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Step-cure Polymerization: Effect of Initial Light Intensity on Resin/Dentin Bond Strength in Class I Cavities

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Clinical Relevance

Step-cure polymerization using a combination of low initial light intensity (10 seconds at 200-250 mW/cm²) followed by final high intensity irradiation provides an increase in resin/dentin bond strength in box-shaped cavities.

SUMMARY

This *in vitro* study assessed the effect of a step-cure light curing method on resin/dentin bond strength on the buccal wall of Class I cavities in human teeth. Occlusal enamel was removed to expose a flat dentin surface. Twenty four box-shaped cavities (C-factor = 4.5) were prepared in dentin. Prime&Bond 2.1 was applied and TPH Spectrum (Dentsply) was inserted using a bulk-filling increment. The composite was light-cured using either a step-cure photoactivation technique or a one-step continuous curing method. For step-cure polymerization, the initial cure intensity was varied by changing the distance between the light source and the resin surface. The light-cured resins were cured using four low light intensities: 150(G1), 200(G2), 250(G3) and 300(G4) mW/cm². In the continuous exposure curing method, the samples were light-activated for 40 seconds at 740 mW/cm² and irradiation was applied in a box-shaped cavity and a flat cavity (exposed buccal wall, C-factor = 0.22). Samples were prepared for TBS testing by creating bonded beams (of approximately 0.8 mm²) obtained from the buccal wall. The data were analyzed using one-way ANOVA, Tukey Test and Dunnett's Test at a significance level of 0.05. The mean TBS values for the continuous exposure group in the flat and box-shaped cavities were 24.31 and 10.23 MPa, respectively. The corresponding TBS for step-cure polymerization was 23.13 (G3), 18.83

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(G2), 14.87 (G1) and 13.26 MPa (G4). Bond strength values to the cavity wall were lower in the three-dimensional cavities and dependent on the light curing method (*p*<0.05). The use of a low initial light intensity (200-250 mW/cm²) for 10 seconds followed by high irradiation intensity provided the best bond strengths, similar to bonding in a flat cavity.

INTRODUCTION

Polymerization shrinkage is still a problem inherent to light-cured resin composites. This process creates contraction stress in the composite restoration, which can disrupt the marginal seal between the composite and the tooth structure (Wall, MacCabe & Murray, 1988). Previous reports indicate that the magnitude of stress varies according to the C-factor (Feilzer, de Gee & Davidson, 1987; Yoshikawa & others, 1999) and elasticity of the structures involved in the bonding process, such as cavity substrate (Suh, Cincione & Sandrin, 1998; Sakaguchi & Ferracane, 1998), hybrid layer (Uno & Finger, 1995) and bonding resins (Choi, Condon & Ferracane, 2000).

Polymerization contraction stress and its relieving mechanisms have become important research topics in dentistry (Davidson & Feilzer, 1997). Reduction of polymerization shrinkage stress can be obtained in several ways. Attempts have been made using incremental layering of the composites during insertion (Uno & Shimokobe, 1994; Versluis, Tantbirojn & Douglas, 1996) and through use of a low elastic modulus liner between the tooth and the restorative composite (Labella & others, 1999). A second alternative is the so-called slow-curing technique (Kemp-Scholte & Davidson, 1990; Uno & Asmussen, 1991). The most recent approach consists of an initially reduced conversion of the resin materials to allow the restoration some freedom of movement between the cavity walls and the center of contraction (Davidson & Feilzer, 1997). This step-cure polymerization involves two-step modulation of the light energy and has been shown to result in a smaller marginal gap and increased marginal integrity without compromising the physical properties and quality of the restorations (Feilzer & others, 1995; Mehl, Rickel & Kunzelmann, 1997; Koran & Kürschner, 1998). However, high light intensity is recommended, based on studies of curing depths and the physical properties of the composites. Despite several studies evaluating the stepped polymerization technique, the benefit of the slow-curing method is somewhat controversial.

