z250 and LS, respectively. After one month, means and SD values were: 18.3(1.6) and 9.2(1.1) for Z250 and LS, respectively. When dentin was used, mean SBS values in MPa measured immediately were: 15.8(1.2) and 13.0(1.8) for Z250 and LS, respectively. After one month, mean and SD values were: 15.1(1.5) and 12.9(1.8) for Z250 and LS, respectively. ANOVA revealed significant difference among groups ($p < .05$) regarding type of composite, however, for substrate and storage time there was no significant difference in mean SBS ($p > .05$). **Conclusions:** Filtek-Z250 resulted in mean SBS values that were significantly-higher than those obtained with Filtek-LS at all test conditions. Tooth substrate type as well as storage time up to one month didn't significantly affect SBS.

KEYWORDS: Composite resin, Silorane, Shear bond strength, Adhesion, Self-etching adhesive

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INTRODUCTION

The improvements in dental adhesives and resin composites, along with a minimally invasive approach to caries treatment, have made these restorative materials widely accepted for direct posterior restorations. Dental composites consist of filler particles embedded in a resin matrix. Conversion of the matrix monomers to a polymer network upon light exposure causes the molecules to be packed more closely, leading to a volumetric shrinkage that varies from 5 to 15vol% for cured, unfilled resins and from less than 1% up to 6% for particle-filled dental composites. Such shrinkage is clinically unfavourable as it performs stress to the vulnerable interface. The magnitude of the resultant stress relies upon material properties like volumetric shrinkage, elastic modulus and degree of conversion.¹⁻³

Polymerization stresses created by polymerization shrinkage may compromise the bond integrity, leading to concerns such as microleakage, postoperative sensitivity, and ultimately secondary caries. If the composite-tooth bond remains intact, stresses transmitted to tooth structure may result in cuspal flexure, enamel fracture, or fractured cusps. Optimizing particle sizes, maximizing filler content, and minimizing the concentration of 'diluent' monomers in the resin formulation are steps taken by the manufacturers to decrease the degree of polymerization shrinkage. In addition, material development by incorporation of ring-opening monomers has resulted in formulation of a recent class of silorane composites with significantly lower volumetric shrinkage (less than 1%). However, conventional composites are still widely used in practice, and polymerization shrinkage remains a clinical concern.⁴

FiltekSilorane, introduced in 2007, is the first commercially available resin composite not based on bisphenolAdiglycidyl methacrylate or urethane dimethacrylate, the dimethacrylate monomers most

commonly used. This novel silorane-based resin takes its name from the combination of its chemical blocks, siloxanes and oxiranes. The silorane molecule has a siloxane core with four attached oxirane rings that open upon polymerization to bond to other monomers. This mechanism implies a slight reduction of the initial distance between monomers, which results in a volumetric shrinkage of less than 1%, which might generate less stress on the adhesive interface. The specific chemistry and curing mechanism of the silorane-based resin composite required the development of a dedicated adhesive by the manufacturer. In the case of FiltekSilorane, a two-step self-etch adhesive was developed.^{2,3,5}

Previous studies have indicated that the new silorane composite system greatly reduces shrinkage and decreases cuspal deflection. It is unclear whether the silorane composite with its lower shrinkage on dentin offers higher bond strength, and, to date, there have been few published studies comparing volumetric shrinkage, stress, and push-out bond strength of this adhesive system to conventional dimethacrylate-based self-etching systems.⁶⁻²⁰ Therefore, the purpose of this study was to determine shear bond strength (SBS) of Low shrinkage composite in comparison to methacrylate-based composite restoratives bonded to enamel and dentin immediately (after one day) and after one month of water-storage.

METHODS

A total of 80 intact human molar teeth, recently extracted for periodontal problems were selected for this study. The teeth were cleaned from blood, debris, soft tissue and calculus using ultrasonic scaler and examined under magnifying lenses (X2.5) to detect any cracks or defects in order to be discarded. The teeth were stored in distilled water (changed periodically every one week) no more than a month after extraction.

