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Incremental Layer Shear Bond Strength of Low-shrinkage Resin Composites Under Different Bonding Conditions

AH Al Musa • HNA Al Nahedh

Clinical Relevance

The chemical compatibility between old and repair composite resins and the use of an appropriate bonding agent are crucial for high incremental bond strength. A silorane-based resin composite should be repaired with a silorane-based composite and silorane adhesive material.

SUMMARY

The purpose of this study was to determine the incremental shear bond strength of a silorane-based composite (Filtek Silorane) repaired with silorane or a methacrylate-based composite (Filtek Z250) under various aging conditions. Also, the incremental bond strength of the silorane-based composite was compared with that of another low-shrinkage methacrylate-based composite (Aelite LS Posterior) under fresh and aged conditions, with and without the use of an adhesive resin between successive layers. The two brands of low-shrinkage composites were compared with a microhybrid, Filtek Z250, which served as the control. Substrate discs were fabricated and second layers were adhered to them immediately, after two weeks of aging, or after four weeks of aging and with and without an adhesive resin. Shear bond strengths were measured and failure modes were evaluated. The incremental bond strength of silorane to the silorane-based composite was not significantly different from that of the methacrylate-based composite. However, repairing a silorane-based composite with a methacrylate-based composite significantly reduced the bond strength. Aelite showed a lower incremental bond strength than Z250 and silorane, but the use of an adhesive significantly improved the bond strength. The absence of an oxygen-inhibited layer did not affect the bond strength of the consecutive layers of the silorane-based composite.

Arwa H. Al Musa, BDS, MSD, King Saud University, Restorative Dental Sciences, Riyadh, Saudi Arabia

*Hend N. A, Al Nahedh, BDS, MSD, King Saud University, Restorative Dental Sciences. Riyadh, Saudi Arabia

*Corresponding author: PO Box 60169, Riyadh, 11545, Saudi Arabia; e-mail: h_nahed@yahoo.com

DOI: 10.2341/13-104-L

INTRODUCTION

Since their introduction to dentistry, resin composite materials have been extensively used by dental professionals. Over the years, these materials have undergone many modifications to improve their characteristics and performance. Nevertheless, they still have some drawbacks, such as poor water stability, poor color stability, and low wear resistance in high-stress areas.[1]

Polymerization shrinkage is another major drawback that could result in microleakage, recurrent caries, cuspal deflection, and postoperative sensitivity.[2] Attempts to overcome these problems include using an incremental layering technique, varying the curing rate or intensity, using a liner with high elasticity at the tooth-restoration interface, modifying the chemical formulation of the resin composite by increasing the filler content, and using a larger monomer with fewer reactive sites.[3-7]

Manufacturers have tried to minimize polymerization shrinkage by modifying resin composites to develop low-shrinkage materials. One example of this is Aelite LS Posterior (Bisco, Schaumburg, IL, USA), a composite material with a high filler volume (75%). The increase in filler levels results in a decrease in the resin volume, thereby reducing the overall amount of polymerization shrinkage.[8] In 2007, a new low-shrinkage silorane-based resin composite material, Filtek Silorane (3M ESPE, Seefeld, Germany), was developed.[9] The reduced polymerization shrinkage attributed to the silorane monomer is the product of the reaction of oxirane and siloxane molecules. This material polymerizes via a cationic ring-opening polymerization reaction, thereby reducing the polymerization shrinkage.[10]

Siloranes exhibit mechanical properties, such as strength, hardness, and modulus of elasticity, that are comparable to those of methacrylate-based composites.[11] In addition, they show less cuspal deflection than methacrylates, which is attributed to their reduced polymerization shrinkage stresses.[12] Silorane-based composites also demonstrate better color stability and gloss retention than methacrylate-based composites.[13]

