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Effect of Curing Protocol on Bond Strength of Dual-cure Self-etching Adhesives

Sara A. Botros [1] ; Mohammed S. Nassif [2] ; Farid S. El-Askary [3]

Abstract

Purpose: The purpose of this study was to evaluate the effect of curing protocols of two dual-cure one-step self-etching adhesives on shear bond strength (SBS) of chemical-cure composite to dentin.

Materials and Methods: A total of 60 anterior bovine teeth were randomly assigned to 6 groups (n=10) according to the two experimental factors being investigated (adhesive type and curing protocol). Labial dentin surfaces were exposed, embedded in acrylic resins and wet ground using #600-grit SiC paper. The adhesives were applied following the manufacturers' instructions and polymerized according to the respective curing protocol. The chemical-cure composite were built-up over the adhesives. The specimens were submitted to shear test until failure; then evaluated for fractographic analysis using a digital microscope. ANOVA and Student t-test were used to analyze the data.

Results: Two-Way ANOVA revealed that the curing protocol and the interaction between the two experimental variables had significant effect on SBS (P=0.000); while the adhesive type showed no effect on SBS (P=0.090).

Conclusion: Increasing light-curing distance affected only the single-dose adhesive. In the self-cure protocol, adhesives failed to produce acceptable bond strength.

KEYWORDS:

Dual cure, self etching adhesives, shear bond strength, chemical cure composite.

1. Master's student, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.
2. Associate Professor, Dental Biomaterials Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.
3. Professor, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.

Introduction

The rapidly rising demand for simplification resulted in development of adhesives that combine all the bonding procedures in a single step.[1] One-step self-etching adhesives became appealing to dental practitioners because of their ease of use and reduced procedure steps.[2]

Optimizing the adhesion to the dental hard tissues is a decisive factor to consider for enhancing the reliability and longevity of these restorations. Since, light activation of adhesives has become a primary mode of polymerization,[3] the quality of the light source used to photo-activate the adhesives is of paramount importance in the polymerization reaction. A minor change in light curing intensity seems to cause a dramatic change in the degree of conversion within the resin material.[4] One of the main factors influencing light intensity is the distance of the light guide tip from the surface of the resin to be exposed.[5] Clinically, there are indeed some cases where it is difficult to control the light curing distance as in proximal boxes.[6] Additionally, many situations entail that the bonding agent undergoes auto-polymerization because of inaccessibility to the visible light source, such as bonding fiber posts and indirect coronal restorations and using dual/chemical-cured core build-up resin composites.[7] In this context, the choice between light-curing and self-curing modes depends on the clinical situation. -

Self-cure composites, as core foundation materials and resin cements, are still frequently used in dental practice especially in areas not readily accessible to light.[8] Unfortunately, an adverse acid-base reaction between unreacted acidic monomers of self-etching adhesives and tertiary amines of self-/dual-cure composites has been reported.[9]

When both photo- and chemical co-initiators are combined in the adhesive material, it is said to be 'dual-curing'.[10] The aim of this dual setting mechanism is to improve the polymerization, especially at areas remote from the light source. So, whenever light irradiation is hampered to reach the adhesive, dual-curing systems are the better choice.

On the basis of these considerations, the present study was conducted to investigate the effect of different curing protocols of dual-cure one-step self-etching adhesives on the bond strength of a chemically-cured resin composite to dentin.

MATERIALS AND METHODS

Two dual-cure one-step self-etching adhesives (Futurabond M+/FBM+DC; Futurabond U/FBU, VOCO GmbH, Germany) and one chemical-cure resin composite (Rebilda SC; VOCO GmbH, Germany) were used in this study.

A total of 60 anterior bovine teeth were randomly divided into 6 groups (n=10/group) according to the two experimental factors of the study: 1) Adhesive type (n=20), 2 groups; 2) Curing protocol (n=30), 3 groups (the adhesives were light cured either at 1 mm or 10 mm light curing distance before the application of resin composite; or allowed to set chemically with the resin composite).

