

Does Single Versus Stepped Curing of Composite Resins Affect Their Shear Bond Strength?

(La résistance au cisaillement des résines composites varie-t-elle selon que la polymérisation se fait en une seule étape ou par couches successives?)

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S o m m a i r e

Contexte : La polymérisation provoque la contraction de toutes les résines composites et ceci a une incidence, non seulement sur les propriétés physiques des résines, mais aussi sur l'intégrité marginale de la restauration. Selon certains, la photopolymérisation par couches successives, plutôt qu'en une seule étape, réduirait au minimum cette contraction. Il semble en outre que l'épaisseur des couches de résine composite à polymériser serait un autre facteur influant sur la résistance finale au cisaillement. La présente étude compare la résistance au cisaillement lors de l'utilisation de ces 2 méthodes de polymérisation, avec des résines composites d'épaisseurs variables.

Méthodologie : Des résines composites d'épaisseurs variables (1,5, 3 et 4,5 mm), photopolymérisées en une seule étape ou par couches successives, ont été liées à la dentine de la troisième molaire de sujets humains, à l'aide de l'adhésif Scotchbond Multipurpose ou de l'adhésif Singlebond; chaque groupe expérimental était constitué de 12 spécimens. La résistance au cisaillement a été évaluée après 7 jours dans l'eau, et le mode d'échec des liaisons a été noté.

Résultats : Dans le cas des liaisons réalisées avec l'adhésif Scotchbond Multipurpose, ni l'épaisseur de la résine composite, ni la méthode de polymérisation, n'a eu d'effet significatif sur la résistance au cisaillement. La résistance a toutefois été nettement moindre lors de l'utilisation de l'adhésif Singlebond sur des résines composites de 4,5 mm d'épaisseur, la proportion des échecs des liaisons par adhésion-cohésion ayant tendance à augmenter avec l'accroissement de l'épaisseur de la résine.

Importance clinique : La photopolymérisation par couches successives ne semble procurer aucun avantage supplémentaire par rapport à la photopolymérisation en une seule étape, si ce n'est que la première méthode améliorerait, semble-t-il, l'adaptation marginale et réduirait la microinfiltration. Ces résultats laissent croire que les couches de résine à polymériser, selon l'une ou l'autre méthode, ne devraient pas dépasser 2 mm, en particulier lorsque la liaison se fait au moyen d'un adhésif monocomposant.

MeSH Key Words: composite resins; dental bonding/methods; light

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Shrinkage occurs during polymerization of all resin composites, affecting both the physical properties of the composites and the marginal integrity of the restorations in which they are used. With any photo-polymerized resin system there is a risk of insufficient conversion of the monomer into copolymer and non-uniform shrinkage.

It has been suggested that shrinkage can be minimized by allowing the resin composite to flow during curing by means of controlled polymerization.^{1,2} One method of achieving such control is to initiate polymerization of the resin composite at low light intensity and then perform the final curing at higher light intensity. This process is termed dual-cure, or stepped,