In clinical situations, it is sometimes necessary to place a composite in increments in areas where the prepared cavity is deep or when repairing an existing composite restoration after it has been in service for some time. Several studies[14,15] have shown that an oxygen-inhibited layer is needed for optimal bonding between consecutive layers of a methacrylate-based composite. The cationic ring-opening reaction of a silorane-based composite is insensitive to oxygen, and no oxygen-inhibited layer is formed on the surface. Therefore, it has been assumed that this would result in decreased bond strength between increments. Furthermore, this is a different class of resin composites and it may be difficult to bond it to preexisting methacrylate-based composites.[16]

The aims of this study were 1) to determine the shear bond strength of a consecutive layer of a silorane-based composite to a silorane-based composite and a silorane-based composite to a methacrylate-based composite under various aging conditions and with and without the use of an adhesive, 2) to compare the bond strength of a silorane-based composite to a low-shrinkage methacrylate-based composite under the same conditions, and 3) to compare the bond strength values of the silorane-based composite and the low-shrinkage methacrylate-based composite with those of a methacrylate to methacrylate-based composites for both fresh and aged conditions and with and without using an adhesive resin between successive layers.

METHODS AND MATERIALS

In this study, two types of low-shrinkage composite materials were tested, Filtek Silorane, and Aelite LS Posterior, and compared to a hybrid methacrylate-based composite (Filtek Z250, 3M ESPE). The adhesives used (Silorane System Adhesive Bond, One Step Plus, and Adper Single Bond 2) were the ones recommended by the manufacturer of each of the brands (Table 1). Filtek Z250, a microhybrid material, was chosen as the control. Following the manufacturer's instructions, 144 specimens were fabricated. Each of the two low-shrinkage composite materials had five groups of 12 specimens each; groups 11 and 12 were the control groups. The test conditions are summarized in Table 2.

The silorane composite was used as a substrate for groups 1-5. In group 1, the substrate was bonded to the silorane composite without using an adhesive. In group 2, the substrate was aged for two weeks and bonded to the silorane composite without using an adhesive. In group 3, the substrate was bonded to the silorane composite after four weeks of aging and without using an adhesive. In group 4, the substrate was aged for four weeks and bonded to the silorane composite using a silorane adhesive. In group 5, the substrate was aged for four weeks and bonded to the Z250 composite using the Adper Single Bond 2 adhesive.

Groups 6-10 were fabricated using the Aelite LS Posterior resin composite as a substrate. In group 6, the substrate was bonded to the Aelite composite without using an adhesive. In group 7, the substrate was aged for two weeks and bonded to the Aelite

Table 1: *Composition of the Resin Composites and Adhesives Used in the Study*

Material	Lot Number	Manufacturer	Material Composition
Composites			
Filtek Z250 (methacrylate based)	N143944	3M ESPE, St Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, silicon dioxide, zirconium dioxide, barium glass, ytterbium trifluoride, mixed oxide perpolymer
Aelite LS Posterior (low shrinkage methacrylate based)	100009783	Bisco, Schaumburg, IL, USA	Ethoxylated bis-GMA, glass filler, amorphous silica
Filtek Silorane (low shrinkage Silorane based)	N175795	3M ESPE, St Paul, MN, USA	Silorane (3,4 epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane), silicon dioxide, ytterbium triflouride
Adhesives			
Adper Single Bond 2	N176361	3M ESPE, St Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, ethanol, water, methacrylate functional copolymer of polyacrylic and polyitaconic acids
One Step Plus	1000010182	Bisco, Schaumburg, IL, USA	Biphenyldimethacrylate, hydroxyethyl methacrylate, acetone, glass frit
Silorane System Adhesive Bond	N170406	3M ESPE, St Paul, MN, USA	TEGDMA, phosphoric acid, methacryloxhexylesters, 1,6-hexanediol methacrylate, bis-GMA, UDMA, bis-EMA

Abbreviations: Bis-GMA, bisphenol A glycol dimethacrylate; bis-EMA, bisphenol A ethoxylated dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

composite without using an adhesive. In group 8, the substrate was bonded to the Aelite composite after four weeks of aging and without using an adhesive. In group 9, the substrate was aged for four weeks and bonded to the Aelite composite using One Step Plus adhesive. In group 10, the substrate was aged for four weeks and bonded to the Z250 composite using the Adper Single Bond 2 adhesive.