Tooth preparation

The incisal halves of the collected teeth were cut off using low speed diamond discs (Komet, Brasseler, Germany) under running water and the enamel of the cervical halves were wet ground using #180-grit SiC paper mounted on a custom-made laboratory grinder to expose flat dentin surface. The roots were then removed at the level of cemento-enamel junction using the low speed diamond discs under copious water irrigation. The pulp was extirpated and the pulp chamber was irrigated with 10 ml distilled water.

An adhesive tape was placed on one side of a specially fabricated metal mold that consisting of multiple holes, 20 mm diameter x 20 mm depth each. The mold was turned over and the dentin surfaces were securely fixed over the adhesive

tape at the bottom of each hole. Auto-cured acrylic resin (Acrostone, Acrostone Dental Manufacture, Egypt) was poured inside each hole. The mold was immersed subsequently in cold tap water to reduce the heat rising during setting reaction of acrylic resin. After setting of the acrylic resin, the acrylic resin blocks with dentin specimens were taken out from the mold. The dentin surfaces were re-ground with #180-grit SiC paper under copious water flow, to remove excess acrylic resin material, if found. Dentin surfaces were wet ground for 30 seconds using #600-grit SiC paper to create a standardized smear layer.

Bonding procedures

A double-faced adhesive tape with a central hole of 5 mm in diameter was fixed at the middle of the dentin surface. Each tested adhesive was applied to the dentin surface through the hole according to the manufacturer's instructions.

One drop of FBM+DC adhesive and one drop of dual-cure activator (DCA) were mixed into a mixing palette for 3 seconds to create a homogenous mix; then applied over the dentin surface using the applicator brush and gently rubbed for 20 seconds. The adhesive layer was gently dried with oil-free air for 5 seconds. The adhesive was subsequently light cured for 10 seconds either at 1 mm or 10 mm before the application of the resin composite, or left to set chemically with the overlying composite.

The Single Dose unit of FBU was activated by pressing the blister. The foil of the dispensing chamber was pierced with the applicator brush and the liquid was stirred thoroughly using the brush to create a homogenous mix. The adhesive was applied to the dentin surface and the steps have proceeded in the same manner as FBM+DC.

The air-drying pressure was calibrated to 1 bar using a pressure regulator connected to the triple air-way syringe of the dental unit. Light curing was performed using a halogen light curing unit (3M 2500, 3M, USA), with an output of 600 mW/cm^2. The output was periodically checked using a hand-held radiometer (Demetron 100, Kerr, USA). In order to standardize the light curing distance, the light curing gun was fixed on a metal stand that was adjusted according to the required distance from the dentin surface. This distance was measured with the aid of a digital caliper.

A transparent polyethylene tube (4 mm internal diameter x 2 mm height) was placed over the double-faced adhesive tape. The chemical-cure resin composite was injected into the silicon tube by means of an auto mix tip supplied by the manufacturer. A polyester strip was applied over the resin composite and gently pressed to extrude excess material. The resin composite was left to set chemically under a 400 g load for 5 minutes. After setting, the adhesive tape was removed and the specimen was stored in 100% relative humidity at room temperature for 1 hour. Afterwards, the tube was cut off using a surgical blade #15, excess adhesive around resin composite cylinder was carefully removed with the blade and the teeth were stored in 100% relative humidity at room temperature for 24 hours before shear bond strength testing.

Shear Bond Strength Test (SBS):

Each specimen was attached to the lower jig of the Universal Testing Machine (LR5K, Lloyd materials testing, UK) using a specially designed metallic holder. A chisel-shaped shearing blade was fixed to the upper jig of the machine and positioned as close as possible to the resin/dentin interface. Shear test was run at a crosshead speed of 0.5 mm/min until failure. The shear bond strength was calculated by dividing the load (Newton) over the respective surface area (mm^2). Each de-bonded dentin surface was evaluated for fracture analysis using a digital microscope at x40 (Veho VMS-004 Discovery Deluxe USB Microscope, UK). The fracture modes were classified as follow:

1. Adhesive failure: The fracture occurred at the adhesive/dentin interface.
2. Mixed failure: The fracture occurred at the adhesive/dentin interface accompanied with part of the resin composite left on the dentin surface.
3. Cohesive failure: The failure occurred within the resin composite or part of the dentin detached from the tooth surface.