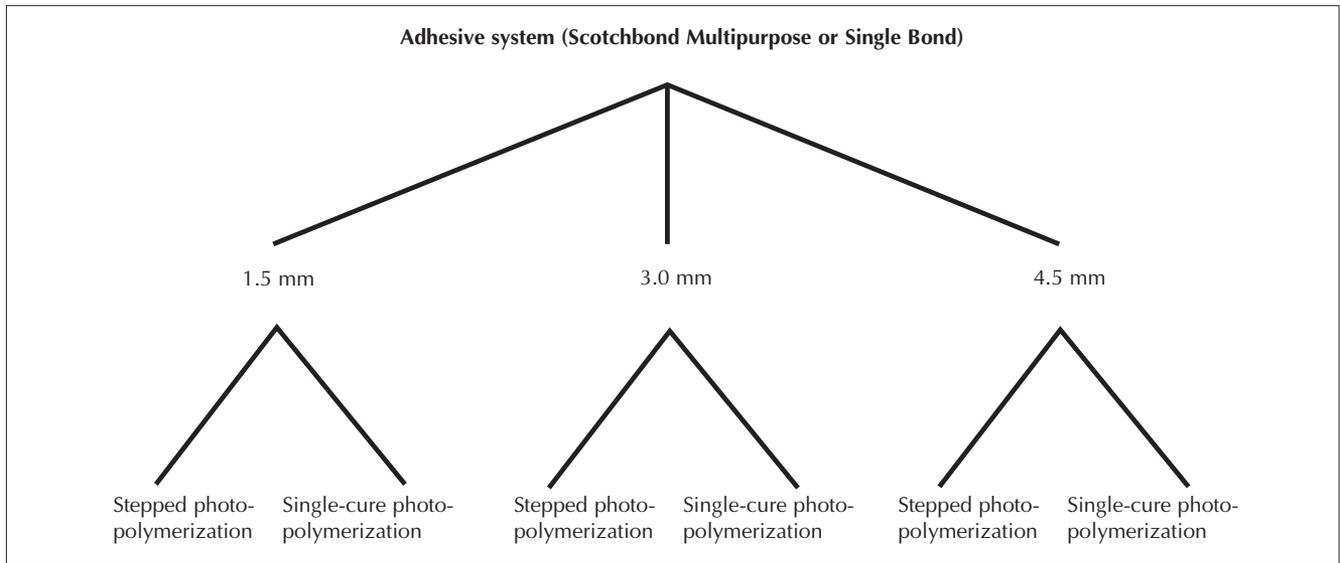


Figure 1: Flow chart showing the experimental protocol. Stepped photo-polymerization: 10 seconds at 150 mW/cm², 30 seconds at 700 mW/cm², energy output 22,500 W-s/cm². Single-cure photo-polymerization: 40 seconds at 700 mW/cm², energy output 28,000 W-s/cm².

photo-polymerization. It has been noted that the benefits of one method over the other may depend on the thickness of the resin increments.³

The aim of this *in vitro* study was to compare photo-polymerization by a single, high-intensity light source with stepped photo-polymerization in terms of the shear bond strength of the cured resin composite. Because the degree of polymerization might be affected by the thickness of the resin composite, the 2 photo-polymerization protocols were carried out with materials of various thicknesses.

Materials and Methods

Tooth Material and Preparation

Caries-free human third molars that had been frozen in water immediately after extraction were used.⁴ The teeth were thawed, half of the root of each was removed, and notches were cut at the buccal and lingual cervical margins to enhance retention in the embedding material. The teeth were then embedded in polymethyl methacrylate in moulds 2.5 cm in diameter and 2 cm deep in such a manner that the occlusal surface was parallel to the horizontal and projected above the surface of the embedding material. The embedded teeth were stored in distilled water at 4°C and were used within 48 hours after embedding. The occlusal surface was progressively ground with water-irrigated #180 and #320 grit silicon carbon (SiC) paper until the dentin just below the deepest occlusal fissure was exposed. The teeth were then kept in distilled water at room temperature. Immediately before application of the resin, the final test surface was refined with water-irrigated #600 grit SiC paper.

Resin Materials and Curing Light

Two resin adhesive systems were used in this study: the 2-bottle Scotchbond Multipurpose system and the single-bottle Singlebond system. The resin composite was Z100, shade A3.

All of these products are manufactured by 3M (St. Paul, MN). The curing light was the Elipar Highlight (ESPE, Seefeld, Oberbay, Germany), which can be programmed to produce a single high-intensity beam of light or a 2-step power output consisting of an initial low-intensity light beam followed by a high-intensity light beam for the final cure. After curing of 12 consecutive specimens was complete, the light intensity was verified by means of a Cure Rite radiometer (Caulk Dentsply, Konstanz, Germany).

Resin Templates

Biostar polyvinyl chloride (PVC) mouthguard material (Scheu-Dental, Iserlohn, Germany) was used to create templates 1.5, 3 and 4.5 mm thick. The thickest template was created by heating together sheets of 1.5- and 3-mm mouthguard material. The sheets were then cut with a guillotine into 3 cm × 3 cm pieces. A leather punch, which had been warmed in a Bunsen flame, was used to punch a hole 4 mm in diameter in the centre of each template. Each 3 and 4.5 mm thick template was then lined with a cylinder cut from a #4 gelatin capsule and trimmed so that it was flush with the edges of the template.