Groups 11 and 12 served as positive fresh and aged control groups, respectively. For group 11, the Z250 composite was bonded to the same material immediately and without using an adhesive. For group 12, the Z250 composite was aged for four weeks and bonded to the same material using Adper Single Bond 2 adhesive.

Table 2: *Test Groups and Their Bonding Conditions*

Group	Substrate/Intermediate Layer/Stub	Bonding Condition
1	FS/no adhesive/FS	Fresh bonding
2	FS/no adhesive/FS	Bonding after 2 wk aging
3	FS/no adhesive/FS	Bonding after 4 wk aging
4	FS/Silorane System Adhesive Bond/FS	Bonding after 4 wk aging
5	FS/Adper/FZ250	Bonding after 4 wk aging
6	Aelite/no adhesive/Aelite	Fresh bonding
7	Aelite/no adhesive/Aelite	Bonding after 2 wk aging
8	Aelite/no adhesive/Aelite	Bonding after 4 wk aging
9	Aelite/One Step Plus/Aelite	Bonding after 4 wk aging
10	Aelite/Adper/FZ250	Bonding after 4 wk aging
11	FZ250/no adhesive/FZ250	Fresh bonding
12	FZ250/Adper/FZ250	Bonding after 4 wk aging

Abbreviations: Adper, Adper Single Bond 2; Aelite, Aelite LS Posterior; FS, Filtek Silorane; FZ250, Filtek Z250.

The resin-based substrates were fabricated using disc-shaped stainless steel molds measuring 10 mm in diameter and 3 mm in thickness. Curing was done for 40 seconds using a halogen light curing unit (Elipar 2500 Halogen Curing Light, 3M ESPE) placed 1 mm from the upper surface of the resin for each side. The samples requiring aging were stored in distilled water until the designated time had elapsed.

For the second layer of composite (the stub), the samples were placed in a Teflon alignment mold containing a central cylindrical hole measuring 4 mm in diameter and 2 mm in thickness (Figures 1a and 1b). The substrates were placed in the lower end of the mold and the mold was assembled. Then the designated surface treatment of the composite surface was carried out according to the manufacturer's instructions. The second composite layer (stub) was packed into the upper cylindrical hole and cured for 40 seconds. The samples were carefully removed after two minutes and inspected for defects or any resin flash; defective samples were discarded (Figure 1c).

The samples were stored in 37°C distilled water for one week, during which they were subjected to 5000 cycles of thermal stressing between 5° and 55°C with a dwell time of five seconds. The larger end of the composite sample was embedded in a plastic ring 2.5