To date, the majority of studies have evaluated the step-cure technique, using several initial low-intensities of light-photoactivation ranging from 20 to 400 mW/cm² (Kanca & Suh, 1999; Yoshikawa, Burrow & Tagami, 2001; Hasegawa & others, 2001; Yap, Ng & Siow, 2001; Amaral & others, 2002; Lim & others, 2002; Uno & others, 2003). Bouschlicher, Rueggeberg and Boyer (2000) found no difference in shrinkage stress or degree of conversion between the standard-cure and step-cure modes with an initial intensity of 100 mW/cm². Longer exposure durations using the same curing modes resulted in higher but equivalent degrees of conversion and stress. Furthermore, step-cure polymerization using a very low starting intensity (150 mW/cm²) did not improve the marginal adaptation of polyacid-modified resins or resin composites in Class V cavity preparations (Friedl & others, 2000). However, Bouschlicher and Rueggeberg (2000) found that ramped light intensity (150mW/cm² logarithmic increase to 800 mW/cm² over 15 seconds following by 25 seconds to 800 mW/cm²) resulted in lower shrinkage stress (equivalent degree of conversion) than standard cure mode (40 seconds to 800 mW/cm²).

There is no consensus regarding which initial low-irradiation step should be applied. This study evaluated microtensile bond strength using the step-cure light-activation technique. This study also verified the TBS developed in box-shaped cavities, since the bond strength values in flat surfaces were overestimated due to elimination of the deleterious effects of the greater cavity configuration factor (Bouillaguet & others, 2001). The hypothesis of this study is that bond strength values are improved with the step-cure curing method due to the slow development of stiffness of the composite, resulting in greater stress relief due to flow. This study determined which initial intensity of the step-cure polymerization method produces greater bond strength values compared to a typical continuous high intensity curing method.

METHODS AND MATERIALS

Twenty-four extracted erupted non-carious, non-restored, human third molars were randomly selected for this study. Pumice stone was used to clean the molars of soft tissue. The teeth were frozen immediately after extraction and stored for up to one week to maintain freshness. The occlusal enamel was trimmed at the level of the main grooves (Figure 1A) using a slow-speed diamond disc (KG 7020, Ø 22 mm, KG Sorensen, Barueri, SP-Brazil) under copious running water, exposing a flat dentin surface. The flat dentin surface was wet-ground with #600 SiC paper to remove imperfections and plane the cut surface. The roots of the prepared teeth were placed in an acrylic resin base that allowed the flat dentin surface to be oriented perpendicular to the long axis of the diamond bur (KG 3145, ISO #012, L # 8.0, batch #020116, KG Sorensen) during standardized cavity preparation. A Class I cavity was prepared in 20 randomly selected teeth measuring 4 mm (mesial-distal) x 3 mm (buccal-lingual) x 3 mm deep. These dimensions yielded a box-shaped cavity with a c-factor of 4.5 (bound surface/unbound surface area= 54 mm^2/12 mm^2= 4.5) (Figure 1C). A control group was made on a flat dentin surface of low C-factor. In the remaining four teeth, diamond burs were used to remove the vertical and floor walls of the Class I cavities to transform and isolate the buccal walls to a flat surface for bonding as shown in Figure 1D. The final finish of the buccal surface was therefore similar to the specimens with cavity preparations. In these flat buccal surface groups, the exposed peripheral dentin surface was defined, leaving a flat bonding area 3 mm long x 4 mm wide, identical to the buccal area of a box-shaped cavity. The flat buccal cavity was similar to a typical configuration for shear bond test, but with the bonding surface parallel to the dentinal tubules. The C-factor for this flat wall was 0.22 (bound surface/unbound surface area= 12 mm^2/54 mm^2= 0.22).

