

Effect of 1-bottle Light-cured Adhesive Acidity on Microleakage of a Self-cured Composite

S Kimyai • S Savadi Oskoe

Clinical Relevance

The high *in vitro* microleakage values, with some combinations of 1-bottle light-cured adhesives and self-cured composite core materials, could be clinically significant.

SUMMARY

This *in vitro* study examined the effect of 1-bottle light-cured adhesives with different acidities on the microleakage of a self-cured composite in Class II restorations. Forty-five Class II cavities were prepared on the proximal surfaces of extracted human intact third molars that were randomly assigned to 3 groups (15 cavities in each group). The following adhesives were applied according to the manufacturer's instructions: Single Bond (Group 1), Excite (Group 2) and One-Step (Group 3). The teeth were restored with a combination of a self-cured resin composite (Concise) and a light-cured resin composite (Filtek P60). After thermocycling (500 cycles at 5°C/55°C) and immersion in 2% basic Fushin, the teeth were sectioned and evaluated for

microleakage using a stereomicroscope (16x). Microleakage was scored on a scale of 0 to 3. Kruskal-Wallis analysis showed that there was a significant difference in microleakage values of 3 adhesives with a self-cured composite, and low pH may have contributed to high microleakage values. Also, 2 by 2 comparisons with the Mann-Whitney U-test revealed a significant difference at the $p < 0.05$ level between Groups 1 and 2 and between Groups 2 and 3. However, there was no statistically significant difference between Groups 1 and 3.

INTRODUCTION

One-bottle light-cured adhesives have been commercially available for several years.¹ These adhesives contain a combination of hydrophilic and acidic monomers, reactive diluent monomers, high viscosity adhesive resins and photoinitiator systems, all provided in a 1-bottle solution containing ethanol or acetone as suitable solvents. These adhesive systems have a low and varied pH.² The adhesive layer that is in direct contact with oxygen will not be completely photocured. This layer contains acidic monomers that are in direct contact with the subsequently applied composite. Thus, it is speculated that these adhesive systems might lead to incompatibility with the resin materials,

*Soodabeh Kimyai, DDS, MS, assistant professor, Department of Operative Dentistry, Dental School, Tabriz University of Medical Sciences, Tabriz, Iran

Siavash Savadi Oskoe, DDS, MS, assistant professor, head of Department of Operative Dentistry, Dental School, Tabriz University of Medical Sciences, Tabriz, Iran

*Reprint request: Tabriz Dental School, Gholghasht Street, Tabriz, Iran Post Cod: 5166614713; e-mail: kimyais@tbzmed.ac.ir

DOI: 10.2341/05-139

depending on the polymerization mode (chemical, light or dual activation).²⁻⁴

Self-cured composites are currently used in dentistry, primarily as core buildup materials, and their suitability as core materials has been demonstrated.⁵⁻⁷ Factors, such as time and simplicity of the clinical steps, have led to an increased use of light-cured 1-bottle adhesives in association with resin composite core materials.⁸ However, clinicians have reported bonding failures when self-cured composite core materials were bonded with 1-bottle light-cured adhesive systems.^{2,9} The decrease in bond strength of self-cured composites to dentin was inversely proportional to the acidity of these 1-bottle systems.¹⁰ Adverse chemical interactions between unpolymerized acidic adhesive resin monomers and a basic tertiary amine catalyst in the composite was thought to be responsible for the observed incompatibility.¹¹ If this incompatibility exists, the risk for a large number of composite core buildups to prematurely debond is substantial. Even if mechanical retentions were incorporated, the resultant microleakage would predispose such core buildups to early failure.⁹ In addition, the relationship between bond strength and microleakage is not clearly understood, and a significant number of studies have failed to demonstrate a relationship between bond strength and microleakage.¹²⁻¹⁴

Little is known about the effect of the acidity of 1-bottle light-cured adhesives on the microleakage of self-cured composites. This *in vitro* study investigated the effect of 1-bottle light-cured adhesives with different acidities on the microleakage of a self-cured composite in Class II restorations.