The selected teeth were mounted horizontally in acrylic resin blocks using splitted aluminum mounting rings. The mounted teeth were stored in distilled water until samples preparation. The 80 mounted teeth were divided into two main groups (of 40 teeth each) according to the type of restorative material used, namely M1 for group of teeth received conventional hybrid composite (Filtek-Z250) (3M/ESPE, USA) and M2 for group of teeth received low-shrinkage composite (Filtek-LS) (3M/ESPE, USA). Each of the previously mentioned groups was subdivided into two sub-groups (of 20 teeth each) according to tooth substrate, S1 for enamel and S2 for dentin. Each of the two subgroups was further divided into four divisions (10 each), according to the time elapsed between composite application and shear bond strength testing namely T1 (One day after composite application) and T2 (One month after composite application). Enamel samples were prepared by flattening the buccal surfaces uniformly across the tooth surface using a grinding wheel on a laboratory trimmer under water coolant to obtain a flat enamel surface. For dentin samples preparation, complete removal of enamel was done using a high-speed dental hand piece together with a diamond wheel round bur of 4mm in diameter (909 wheel round bur, Midwest Diamonds, Dentsply-professional, USA) to make a horizontal guide groove at the maximum convexity of the external buccal surface to a 2mm depth which

was verified using a periodontal probe.²² After that, the enamel was removed uniformly across the tooth surface using a grinding wheel on a laboratory trimmer under water coolant until exposure of the superficial layer of dentin. The exposed dentin surfaces were inspected with magnifying lenses (X2.5) to ensure that no enamel was remained. The dentin surfaces were hand-polished in a clockwise circular motion using wet-600 grit silicone carbide paper for 30 seconds to produce a standard smear layer. The bonding surface was rinsed with water and gently dried with an air stream. After that, a 1.8mm thick circular splitted Teflon ring, with a central hole of 3 mm internal diameter, was firmly fitted on the pretreated samples and secured in place with the aid of a specially constructed copper ring of 38mm internal diameter and 5mm in height (figure 1). Then, each hole was filled with the appropriate composite material according to manufacturer's instructions as a special adhesive system (Silorane System Adhesive Self-Etch Primer and Bond (SSA), 3M ESPE) was used for (Filtek-LS) while an etch-and-rinse adhesive system (Single Bond, 3M ESPE) was employed for (Filtek-Z250). Both adhesive systems were applied and polymerized according to the manufacturer's instructions. Both Composite materials were cured using visible light curing unit for 40 seconds from the top of the Teflon ring over a celluloid strip at zero distance. The intensity of the light source was not less than 600 mW/cm² and the

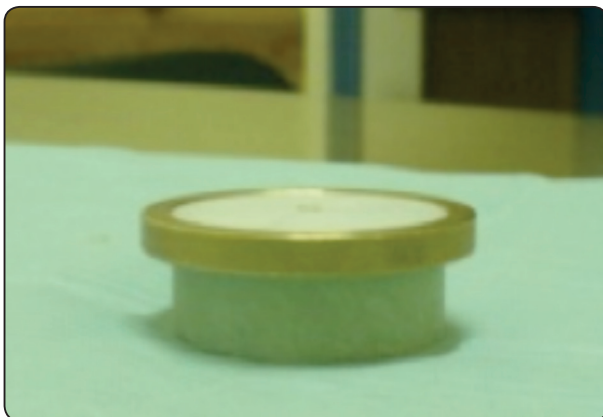


Fig. (1) Specially designed splitted Teflon ring used to create composite cylinders to tooth samples

visible light curing unit was checked for light output each five curing sessions using a radiometer.²³ After materials application, samples for T1 subdivision were subjected to shear bond strength testing using a Universal Testing Machine (Lloyed testing machine, Lloyed Instrument LR5R series-UK) after one-day storage in distilled water at 37°C using thermostatically controlled incubator, while T2 subdivision was tested after one-month of water storage.