Standardized uniform box-shaped Class I cavities were prepared with a high-speed handpiece under copious air-water spray (Figure 1B). The burs were replaced after every five preparations. The whole displacement of the high-speed handpiece was measured with a digital micrometer (Mitutoyo Co, Tokyo, Japan) coupled to a precision cavity preparation device (Dias de Souza & others, 2001). At this point, specimens presenting any visible pulp exposures were excluded from the study. The standardization of cavity preparation was a critical factor for the execution of this study, because having cavities with identical dimensions is essential to inserting and photoactivating a standardized volume of composite in each sample.

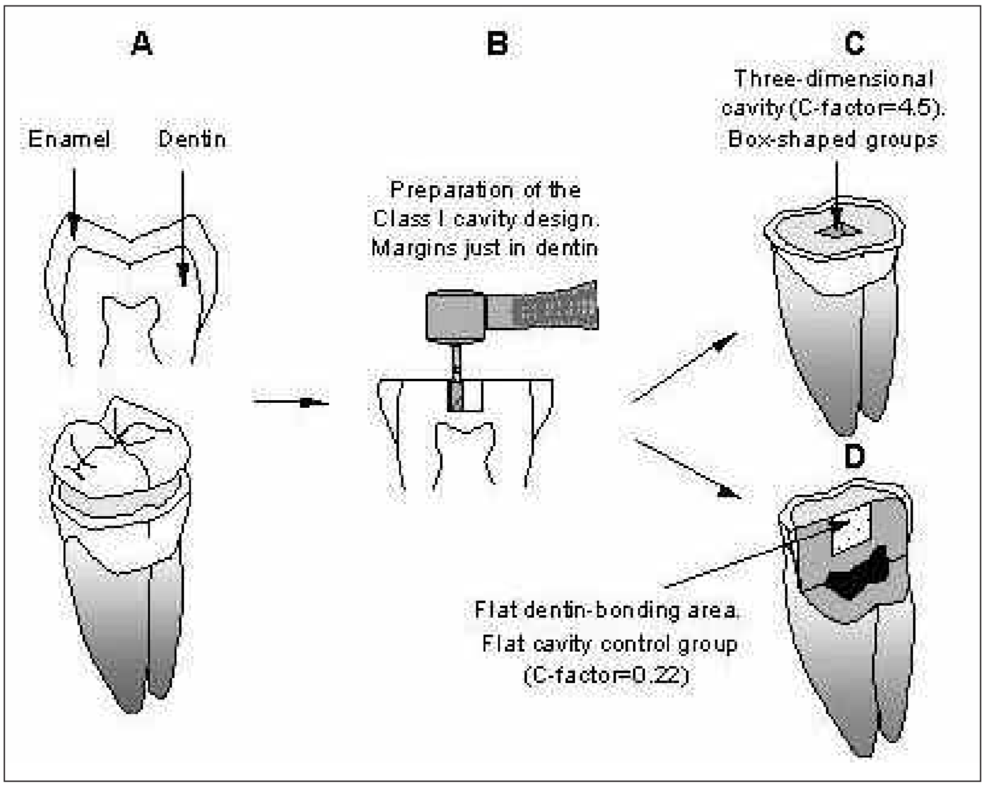

Figure 1. *Preparation of cavities. (A) extracted human tooth before preparation; (B) occlusal enamel was ground away, flat dentin exposed and the cavity was prepared; (C) cavity 3 mm deep; (D) walls of cavity were removed to create flat buccal dentin.*

Table 1: *Curing Conditions and the Energy Density Respective, Using Five Light Intensities and Various Irradiation Periods*

Curing Conditions	Energy Density
740 mW/cm^2 at 40 seconds, continuous exposure	29,600 mJ/cm^2
150 mW/cm^2 at 10 seconds + 5 seconds (interval) + 740 mW/cm^2 at 38 seconds	29,620 mJ/cm^2
200 mW/cm^2 at 10 seconds + 5 seconds (interval) + 740 mW/cm^2 at 37 seconds	29,380 mJ/cm^2
250 mW/cm^2 at 10 seconds + 5 seconds (interval) + 740 mW/cm^2 at 36 seconds	29,140 mJ/cm^2
300 mW/cm^2 at 10 seconds + 5 seconds (interval) + 740 mW/cm^2 at 35 seconds	28,900 mJ/cm^2