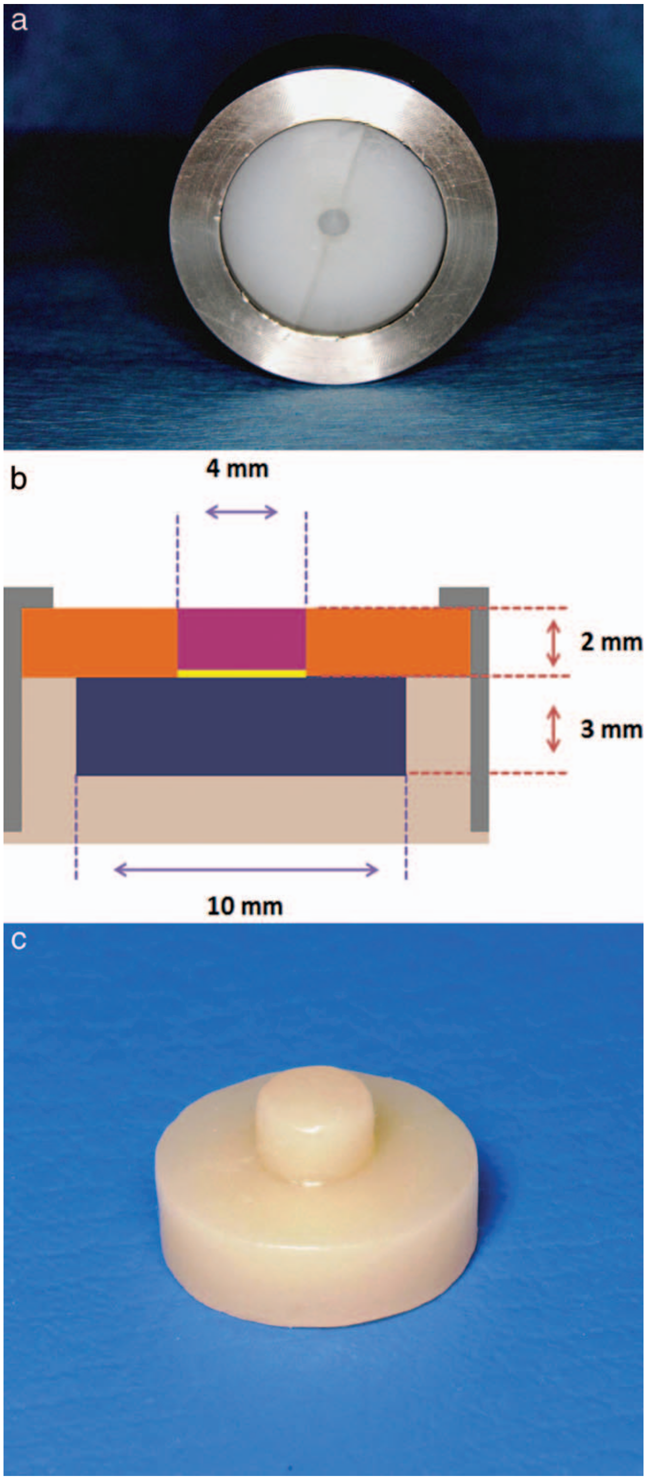

Figure 1. *(a): Teflon alignment mold with metal casing. (b): Schematic diagram of the Teflon alignment mold with metal casing (cross sectional view). (c): Completed sample.*

cm in diameter using Orthoresin (DeguDent GmbH, Hanau, Germany) to fit the specimens to the jig of the Instron machine. Each sample was tested in shear mode using the Instron Universal Testing Machine (Instron 8500, Instron Corp, Norwood, MA, USA) at a cross-head speed of 0.5 mm/min. All results were expressed in megapascal (MPa). All fractured samples were examined using an optical microscope at 50× magnification for failure analysis. Failure modes were categorized as "adhesive" (between the composite substrate and stub), "cohesive" (failure of the composite resin), or "mixed" (cohesive failure of the composite accompanied by adhesive failure at the interface).

The shear bond strength values were analyzed with one-way analysis of variance (ANOVA), and Tukey post hoc analysis at the ($p<0.05$) significance level, using statistical software (SPSS, version 16, SPSS, Chicago, IL, USA)

RESULTS

The mean shear bond strength and standard deviations are presented graphically in Figure 2. One-way ANOVA showed that there was a significant difference among the groups ($p<0.005$) (Table 3).

In the silorane groups, the incremental bond strength values of the fresh specimens tested in group 1 were not statistically significantly different from the fresh Z250 composite, group 11. Aging the silorane discs for two and four weeks (group 2 and 3, respectively) resulted in a significant drop in the incremental bond strength values (20% and 30%, respectively); however, the difference was statistically significant only with group 3 ($p<0.05$).