Statistical analysis

Shear bond strength data were presented as mean, standard deviation (SD) and 95% Confidence Interval (95% CI) values. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests which showed that data were normally distributed. Three-way Analysis of Variance (ANOVA) was used in testing significance for the effect of material, substrate, storage time and their interactions on shear bond strength. The significance level was set at $P \leq 0.05$. Statistical analysis was

performed with SPSS 16.0® (Statistical Package for Scientific Studies) for Windows.

RESULTS

The mean shear bond strength for (Z250) to enamel after 1 day was 18.4 ± 1.9 MPa and after 1 month 18.3 ± 1.6 MPa while, the mean shear bond strength for (Z250) to dentin after 1 day was 15.8 ± 1.2 MPa and after 1 month 15.1 ± 1.5 MPa. On the other hand, the mean shear bond strength for (LS) to enamel after 1 day was 9.4 ± 1.3 MPa and after 1 month 9.2 ± 1.1 MPa and while to dentin after 1 day was 13 ± 1.8 MPa and after 1 month 12.9 ± 1.8 MPa.

Z250 showed statistically significant higher mean SBS values than LS. For Z250, there was higher mean SBS values with enamel rather than dentin but not significantly different. On the contrary, Filtek LS showed higher mean SBS values with dentin rather than enamel but also not significantly different. There was no statistically significant difference between mean SBS values of the two substrates and the two storage times. (table 1)

TABLE (1): Comparison between different variables

		Mean	SD	95% CI	P-value
Material	Z250	16.9	2.1	16.2 – 17.6	<0.001*
	LS	11.1	2.4	10.4 – 11.8	
Substrate	Enamel	13.9	4.9	13.1 – 14.6	0.472
	Dentin	14.2	2	13.5 – 14.9	
Storage time	1 day	14.1	3.7	13.4 – 14.9	0.644
	1 month	13.9	3.6	13.2 – 14.6	

*: Significant at $P \leq 0.05$

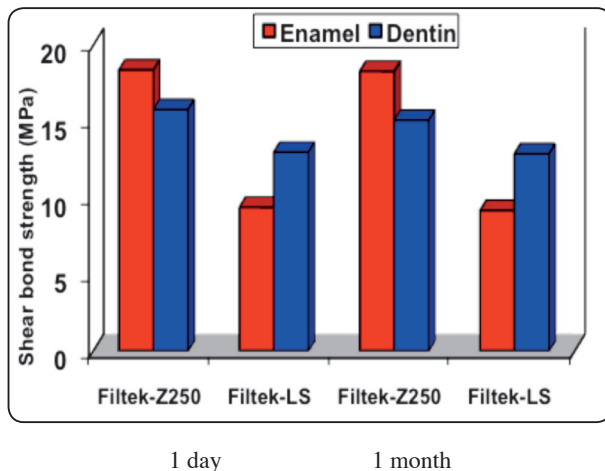


Fig. (2): Means shear bond strength of the two composite materials

DISCUSSION

The ring-opening polymerization of silorane composite occurs with the initiation process of cationic intermediates, which proceed to interact with other oxirane monomers by opening the oxirane ring to further chain propagation. Subsequently, this expansion of ring-opening propagation balances the conversion of intermolecular distances where the discrete species, which previously exist at the van der Waal's force distances, become linked through shorter covalent bonds. This mechanism implies a slight reduction of the initial distance between monomers, which results in a volumetric shrinkage of less than 1%.^{17,21} Low-shrinking composites were developed to relieve stress being imposed immediately on the vulnerable bond to dentine (tooth), thereby aiming to prolong the clinical lifetime of tooth restorations in composite. Hence, we investigated if such a stress-relieving effect could have resulted in a less harmed interface and thus higher remaining bond strength.