METHODS AND MATERIALS

Twenty-four extracted intact human third molars were selected for this study. The teeth were randomly assigned to 3 groups (8 teeth in each group). They were

stored in a 1% chloramine solution and used within 2 months following extraction. Gentle scaling was performed on the teeth to remove all traces of calculus from the cemento-enamel junction. In 7 teeth in each group, 2 box-shaped Class II cavities (mesio-occlusal and disto-occlusal) were prepared with 014 diamond burs (Diatech Dental AG, Swiss Dental Instruments, CH-9435 Heerbrugg) using an air/water cooled high speed turbine. In the remaining tooth in each group, a box-shaped Class II cavity was prepared on 1 of the proximal surfaces, totaling 15 cavities in each group. A new bur was used after every 5 preparations. The dimensions of the proximal cavities were 3 mm buccolingually and 1.5 mm pulpally. The cervical margins were positioned 1 mm beyond the CEJ. The margins were not beveled.

In all groups, enamel and dentin were etched for 15 seconds with 35% phosphoric acid gel (3M Scotchbond Etchant, St Paul, MN, USA), rinsed thoroughly for 20 seconds and lightly air dried without desiccating. This was followed by the application of adhesive systems Single Bond (3M Dental Products) in Group 1, Excite (Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein) in Group 2 and One-Step (BISCO Inc, Schaumburg, IL, USA) in Group 3. A Tofflemire matrix retainer and metal band (Tofflemire Type Matrix Bands, #1 universal, Arnel Dental Products, Hempstead, NY, USA) were then placed on the teeth. The matrix was tightened and held by finger pressure against the gingival margin of the cavity so that the preparations would not be over-filled at the gingival margin.

Equal quantities of base and catalyst of the self-cured composite (Concise, 3M Dental Products) were mixed and placed in the cavity gingival floor 2-mm thick and allowed to cure for 3 minutes. To ensure the thickness of the layers, a comparison between the former occluso-gingival dimension of the cavity and the dimension after material placement was carried out using a probe. If the thickness of the self-cured composite was less than 2 mm, self-cured composite was added to ensure a

Table 1: Protocol for Various Adhesive Systems

Adhesive System	Batch #	Manufacturer	Components	Protocol
Single Bond	20040917	3M Dental Products	Bis-GMA, PAAC, HEMA, Dimethacrylates, Ethanol, Photoinitiators, Water	<ul style="list-style-type: none"> • Apply 2 consecutive coats, thoroughly air dry • Light cure for 10 seconds
Excite	F68154	Ivoclar Vivadent	HEMA, Dimethacrylates, Phosphonic acid acrylate, Silicon dioxide, Ethanol, Photoinitiators	<ul style="list-style-type: none"> • Apply 1 coat, gently agitate for 10 seconds, thoroughly air dry • Light cure for 20 seconds
One-Step	0400002931	BISCO, Inc	Bis-GMA, HEMA, Photoinitiators, BPDM, Acetone	<ul style="list-style-type: none"> • Apply 2 consecutive coats, gently agitate for 10 seconds, thoroughly air dry • Light cure for 10 seconds

Bis-GMA, bisphenol-glycidyl-methacrylate; PAAC, polyalkenoic acid co-polymer; HEMA, 2-hydroxyethyl methacrylate; BPDM, bisphenyl dimethacrylate

thickness of 2 mm; however, if the thickness of the self-cured composite was more than 2 mm, the sample was excluded from the study and another sample was prepared. Immediately after setting the self-cured composite, the remainder of the box was restored with the light-cured composite Filtek P60 A3 (3M Dental Products), using an oblique technique. Each layer was polymerized from the occlusal aspect for 40 seconds with a conventional quartz halogen LC unit (Astralis 7, Ivoclar Vivadent) at a light intensity of 400 mwcm⁻² immediately after placement. All the restorations were finished with finishing diamond burs (Diamant GmbH, D & Z, Goerzallee 307, Berlin, Germany) and finishing disks (Sof-Lex, 3M ESPE, Dental Products).