Immediately after cavity preparation, four molars were allocated to each of the six groups. The resin-based-composite and adhesive system used in this investigation were TPH Spectrum (shade A2, Dentsply-Brazil, Petrópolis, RJ-Brazil batch #567) and Prime&Bond 2.1 (Dentsply-Brazil, Petrópolis, RJ-Brazil batch #65713). The composite was photoactivated with one of two curing methods: step-cure and continuous exposure. In the step-cure groups, the composite was polymerized under different initial curing conditions followed by a final cure at full light intensity. The light-cured resins in box-shaped cavities were cured using five curing conditions as shown in Table 1.

The initial curing intensity was modulated by changing the distance between the light source and the resin surface. The visible light-curing unit used was KM 200R (DMC Equipment's Ltd, São Carlos, São Paulo, Brazil). For each distance, the intensity at the top of the specimens was measured with a digital Curing Radiometer of the curing unit. An intensity of 100% corresponded to 740 mW/cm^2 (with the light tip to resin distance of 0 mm) and the distances of 12.4, 11.3, 10.2 and 9.1 mm to light intensities of 150, 200, 250 and 300 mW/cm², respectively. The light tip was affixed to a vertical metallic axis, guaranteeing maintenance of a constant long-axis inclination of the light guide (at a right angle to the composite surface). The possible displacement of the light guide was monitored by a digital caliper. An initial cure of 10 seconds was carried out at one of the distances. Following a 5 second interval, the light tip was placed in contact with the composite surface and curing was completed. All curing conditions possessed equivalent light energy densities. Additionally, the first curing condition was used to light-cure the composites within a flat cavity, serving as a control group. The light intensity was checked periodically with the digital radiometer.

In all groups, the composite was inserted in one single increment. After storing the restored specimens in water at 37°C for 24 hours, the restored teeth (Figure 2A) and flat dentin-resin composite block (Figure 2B) were sectioned. Under copious amounts of water in a saw microtome (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA), a super-fine diamond disc (Extec XL-12205, Extec Corp, Enfield, CT USA, batch #453907CO) was used to serially section the specimens perpendicular to the bonded buccal surfaces, creating bar-shaped specimens (Figure 2C) with an 0.8 mm² square cross-section. Four specimens were selected from the restored teeth and mounted on a testing apparatus with cyanoacrylate adhesive (Zapit, MDS Product Co, Corona, CA, USA) attached to a universal testing machine (4411, Instron Co, Canton, USA) as shown in Figure 2D. The specimens were tensioned at a crosshead speed of 0.5 mm/minute until failure. Following completion of the bond test, the dimension of the fractured cross-section of each specimen was checked using digital calipers (Mitutoyo Co, Tokyo, Japan). The adhesive area tested was calculated and the values for bond strength were transformed into MPa.

The data were analyzed in a one-way analysis of variance (ANOVA) design. The Tukey post-hoc test was used to determine significance. Dunnett's Tests for tensile bond strength was carried out to compare the experimental groups to the control group. Statistical significance was set at $p<0.05$. All analyses were performed using SAS software for the personal computer (SAS Institute, Cary, NC, USA).

Table 2: *Tensile Bond Strength (MPa) for All Groups vs Flat Cavity Group*

Box-shaped Cavity Groups	Means (SD)	*p*
Flat cavity control group	24.31 (8.1)	
250 mW/cm²	23.13 (9.13)[a]	NS
200 mW/cm²	18.83 (7.55)[ab]	NS
150 mW/cm²	14.87 (5.29)[bc]	0.0011
300 mW/cm²	13.26 (4.62)[bc]	0.0002
Continuous exposure	10.23 (3.68)[c]	0.0000

[a]*Groups identified with different lower case letters are significantly different at* p<*0.05.*
p *values are probability values obtained in Dunnett Simultaneous Tests for comparisons with flat cavity control group.*
NS = not statistically significant.