Resin Application

The embedded teeth were removed from the distilled water and blown dry. A strip of Teflon tape with a hole 4 mm in diameter was applied over the dentin surface of each tooth. The area of dentin defined by the hole in the tape was etched for 15 seconds with 35% phosphoric acid gel and then rinsed with water for 30 seconds. The etched area was blotted dry but left moist. The adhesive was applied in accordance with the manufacturer's instructions and cured with visible light for 10 seconds at 700 mW/cm². The PVC template was aligned with the bonded area and filled with Z100 resin composite so that it was flush with the surface; the various samples of resin composite

Table 1 Shear bond strength (mean \pm standard deviation, in megaPascals) for resin composites bonded with fourth- and fifth-generation single-bottle adhesive resins and cured by single-cure and stepped photo-polymerization

Adhesive and Curing Method	1.5-mm Resin	3-mm Resin	4.5-mm Resin
Scotchbond Multipurpose			
Single-cure	17.97 \pm 4.92	17.30 \pm 6.27	17.48 \pm 5.27
Stepped	16.02 \pm 4.92	17.91 \pm 4.74	16.20 \pm 5.18
Single Bond			
Single-cure	23.88 \pm 3.74	23.19 \pm 4.05	16.71 \pm 5.00 ^a
Stepped	22.58 \pm 5.61	21.85 \pm 3.66	16.71 \pm 5.76 ^a

^aSignificant reduction in shear bond strength ($p < 0.05$).

Table 2 Mode of fracture for resin composites of various thicknesses bonded with fourth- and fifth-generation single-bottle adhesive resins and cured by single-cure or stepped photo-polymerization, presented as number (and %) of samples*

Type of Failure	1.5-mm Resin		3-mm Resin		4.5-mm Resin	
	Single-Cure	Stepped	Single-Cure	Stepped	Single-Cure	Stepped
Scotchbond Multipurpose resin						
Adhesive-cohesive	2 (17)	2 (17)	7 (58)	8 (67)	3 (25)	4 (33)
Mixed	10 (83)	10 (83)	5 (42)	4 (33)	9 (75)	8 (67)
Single Bond resin						
Adhesive-cohesive	0	1 (8)	4 (33)	2 (17)	7 (58)	5 (42)
Mixed	12 (100)	11 (92)	8 (67)	10 (83)	5 (42)	7 (58)

*For each experimental group, $n = 12$.

were then light cured according to the protocols and light intensities shown in **Fig. 1**. For each combination of resin thickness and bond adhesive, 12 teeth were processed.

Each group of 12 teeth was stored in water at 37°C for 24 hours, by which time the gelatin lining had dissolved and the templates had become more pliable, which allowed removal of each thickness of template without stress to the resin-dentin bond. The teeth were then stored in water at 37°C for a further 6 days to ensure complete polymerization, at which time they were shear tested to failure.

Testing of Shear Bond Strength

The specimens were shear tested to failure on an Instron Universal Testing Machine, model 4301 (Instron Corporation, Canton, MA). The embedded tooth with its resin composite cylinder was clamped in a fixed base so that the cylinder projected parallel to the horizontal. A loop of prestretched stainless steel wire was placed under the cylinder at the resin-dentin interface; at a crosshead speed of 0.5 cm/min and a load cell of 50 kg, the specimens were shear tested to failure. The peak force was recorded in Newtons and converted to megaPascals.

Under field emission microscopy at $\times 30$ magnification, the mode of fracture was also recorded for each specimen. An

adhesive-cohesive fracture was recorded when the cylinder of resin composite detached with no adherent dentin, and a mixed fracture was recorded when it detached with dentin adhering to its base.

Statistical Analysis

The SAS system (SAS for Windows, version 6.12, SAS Institute, Cary, NC) was used to analyze the data, by means of one-way or 2-way analysis of variance as appropriate. Multiple pairwise comparisons were performed with Fisher's least-significant difference test, with levels adjusted according to Sidak's inequality. Significant interactions were investigated by means of the LSMEANS (least squared means) test, and statistical significance for all tests was set at the 5% level.

Results

Specimens prepared with the Scotchbond Multipurpose system had the same shear bond strength, regardless of the thickness of the resin and the method of curing (**Table 1**). The same was true for specimens prepared with the Singlebond system for resin composites of 1.5 and 3 mm thickness, but for resin composites of 4.5 mm thickness, shear bond strength was significantly lower at $p < 0.05$ (**Table 2**).

For resin composite 1.5 mm in thickness, mixed fractures

predominated in specimens prepared with both the Scotch-bond Multipurpose and the Singlebond adhesives. With increasing thickness of the resin composite, there was a tendency toward an increase in the percentage of adhesive-cohesive failures, but neither adhesive demonstrated a clear and definable pattern of fracture that correlated with increase in thickness.