The specimens were then stored in distilled water for 24 hours at room temperature. All specimens were subjected to 500 cycles of 5°C/55°C water with a 30-second dwell time at each temperature. The apices of the teeth were sealed with utility wax, and all tooth surfaces were covered with 2 coats of fingernail varnish with the exception of 1 mm around the tooth-restoration interface. The teeth were immersed in 2% basic Fushin for 48 hours, then removed, rinsed with tap water, embedded in a self-cured acrylic resin and sectioned with a diamond disk (Diamant GmbH, D & Z, Goerzallee 307, Berlin, Germany) along the mesio-distal direction, corresponding to the center of the restoration. All samples were prepared by one operator. Two examiners evaluated both hemisections of each tooth at 16x using a stereomicroscope (Olympus SZX9; Olympus, Tokyo, Japan) for dye penetration at the cervical margins. The examiners were blind as to which group the teeth belonged. Microleakage was scored on a 0 to III scale:

- 0. No dye penetration.
- I. Dye penetration up to half of the cavity depth.
- II. Dye penetration greater than scale I up to the axial wall.
- III. Dye penetration greater than scale II.

Any discrepancies between the 2 main examiners were reevaluated and, when necessary, a third examiner determined the score.

PH Measurement of 1-bottle Adhesives

Water-free adhesives dissolved in polar solvents do not normally dissociate into ionic species required for pH measurement.² To circumvent this problem, 2 ml of each adhesive was collected and dispensed into a clean glass vial containing 3 ml of 70% ethanol and 30% water, which was diluted from absolute ethanol (ACS analytical grade, Merck Sharp & Dohme, Germany). The mixture was stirred continuously for 5 minutes, and the pH value of the adhesive solution was measured at ambient temperature (20-25°C) using a digital pH meter (Methrom, Model 744, Sweden). These read-

Table 2: pH Values of Different 1-bottle Adhesives

1-bottle adhesives	Adhesive pH Mean ± SD
Single Bond	3.60 ± 0.03
Excite	2.25 ± 0.06
One-Step	4.60 ± 0.04

Table 3: Cementum Leakage Scores

Groups	0	I	II	III	Total
1	8	2	2	3	15
2	0	0	1	14	15
3	12	2	0	1	15
Total	20	4	3	18	45

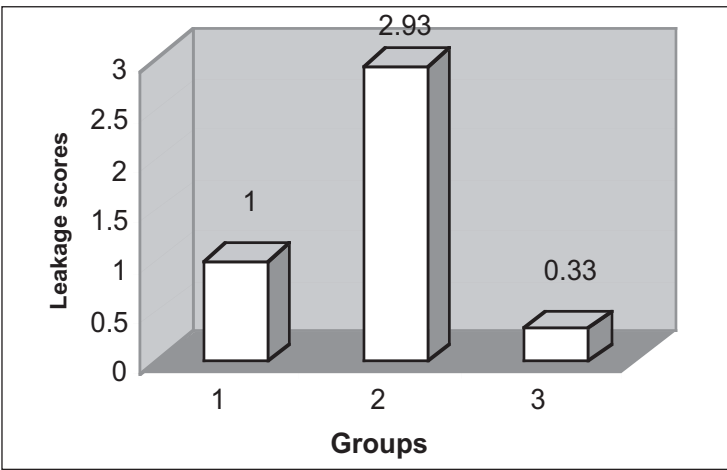


Figure 1: Cementum mean leakage scores (Minimum = 0, Maximum = 3).

ings were taken 3 times for each adhesive, from which the mean pH value was calculated (Table 2).

The correlation between the acidity of the adhesives and their microleakage values was analyzed by non-parametric Kruskal-Wallis test, then the groups were analyzed with the Mann-Whitney U test at the *p*<0.05 level of significance to evaluate differences between the groups.

RESULTS

Table 3 shows the leakage values for the different groups. Statistical analysis using the non-parametric Kruskal-Wallis test indicated significant differences in microleakage values among the 3 adhesives with a self-cured composite (*p*<0.05). The mean microleakage scores are shown in Figure 1. The lowest microleakage values were obtained from One-Step (0.33), followed by Single Bond (1.00) and Excite (2.93). The Mann-Whitney U-test showed a significant difference at the *p*<0.05 level between Groups 1 and 2 and between Groups 2 and 3. However, there was no statistically significant difference between Groups 1 and 3.