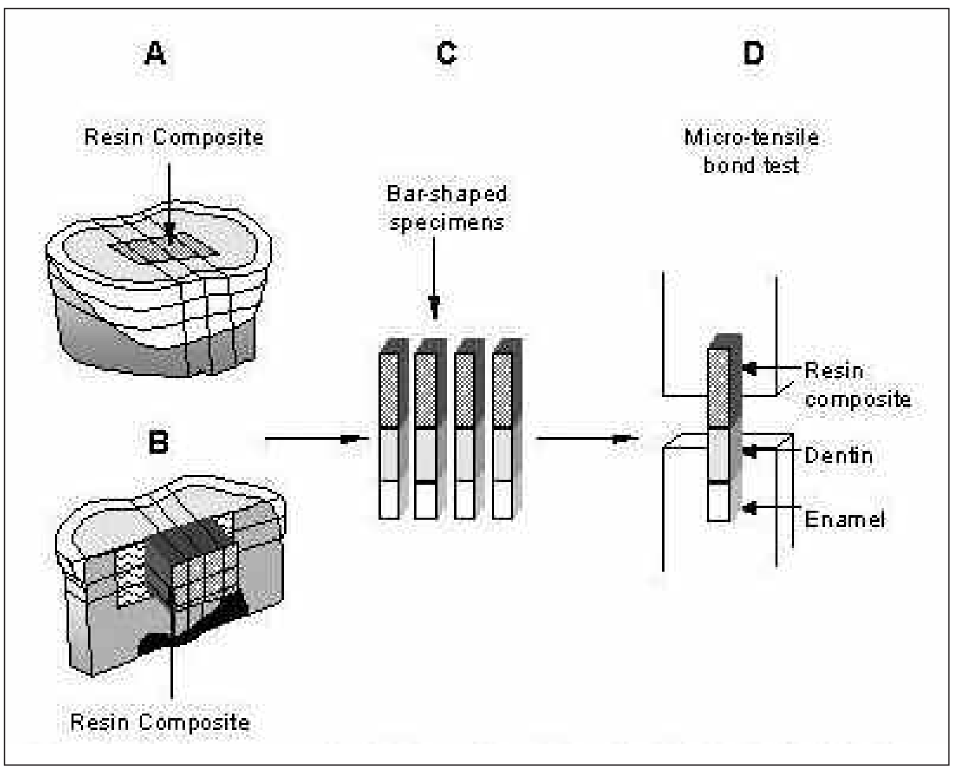

Figure 2. *Specimen preparation for tensile bond strength test: The teeth of the box-shaped cavity groups (A) and flat cavity control group (B) were restored with adhesive and composite; (C) bar-shaped specimens with 0.8 mm² cross-section area; (D) trimmed specimens were subjected to microtensile bond testing at a cross-head speed of 0.5 mm/minute.*

RESULTS

The results of the analysis of variance revealed significant differences in mean tensile bond strengths for initial light intensity ($p<0.0019$). The tensile bond strength results (TBS) are presented in Table 2. The TBS mean values varied from 10.23 MPa (continuous exposure group) to 24.31 MPa (flat cavity control group). When the tensile bond strengths of the experimental group were compared, there were statistically significant differences between the continuous exposure group and the 200 and 250 mW/cm² groups. Dunnett's test showed that the 200 and 250 mW/cm² groups presented no statistically significant difference compared to the flat cavity control group (Table 2). The bond strength means of the step-cure technique that presented initial intensities of 300 mW/cm² and 150 mW/cm² were not statistically different from those of the continuous exposure groups ($p<0.05$).