Group 4 resulted in increased bond strength values that were close to those of group 1 and were not statistically different from those of group 12. On the other hand, group 5 resulted in statistically lower bond strength values compared with groups 1 and 12.

For the Aelite groups, the incremental bond strength values of the fresh specimens (group 6) were slightly lower than those of groups 1 and 11, but the difference was not statistically significant. In addition, aging for two and four weeks (groups 7 and 8, respectively) slightly lowered the bond strength values; however, the difference was not statistically significant compared with group 6.

Bonding the four-week-aged Aelite specimens using One Step Plus adhesive to stubs of the same material (group 9) resulted in bond strength values that were higher than those of group 6; however, the difference was not statistically significant. Furthermore, the incremental bond strength values were not significantly different from those of group 12.

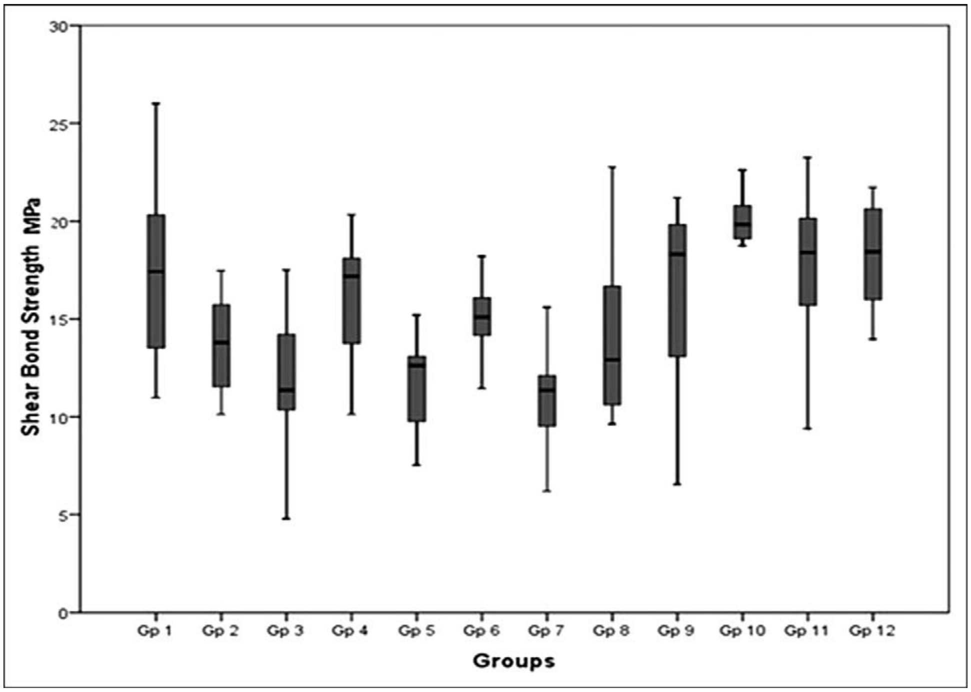

Figure 2. *Box and whisker plot of the shear bond strength values. The upper and lower horizontal lines represent the highest and lowest value, respectively, in each group. The horizontal line within the box represents the median, and the box contains 50% of the values.*

Bonding Z250 specimens to the four-week-aged Aelite stubs using Adper Single Bond 2 adhesive (group 10) resulted in high bond strength values that were not statistically different from those of groups 9 and 1. Similarly, Aelite repaired with Z250 using the Adper adhesive was not significantly different from group 12, but was significantly higher than groups 7 and 8, which were bonded without an adhesive ($p<0.05$).

Mode of Failure

The mode of failure results for the 12 groups are presented in Figure 3. The fresh silorane and Aelite groups bonded without an adhesive showed modes of failure that were either entirely (group 1) or mostly (group 6) cohesive in nature. Group 6 had one specimen fail adhesively.