Comparing the bonding effectiveness of Filtek-LS to that of Filtek-Z250 in this study, the significantly highest SBS values were recorded for Z250 by which the above Hypothesis should be rejected. This was in agreement with Van Ende et al, who found that microtensile-bond strength values of

conventional composite to dentin were significantly higher than those for Silorane composite. They explained this as due to the stiffer consistency of low-shrinking composites, air bubbles were more frequently enclosed. The pores self-evidently provide a free surface, where the composite can shrink and thus can relieve shrinkage stress, yet may provide structural discontinuities affecting bond strength.¹

Guiraldo et al found that there was a greater degree of subsurface polymerization of the methacrylate-based composites in comparison to Silorane composites and concluded that subsurface polymerization has been shown to have a relation to the bond strengths.²⁴ Furthermore, composition of RBCs is also crucial in assessing the bond strengths. Filtek-LS has lower filler content when compared to Filtek-Z250 and as bond strength increased with increasing filler content, this may in part be responsible for higher bond strength values for methacrylate-based composite resin.⁸

In vitro shear bond strengths are useful and essential for predicting the performance of new composite materials and adhesive systems in comparison to conventional bonding systems. The main objective of the bond strength tests is to determine bonding of an adhesive system to dental hard tissues. In the present study, etch-and-rinse (Single Bond) for Filtek-Z250 and Silorane self-etch bonding systems (SSA) for Filtek-LS were used as bonding systems for shear bond strength testing as Filtek-Z250 gave a significantly higher value of shear bond strength compared to Filtek-LS, which is in accordance with previous literature.^{2,5,26} Erickson et al compared three self-etch adhesive systems and eleven etch-and-rinse systems by measuring shear bond strength and concluded that although chemical bonding may be present for some self-etch systems, it does not compete with the bond produced by etch-and-rinse systems. They explained this as self-etching priming systems combine etching

and priming steps, where the primer is air dried which results in calcium and phosphate ions being solubilized from apatite crystals and suspended in alcohol and water solvents in the primer. When these volatile solvents are evaporated, the concentration of calcium and phosphate may exceed the solubility product constants for calcium phosphate, resulting in its precipitation within the primer. This limits the ability of adhesives to penetrate the primed surface, leading to lower bond strength values.²⁵

Silorane self etch (SSA) requires separate light-curing of the firstly applied SSA-Primer and the secondly applied SSA-Bond, according to manufacturer's instructions, consequently differs from typical two-step self-etch adhesives, of which the successive application of a primer and adhesive resin is finalized by only one light-curing step. Therefore, SSA's bond to tooth enamel/dentin was established in the first application step, similarly as achieved by one-step self-etch adhesives. Considering its interaction with enamel/dentin is limited to a few hundreds of nanometers, the bonding mechanism involves a form of 'nanointeraction', like it was disclosed before for so-called 'ultra-mild' self-etch adhesives. This shallow interaction should be attributed to the relatively high pH of SSA-Primer, which is 2.7 and consequently the low SBS values.^{2,5,14}

For dentin samples preparation, the dental samples were carefully prepared to ensure only the outer dentin was exposed for bonding. Bonding to deeper dentin is complicated by its more heterogeneous structure, variable tubular density and tubular fluid flow. Therefore, bonding to inner dentin may add more variables that could interfere with the ability to evaluate the bond strength.²⁷

There was no significant difference in SBS values between enamel and dentin in the present study, this may be due to the fact that the SBS values with Filtek-Z250 for enamel was higher than for dentin although not significantly different while for Filtek-LS, the SBS values were higher for dentin

rather than for enamel but also with no significant difference.

From the results of this study, there was no significant difference for the effect of the storage times (1 day Vs 1 month), this may be due to the fact that one month is not enough time to determine the durability of the attained bond strength and further studies should be made with long-term bond testing.

CONCLUSION

It is safe to assume, within the limitations of this in-vitro study, that:

- 1) Filtek-Z250 bonds better to enamel and dentin rather than Filtek-LS.
- 2) The low shrinkage effect of Filtek-LS doesn't improve its shear bond strength to enamel and/or dentin.
- 3) Neither tooth substrate nor storage time up to one month has an effect on shear bond strength measurements.

Clinical relevance: Further shear bond strength studies should be carried out with increased storage times.

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