DISCUSSION

In adhesive restorations, the maturing bond strength of adhesive to dentin competes with the developing shrinkage stress of the setting material (Davidson, de Gee & Feilzer, 1984). When resin is light cured and well bonded to structures, its shrinkage produces contraction stresses at the bonding interface. This stress tends to destroy the bond between composite and tooth (Kemp-Scholte & Davidson, 1990). In high C-factor box-shaped cavities, the shrinkage forces cannot be relieved by resin flow, resulting in debonding from one or more walls (Bouillaguet & others, 2001). Bond strengths recorded in this study were more than 30 MPa, with many measurements falling below 10 MPa. The highest values were observed in the flat cavity control group, with the smallest values in the conventional exposure box-shaped cavity group. Thus, identification of the consistent effect of cavity configuration over bond strengths is established; the mean TBS of conventional exposure was reduced by nearly 60% ($p<0.0001$) when compared to that measured in the flat cavity bonding group. Yoshikawa and others (1999) also reported smaller TBS values for bulk-filled Class I cavity surfaces. However, they evaluated bonding to the pulpal floor. In the current study, the C-factor of 4.5 generated by bulk-filling of Class I cavities produced sufficient polymerization contraction stress to lower resin bonds.

A number of studies using a low initial curing light intensity have reported that step-cure polymerization did not improve marginal sealing (Friedl & others, 2000; Hasegawa & others, 2001; Amaral & others, 2002) and did not significantly reduce polymerization shrinkage (Yap & others, 2001) or shrinkage stress (Bouschlicher & others, 2000). However, other studies have suggested that the use of stepped polymerization results in better cavity wall adaptation (Yoshikawa & others, 2001; Uno & others, 2003), improved marginal adaptation (Koran & Kürschner, 1998), reduction of polymerization contraction stress in dental composites (Claus-Peter & others, 2000; Bouschlicher & Rueggeberg, 2000; Lim & others, 2002) and reduced polymerization shrinkage (Denison & others, 2000).

This study reinforces the idea that the step-cure polymerization method has beneficial effects when a cavity design with high C-factor is used. In theory, a slower rate of conversion allows for better flow by molecular rearrangement of the polymer chains, which, in turn, decreases contraction stress in the filling material (Feilzer, de Gee & Davidson, 1990; Koran & Kürschner, 1998). This effect is thought to delay the initiation of gelation of the resin and the onset of shrinkage strain (Sakaguchi & Berge, 1998). Lower irradiance results in a lower rate of stress formation (Kinomoto & others, 1999). Chemical-cure composites also have slower rates of stress formation when compared to faster polymerizing light-activated materials (Feilzer & others, 1995).