Aging increased the adhesive mode of failure. After four weeks of aging without using an adhesive,

Table 3: *Homogenous Subsets of Mean Shear Bond Strength Values (MPa)*

Group	Subset for alpha = 0.05			
	1	2	3	4
Group 7: Aelite/no adhesive/Aelite (2 wk)	10.88			
Group 5: FS/Adper/FZ250 (4 wk)	11.52	11.52		
Group 3: FS/no adhesive/FS (4 wk)	11.95	11.95		
Group 2: FS/no adhesive/FS (2 wk)	13.67	13.67	13.67	
Group 8: Aelite/no adhesive/Aelite (4 wk)	14.11	14.11	14.11	
Group 6: Aelite/no adhesive/Aelite (fresh)	14.91	14.91	14.91	
Group 4: FS/Silorane System Adhesive Bond/FS (4 wk)		16.15	16.15	16.15
Group 9: Aelite/One Step Plus/Aelite (4 wk)		16.21	16.21	16.21
Group 1: FS/no adhesive/FS (fresh)			17.23	17.23
Group 11: FZ250/no adhesive/FZ250 (fresh)			17.43	17.43
Group 12: FZ250/Adper/FZ250 (4 wk)			18.23	18.23
Group 10: Aelite/Adper/FZ250 (4 wk)				19.94
Significance between groups	.17	.05	.07	.25

Abbreviations: Adper, Adper Single Bond 2; Aelite, Aelite LS Posterior; FS, Filtek Silorane; FZ250, Filtek Z250.

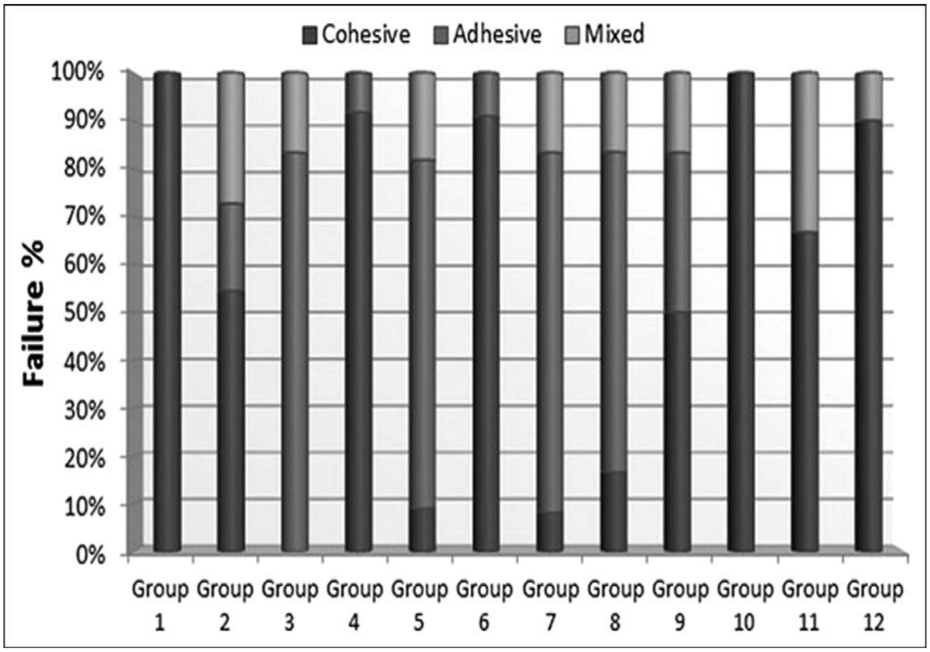

Figure 3. *Diagrammatic representation of the failure modes of the sheared samples for all the tested groups.*

83.3% (10 samples) of the silorane samples and 66.7% (8 samples) of the Aelite samples, groups 3 and 8, respectively, failed adhesively. The use of the silorane adhesive dramatically increased the cohesive type of failure to 91.7% (11 samples) when bonded to the same material after four weeks of aging (group 4), whereas bonding the four-week-aged silorane specimens to the Z250 composite using Adper Single Bond 2 (group 5) resulted in a high percentage of adhesive and mixed failures (91.7%, 11 samples); however, only one specimen failed cohesively.