DISCUSSION

The results of this study indicate a positive correlation between the acidity of the 3 adhesives and the microleakage values of a self-cured composite. Although there is a lack of literature on the microleakage of 1-bottle light-cured adhesives used with self-cured composite core materials, numerous studies have measured the bond strengths of 1-bottle light-cured adhesive systems in combination with self-cured composites.^{4,7,9} The literature has reported that there is an inverse relationship between the acidity of these 1-bottle adhesives and the bond strength obtained from the use of self-cured composites.^{2,8}

According to this study, the positive correlation between acidity and microleakage could be attributed to the acid-base reaction between the acidic monomers in the oxygen inhibited layer and the tertiary amines from the self-cured composites, since these amines, in association with benzoyl peroxide, are responsible for the polymerization process.^{8,15} To date, commercially available adhesive systems are cured via a free radical vinyl polymerization mechanism. When an adhesive is light cured in the presence of atmospheric air, an oxygen inhibition layer is inevitably formed due to oxygen's ability to quench free radicals. In the case of light-cured 1-bottle adhesives, the oxygen inhibition layers contain, in addition to other dimethacrylates, acidic vinyl monomers with carboxylic or phosphate ester groups. When these adhesives are used together with self-cured composites, there is an interaction of the residual acidic resin monomers from the adhesive inhibition layer, with the binary peroxide-amine catalytic components that are commonly employed in self-cured composites.² Acidic resin monomers are polymerized poorly in the presence of peroxide-amine redox systems, as the tertiary amines are neutralized by acidic resin monomers and lose their ability to reduce agents in redox reactions.¹⁶ The lower degree of polymerization of resin monomers can cause poor adhesion and impaired physical properties.^{8,17-18}

In this study, One-Step, being the least acidic of the 3 adhesives, exhibited the lowest microleakage values. In previous studies, One-Step performed well in bond strength tests in *in vitro* studies due to its higher pH than the other systems.^{2,6} Also, in a recent study in a One-Step/self-cured composite subgroup, ultrastructural features, such as an enormous number of voids within the adhesive layer or the presence of a discrete microporous inhibition film found in the other subgroups, were not found in One-Step.²

According to the results of this study, there was a trend, although not statistically significant, for more microleakage in the Single Bond group compared to the One-Step group. Sanares and others² reported that microtensile bond strength in the Single Bond/self-

cured composite subgroup was significantly lower than the One-Step/self-cured composite subgroup. The authors stated that it could be attributed to the pH value of Single Bond, which is more acidic than the pH value of One-Step. Furthermore, ultrastructural observations in the Single Bond group represented an area that is highly concentrated in the polyalkenoic acid copolymer.² The difference in results found in this study, compared to the results by Snares and others, can be attributed to the difference in test methods.

In this research study, Excite, being the most acidic of the 3 adhesives, exhibited the highest microleakage values. This adhesive system contains phosphonate monomers. It is possible that the amine-peroxide system is affected by the presence of acidic monomers, which result in a lower degree of polymerization and high microleakage values.

This study utilized only 1 vertical section in a mesiodistal direction through the proximal box to evaluate microleakage, which is consistent with previous studies.¹⁹⁻²⁰ Further studies should evaluate microleakage using multiple-surface evaluations or 3-dimensional methods to provide additional microleakage information. Thus, further research on other self-cured and dual-cured resin composite core materials and their suitability with other 1-bottle light-cured adhesives is warranted. In addition, further research into microleakage should be conducted with 1-step and 2-step self-etching adhesives, because they are aggressive and even more acidic than the 1-bottle adhesives investigated in this study. Until further experiments are carried out, the use of self-cured composites with 1-bottle light-cured adhesive systems should be restricted to combinations of materials that have been adequately tested or a ternary redox catalyst should be used. To overcome this incompatibility, a number of ternary redox catalysts have been introduced.²¹ Several 1-bottle total-etch adhesives are now supplemented with an additional bottle of activator solution containing a sodium salt or aryl sulphonic acid to enable them to bond to self-cured or dual-cured composites. A co-initiator or a ternary catalyst reacts with acidic resin monomers to produce either phenyl or benzene sulphonyl free radicals that initiate polymerization of self-cured composites.¹⁶

CONCLUSIONS

Considering the limitations of this study, a positive correlation was observed between the acidity of 1-bottle light-cured adhesives and the microleakage values of a self-cured composite. It can be concluded that the low pH values of 1-bottle light-cured adhesives may contribute to high microleakage values.