Statistical Analysis

Statistical analysis was performed using SPSS program for windows (Version 21). The data were analyzed by two-way ANOVA to evaluate the effect of adhesive type, curing protocol and their interaction on SBS. One-way ANOVA followed by Tukey's HSD post-hoc test was used to evaluate the effect of curing protocol within each adhesive type. Student t-test was performed to compare the effect of adhesive type on SBS for each curing protocol.

RESULTS

Two-way ANOVA (Table 1) showed that the curing protocol and the interaction between the two independent variables (adhesive type and curing protocol) had a significant effect on SBS (P=0.000). The adhesive type showed no effect on SBS (P=0.090).

One-way ANOVA followed by Tukey's HSD post-hoc test (Table 2) showed that Within each adhesive type, SBS values of both 1 mm and 10 mm light curing distances had no statistically significant difference in the case of FBM+DC; but were significantly higher than those of the self-cure protocol. Whereas, FBU achieved higher SBS mean values at 1 mm, while there was no statistically significant difference observed for 10 mm and self-cure protocol. Student t-test (Table 2) revealed that within each curing protocol, there was no statistically significant difference between FBM+DC and FBU at 1 mm. However, they were significantly different at 10 mm and self-cure protocol.

Table (1) *Two-Way ANOVA for the effect of adhesive type, curing protocol and their interaction on the SBS of dual-cure adhesives.*

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	515.979[a]	5	103.196	25.647	.000
Intercept	1747.441	1	1747.441	434.287	.000
Adhesive type	11.971	3	11.971	2.975	.090
Curing protocol	413.612	2	206.806	51.397	.000
Adhesive type*Curing protocol	90.396	2	45.198	11.233	.000
Error	217.280	54	4.024		
Total	2480.700	60			
Corrected Total	733.259	59			

Table (2) *Means ± Standard Deviation (MPa) for the effect of adhesive type and curing protocol on SBS of the dual-cure adhesives.*

	FBM+DC	FBU	P-value
1mm	8.3 ± 2.8[A]	8.2 ± 2.8[A]	0.944
10mm	8.1 ± 2.6[A]	3.9 ± 0.6[B]	0.001
Self-cure protocol	1.1 ± 0.5[B]	2.7 ± 0.8[B]	0.000

Means with identical uppercase letters within the same column are not statistically significant at P=0.05.

Fractographic analysis revealed that the predominant failure mode was the adhesive type, which represented 86.6%; followed by 13.3%. mixed type.

DISCUSSION

Light-cured restorative materials have increasingly revolutionized the practice of dentistry. For that reason, activation by visible light has become a crucial mode of initiating the polymerization reaction. Adequate polymerization of dental adhesives is believed to be a requisite for good clinical performance. However, for regions remote from the light-curing source, achieving proper polymerization might be questionable,[11] which would consequently affect the bonding complex.[12] Due to the widespread use of dual- or self-cure resin materials, such as resin cements or core build-up restorations; dual-curing adhesive systems have been introduced in the market. These dual-cured adhesives are composed of a mixture of monomers and catalysts that do not rely solely on photo-activation.[7]

Unfortunately, many studies have highlighted that an incompatibility does exist between light-cured one-step SEAs and dual- or self-cure resin composites.[8,9] This event has been related to the inherent hydrophilicity and acidity of such adhesives that could interfere with the polymerization reaction of these resin composites.[9] Hence, some manufacturers launched dual-cure activators containing DC catalysts, to be mixed with their respective SEAs. The main function of the DC catalyst is to react with the amines in the dual- or self-cure resin composites, so as to render them more reactive with BPO under acidic conditions.[13] Because of such chemistry, it might not be necessary, as suggested by some manufacturers, to photo-activate these adhesives prior to cementation of either indirect restorations or fiber posts with dual-cure resin cements, in the so-called “co-cure” technique. In such a case, both self-cure resin cement and dual-cure adhesive will be left to cure chemically. In order to optimize the degree of conversion of the adhesive, the light guide tip should be placed as close as possible to tooth surface.[11] In certain cases, as in class II cavities, the light-curing tip could be up to 8 mm away from the gingival floor.[6] Moreover, luting of fiber posts also dictates that both adhesive and self-cure resin cement should be allowed to cure chemically. Accordingly, three curing protocols simulating various clinical situations were tested in this study, so as to determine the bonding efficacy of chemical-cure resin composite in conjunction with two different dual-cure SEAs.