The use of an adhesive in the Aelite groups increased the cohesive type of failures. When the Aelite substrates were bonded to the same material after four weeks of aging using One Step Plus adhesive (group 9) about half the group failed cohesively, whereas when the Z250 stubs were bonded to the four-week-aged Aelite substrates using the Adper Single Bond 2 adhesive, all the samples failed cohesively.

DISCUSSION

In the current study it was found that the bond strength between immediately placed incremental layers of the silorane composite was not different from that of the methacrylate-based composite, Z250. This shows that the absence of an oxygen-inhibited layer has no effect on the resultant bond strength in consecutive layers. This is in agreement with the findings of Shawkat and others.[16] In their study, they compared the bond strength of incremental layers of silorane composite in oxygen and nitrogen atmospheres. They found no significant difference between the two groups, thus concluding that the absence of an oxygen-inhibited layer did not affect the shear bond strength between two successive layers of silorane-based composites for the fresh conditions. However, within the confines of this study, the effect of this layer on the aged specimens could not be ruled out completely. In addition, there was no difference between the silorane-based composite and the methacrylate-based composite in incremental bond strength values.

The other low-shrinkage material tested in this study was Aelite LS Posterior, which is a methacrylate-based resin composite material with a high filler content (75% vol). The incremental bond strength of this material after immediate placement was lower than that of Silorane and Z250. There is no agreement among researchers on the minimal acceptable limit of composite repair bond strength. However, Teixeria and others[17] suggested that the shear bond strength of repaired composites should range from 15 MPa to 25 MPa. The bond strength of the Aelite group was lower than that limit. This may be the result of the higher viscosity of this material caused by its higher filler level. Composites with a low viscosity are believed to offer better adaptation to the repaired composite resin surface, thereby increasing retention through micromechanical interlocking by flowing into the irregularities of the material's surface.[18] Furthermore, the high filler content of Aelite LS Posterior increases the material's stiffness. Aelite has a high elastic modulus compared with the Z250 and Silorane composites.[19] As a result, Aelite exhibits more contraction stresses at the adhesive interface even though it has low contraction shrinkage.[20] These stresses may have affected the bond strength of the material. However, this issue is controversial as other studies have found that Aelite demonstrated less stress values compared with the Z250 composite.[19,21]

Aging for two and four weeks decreased the bond strength for the two low-shrinkage composites tested. The decrease was statistically significant for the Silorane groups but not for the Aelite groups. In addition, a greater number of adhesive failures were seen between the incremental layers for both groups. This is in agreement with the findings of Shawkat and others[16] and Tezvergil-Mutluay and others.[22] Aging negatively affects the resin composites by causing water sorption, chemical degradation, and leaching out of some of the constituents of the material. Also, the amount of available unreacted double bonds is reduced, which, in turn, decreases the chemical reactivity of the material. All these changes result in a diminished bond strength between the aged sample and the repair composite material. [23,24]

Surprisingly, the repair strength of the Aelite specimens that were stored for four weeks (group 8) was not statistically different from that of the fresh group. However, when the failure modes were examined, the fresh layering specimens had a 9.1% (1 sample) adhesive failure while 66.7% (8 samples) showed adhesive failure in group 8. This indicates a diminished adhesive potential that could lead to a problem with the reliability and longevity of the repair.