Discussion

The findings of this and another study⁵ suggest that the stepped photo-polymerization system offers no advantages over a single high-intensity curing other than that the former is reported to improve marginal adaptation and hence reduce marginal leakage.¹ This conclusion held for the various thicknesses of composite resin tested. It is particularly interesting given that there is a difference of approximately 20% in energy output between the single-cure and stepped systems (Fig. 1). It appears that this small difference in energy output is not of sufficient magnitude to cause large differences in shear bond strength.

The use of a high-intensity visible curing light for polymerizing resin composite produces a greater degree of conversion, which thereby maximizes the composite's physical and mechanical properties, but this process also results in significant shrinkage.^{2,6} Conversely, a low-intensity light produces less shrinkage but results in poorer physical and mechanical properties.⁷ In other words, optimizing one side of this equation compromises the other.² It has been suggested, however, that stepped photo-polymerization is a reliable way to obtain better physical properties while reducing shrinkage. The result is an improvement in the marginal integrity of resin composite restorations at the dentin-composite junction.¹ Interestingly, though, it was reported in another study that there was no difference in volumetric shrinkage between dual-cure polymerization and single-cure polymerization with a high-intensity light.⁸

Because testing of shear bond strength continues to be a universally accepted standard test for in vitro laboratory investigations, with parameters that are clearly outlined by the International Organization for Standardization (ISO), valid comparisons can be made between the 2 curing methods.⁹ In the present study, flat, smooth dentin surfaces were used as the adhesion substrate; such surfaces have been shown to produce less stress on the resin-dentin bond than those seen in conventional cavity preparations.¹⁰ It has also been shown that as the ratio of bonded to unbonded surface area increases, the stress developed during polymerization increases, so that more stress would be generated in a cavity preparation than on a flat surface.¹¹ A recent study showed that stepped photo-polymerization of resin composite with a mean thickness of less than 2 mm had no beneficial effect on the shear bond strength to dentin.⁵

In the study reported here significantly lower shear bond strength was noted only with the 4.5 mm thick Z100 resin composite bonded with Singlebond adhesive. In a recent study comparing the shear bond strength of cylinders of resin compos-

ite 2 and 5 mm thick, significantly lower shear bond strengths were recorded with the thicker material.³

That study³ used a split ring mould, which allows visible light to penetrate from the surface of the resin, whereas the method used in the study reported here permitted light penetration from all sides. The authors believe that the clear templates used in the present study more closely mirror what takes place under clinical conditions. The results of this study indicate that 4.5 mm is, in all probability, the maximum thickness of resin composite that can be polymerized by either of the curing systems used in this study without serious compromise to shear bond strength.

Although the ISO⁹ recommends that failure modes be recorded as adhesive, cohesive or mixed in nature, it is the investigators' opinion that true adhesive failures do not occur with the all-etch technique. This is because the adhesives currently in use can penetrate some or all of the demineralized layer. In this study 2 distinct failure patterns were observed. Either there was a combination of shiny and matte areas within the circular area of the adhesive or pieces of dentin had been removed and could be seen adhering to the resin composite cylinder. The first pattern was designated as adhesive-cohesive failure and the second pattern as mixed failure. There was also a tendency for more adhesive-cohesive failures with the thicker resin composite. It should further be noted that the manufacturer recommends that Z100 resin composite be cured with visible light for 40 seconds in approximately 2 mm increments.

It has been suggested that if a good replacement for both silver amalgam and currently available resin composites is to be developed, polymerization shrinkage, the degree of polymerization, mechanical wear and chemical degradation from enzymes and other chemicals found in saliva must be meticulously researched.¹² Some additional factors that are said to influence the depth of cure and the degree of conversion of resin composites include the shade of the restorative material, the duration of the curing process and the intensity of the curing light.¹³⁻¹⁸

It is clear, therefore, that until further research is carried out, only small increments of resin composite should be cured with visible light. The results of the present study show that there is no difference in the shear bond strength of resin composite 1.5 mm or 3 mm in thickness that has been cured by stepped or single-cure photo-polymerization. This finding is in agreement with the manufacturers' suggestion that Z100 resin composite should be cured in increments of approximately 2 mm. The results further suggest that the thickness of the resin composite should not exceed 3 mm particularly when the bond is mediated by a fifth-generation single bottle adhesive. ♦

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