The selection of the dual-cure adhesives was mainly based on delivery form. FBM+ is a light-cured SEA supplied with an optional dual-cure activator (DCA) supplied in a separate bottle, to allow it to be used with dual- and self-cure resin composite materials. While, FBU is marketed as a single-dose dual-cure SEA (no-bottle system) and the activator is incorporated within its formulation. In account of that, it was important to examine whether or not the difference in adhesive delivery system would influence SBS.

Regarding the findings of the current study, SBS results of both FBM+DC and FBU had no significant difference when light cured at 1 mm. This could be referred to their relatively similar chemical composition. As stated by their manufacturer, the acidic monomers of both adhesives are described as organic acids that have phosphoric acids which decalcify dental hard tissues creating retentive pattern and methacrylate groups that adjoin the monomers to form a stable cross-linked network during polymerization. The acidic monomers also reduce the surface tension and the viscosity of the adhesive layer, improving its wetting property. Besides, the presence of nanoparticle fillers ensures the formation of an optimum film thickness that enables homogenous penetration of the retentive micro-pores. (FBM+ and FBU, technical product information, VOCO).

On the other side, when irradiated at 10 mm, FBM+DC yielded significantly higher SBS values

compared to FBU. According to the manufacturer's instructions, the activation of FBU was done by pressing the blister to deliver the enclosed ingredients that will be subsequently mixed with the liquid of the dispensing chamber. The pressure on the blister may not be the same each time, so this procedural variation do not guarantee total liquid flow from first to second compartment; thereby the exact percentage of the final mixed contents might differ. Also, after piercing and enlarging the foil of the dispensing chamber with the micro-brush, part of the solution might be hidden anyway underneath the punctured foil, which could restrict proper stirring of the two liquids. The rationale above is meant to explain how the dispensing form of such adhesive could possibly have interfered with the action of the DC catalyst, which could not counteract the interaction between the unreacted acidic monomers of the adhesive and the tertiary amines of the chemical-cure resin composite. Hence, acid-base incompatibility might have existed, thereby retarding the polymerization reaction of the resin composite and rendering the bonded interface less resistant to shear stresses imposed during testing. Contrastingly, the high irradiance maintained at 1 mm light-exposure distance might have helped overcome this notable shortcoming. In the case of FBM+DC, the incorporation of DC catalyst in a separate vial might have allowed correct proportioning of the mixed liquids. As consequence, the DC catalyst could have performed properly, preventing adverse reaction between the adhesive and the resin composite.

When the adhesives were left in the uncured state, the bond strengths have been considerably reduced in comparison with the light-curing protocol. It was concluded in an earlier study[13] that dual-cure SEAs should be light-cured to obtain adequate degree of conversion, as the polymerization reaction cannot depend totally on chemical activation. More specifically, FBM+DC attained lower results than FBU. The low SBS values could be ascribed to the high ethanol/water content present in both the adhesive and the DCA solutions, (Product Profile, VOCO) which might not be adequately evaporated with the recommended 5-second air-drying time. It was argued that addition of water to co-monomer/ethanol mixtures could result in retention of both water and ethanol even after evaporation, because they readily form hydrogen bonds with monomers.[14] In the absence of light, the unpolymerized acidic monomers together with the supposedly entrapped residual solvent in the uncured adhesive layer might have been brought in direct contact with the chemical-cure composite. The residual solvent could have acted as a resin plasticizer leading to composite softening,[15] which most likely had a detrimental effect on bonding assembly.

CONCLUSION

Under the limitations of this study, it could be concluded that light-curing the dual-cure self-etching adhesives before the application of the chemical-cure resin composite is an essential step to obtain better SBS results. However, the effect of increasing the light-curing distance on SBS seems to be adhesive-dependent. Since, the delivery form of the adhesive has been shown to have an influence on bond strength; it would be preferable to include the DC catalyst in a separate bottle rather than within the adhesive formulation.

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