After four weeks of aging, the bond strength between the incremental layers of the silorane composite without using an adhesive (group 3) was significantly lower than that of the fresh Silorane and fresh Z250 groups without using an adhesive (groups 1 and 11, respectively). Furthermore, the mode of failure in group 3 was mostly adhesive, in contrast to all cohesive failure in groups 1 and 11. Because the cationic ring-opening polymerization reaction is insensitive to oxygen, the incremental bond strength is dependent on the chemical reactivity of the material, which decreases over time. The chemical reactivity of silorane-based composite materials seems to undergo a significant drop with aging that could cause a low incremental bond strength in the Silorane restorations.[22]

In a clinical situation, clinicians usually remove the superficial layer of the old composite and roughen the surface before repairing the composite material. In addition, an intermediate layer, such as an adhesive material or a flowable composite material, is used to bind the old composite material to the new.[25,26] Therefore, the effect of the presence or absence of the oxygen-inhibited layer is eliminated. The resultant bond strength would therefore depend on the compatibility of the two types of composites and on the intermediate adhesive layer, if used.

In the present study, all the groups using an adhesive material between the layers showed high bond strength values, which were statistically similar to those of fresh bonding. The only exception was the repair of Silorane with Z250 using Adper Single Bond 2 adhesive (group 5). When the silorane was repaired after four weeks of aging using the silorane composite and adhesive (group 4), the bond strength increased to 93.7% of the value for fresh bonding and the predominant mode of failure was cohesive. In contrast, the four-week-aged group that was repaired without an adhesive (group 3) failed mostly adhesively. However, the use of a methacrylate-based adhesive and restorative material for the repair (group 5) resulted in a bond strength that was significantly lower than that of the fresh group (group 1). This is apparently due to the different chemical compositions of the two materials, which had an adverse effect on the bond strength between them. Shawkat and others[16] and Tezvergil-Mutluay and others[22] reported similar results regarding the incompatibility of the restorative composite resins. Therefore, the chemical compatibility between the old and the repair composite resins and use of the appropriate bonding agent are crucial for high incremental bond strength.

Although the repair of the aged Silorane substrates with Z250 drastically decreased the bond strength values of the specimens, it had a totally opposite effect on Aelite. Repairing the four-week-aged Aelite using the Z250 composite and Adper Single Bond 2 adhesive (group 10) resulted in a bond strength that was higher than that of both the fresh Aelite group (group 6) and the four-week-aged group repaired using the same material and One Step Plus adhesive (group 9). Furthermore, all the samples in group 10 failed cohesively. This result could be due to the lower viscosity of the Z250 composite compared with Aelite, which may have promoted better adaptation and, therefore, resulted in a higher bond strength. In addition, Z250 has a lower elastic modulus compared with Aelite, which might have relieved the contraction stresses at the interface, thereby increasing the bond strength.

Further research is needed to examine the effect of C-factor on the incremental bond strength of different low-shrinkage composites. In addition, a further examination of the surface preparation procedures, such as roughening, removing the superficial layer, or using an adhesion promoter on the incremental bond strength of low-shrinkage resin-based composites, is warranted.

CONCLUSIONS

Within the limitations of this study, it can be concluded that

- Silorane-based resin composite incremental bond strength is comparable to that of methacrylate-based composite and is not affected by the absence of the oxygen-inhibited layer.
- Silorane-based resin composite should be repaired with the same material and a silorane adhesive should be used to gain adequate bond strength; repairing it with a methacrylate-based resin composite results in a significantly lower bond strength value.
- The low-shrinkage composite Aelite exhibited a lower incremental bond strength than Z250 and the silorane composite.
- Aelite low-shrinkage composite can be repaired with other brands of methacrylate-based resin composites without a deleterious effect on bond strength.

Acknowledgements

The authors would like to extend their appreciation to Professor Salwa Khair and Professor Wedad Awlya for reviewing the manuscript, King Saud University College of Dentistry Research Center, Mr Bong Tuazon for his assistance in the lab work, and last but not least, Dr Shaikh Shaffie for his help in the statistical analysis.

Note

This work was based on a thesis submitted by Arwa Al Musa to the graduate faculty of King Saud University in partial fulfillment of the requirements for the MSD degree.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 2 December 2013)

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