Acknowledgements

The authors thank Dr Kamran Sadaghat for statistical analysis of the data. Furthermore, the authors thank Dr Salar Hemmati for his technical assistance in pH measurement.

(Received 10 October 2005)

References

- Vargas MA, Cobb DS & Denehy GE (1997) Interfacial micro-morphology and shear bond strength of single-bottle primer/adhesives *Dental Materials* **13**(5) 316-324.
- Sanares AM, Itthagarum A, King NM, Tay FR & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17**(6) 542-556.
- Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69**(10) 1652-1658.
- Tay FR, King NM, Suh BI & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *The Journal of Adhesive Dentistry* **3**(3) 207-225.
- O'Keefe KL & Powers JM (2001) Adhesion of resin composite core materials to dentin *The International Journal of Prosthodontics* **14**(5) 451-456.
- Swift EJ Jr, Perdigão J, Combe EC, Simpson CH 3rd & Nunes MF (2001) Effects of restorative and adhesive curing methods on dentin bond strengths *American Journal of Dentistry* **14**(3) 137-140.
- Foxton RM, Nakajima M, Tagami J & Miura H (2003) Bonding of photo and dual-cure adhesives to root canal dentin *Operative Dentistry* **28**(5) 543-551.
- Franco EB, Lopes LG, D'alpino PH, Pereira JC, Mondelli RF & Navarro MF (2002) Evaluation of compatibility between different types of adhesives and dual-cured resin cement *The Journal of Adhesive Dentistry* **4**(4) 271-275.
- Hagge MS & Lindemuth JS (2001) Shear bond strength of an autopolymerizing core buildup composite bonded to dentin with 9 dentin adhesive systems *Journal of Prosthetic Dentistry* **86**(6) 620-623.
- Cheong C, King NM, Pashley DH, Ferrari M, Toledano M & Tay FR (2003) Incompatibility of self-etch adhesives with chemical/dual-cured composites: Two-step vs one-step systems *Operative Dentistry* **28**(6) 747-755.
- Tay FR, Suh BI, Pashley DH, Prati C, Chuang SF & Li F (2003) Factors contributing to the incompatibility between simplified-step adhesives and self-cured or dual-cured composites. Part II. Single-bottle, total-etch adhesive *The Journal of Adhesive Dentistry* **5**(2) 91-105.
- Prati C, Simpson M, Mitchem J, Tao L & Pashley DH (1992) Relationship between bond strength and microleakage measured in the same Class I restorations *Dental Materials* **8**(1) 37-41.
- Fortin D, Swift EJ Jr, Denehy GE & Reinhardt JW (1994) Bond strength and microleakage of current dentin adhesives *Dental Materials* **10**(4) 253-258.
- Guzman-Armstrong S, Armstrong SR & Qian F (2003) Relationship between nanoleakage and microtensile bond strength at the resin-dentin interface *Operative Dentistry* **28**(1) 60-66.
- Cook WD & Standish PM (1983) Polymerization kinetics of resin-based restorative materials *Journal of Biomedical Materials Research* **17**(2) 275-282.
- Suh BI, Feng L, Pashley DH & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *The Journal of Adhesive Dentistry* **5**(4) 267-282.
- Beznos C (2001) Microleakage at the cervical margin of composite Class II cavities with different restorative techniques *Operative Dentistry* **26**(1) 60-69.
- Dong CCS, McComb D, Anderson JD & Tam LE (2003) Effect of mode of polymerization of bonding agent on shear bond strength of auto cured resin composite luting cements *Journal of the Canadian Dental Association* **69**(4) 229-234.
- Jain P & Belcher M (2000) Microleakage of Class II resin-based composite restorations with flowable composite in the proximal box *American Journal of Dentistry* **13**(5) 235-238.
- Chuang SF, Liu JK, Chao CC, Liao FP & Chen M (2001) Effects of flowable composite lining and operator experience on microleakage and internal voids in Class II composite restorations *Journal of Prosthetic Dentistry* **85**(2) 177-183.
- Tay FR, Pashley DH, Yiu CK, Sanares AM & Wei SH (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part I. Single-step self-etching adhesive *The Journal of Adhesive Dentistry* **5**(1) 27-40.