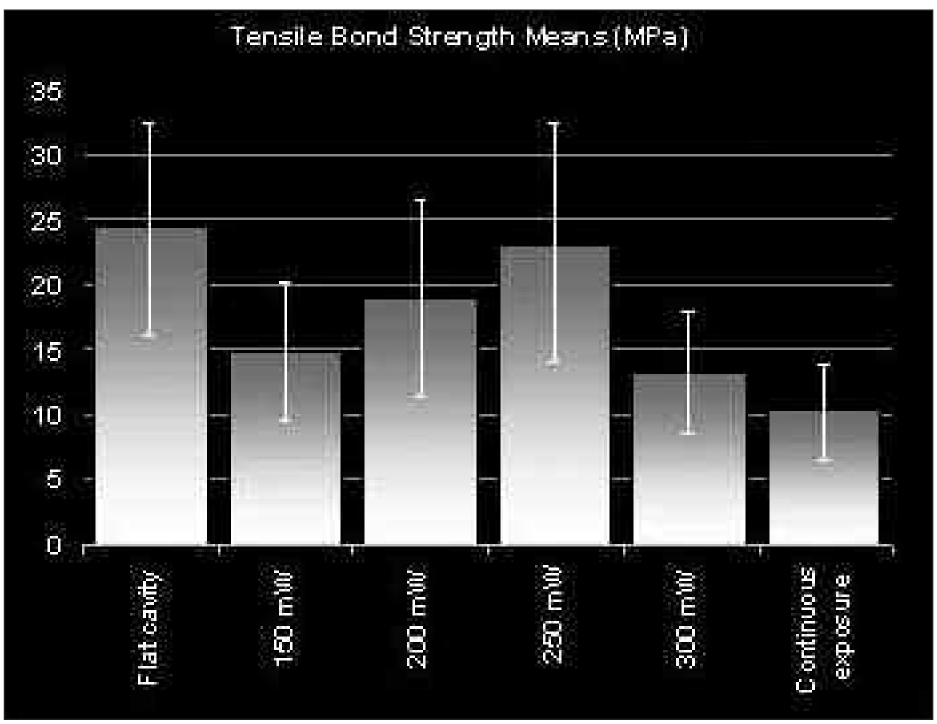

Figure 3. *Results of microtensile bond strength testing. Each column represents a mean value of the groups evaluated.*

Another explanation for stress relaxation with the step-cure photoactivation method involves the extent of cross-linking within the polymer network. Slower polymerization during the first exposure with the step-cure method may favor the formation of extended polymer chains and less cross-linking and, consequently, slow development of the elastic modulus (Kloosterboer & Lijten, 1990). Feilzer and others (1995) reported that initial low-intensity only retarded the polymerization rate at early periods but brought about the same final shrinkage as did a higher light intensity. Therefore, differences in contraction stress cannot be accounted for by differences in the extent of cure or volumetric contraction. This lends support to the hypothesis that a slower curing reaction accompanied by either a prolonged gel stage or a slow development of elastic modulus are responsible for reduced stress with step-cure polymerization (Lim & others, 2002). In this study, the step-cure methods probably produced similar overall conversion to that of the continuous exposure curing method, whereas, the light energy density was similar for all irradiation conditions.

The diversity of results in the various step-cure studies can be attributed to methodological differences, low cavity configurations and several initial intensities that were employed. The interaction between a narrow range of intensity and photoinitiator concentrations can be another explanation (Braga & Ferracane, 2002). In the box-shaped cavities used in this study, the light-cured resin composite with the very low initial light intensity of 150 mW/cm^2 and the very high initial light intensity of 300 mW/cm^2 showed the lowest TBS results for the step-cure groups and were not statistically different from the continuous exposure group. These findings agree with Friedl and others (2000) and Amaral and others (2002), who used an initial light intensity of 150 and 400 mW/cm^2, respectively. They concluded that the step-cure method used in their studies, when compared to the conventional curing method, was not able to improve marginal adaptation.

It had been previously claimed that light curing composites shrink toward the light (Hansen, 1982), because the energy at the surface nearest the light would be higher than that in the deeper parts of the composite. Thus, an energy gradient would be created within the composite, which would result in quicker polymerization closer to the light source. However, in a composite layer of 2 mm or less, the energy gradient is virtually irrelevant if sufficient light intensity was provided (Unterbrink & Muessner, 1995), as the shrinkage is directed toward the fixed boundaries (Versluis, Tantbirojn & Douglas, 1998). The use of very low initial light intensity cannot be appropriate for cavity designs, since it is inadequate to initiate the reaction at deeper subsurface levels due to light attenuation. If the light intensity is sufficiently low to create a gradient in the polymerization velocity within the bulk of the materials, the free superficial layer would cure first. The still flowing composite in deeper areas would shrink toward the bonded surfaces without any free surfaces to compensate the loss of volume by the flow of the composite. Thus, one can speculate that the step-cure polymerization method that uses a “very” low initial intensity, followed by a high intensity period to increase the degree of conversion, may generate greater shrinkage stress, and the integrity of the dentin/resin bond strength will be disrupted in deeper areas. This is one possible explanation for smaller TBS values in the 150 mW/cm^2 initial intensity group.

According to the results of this study, the 250 mW/cm^2 at 10 seconds + 740 mW/cm^2 at 36 seconds curing method showed better bond strength, similar to TBS means in flat cavities. According to Feilzer and others (1995), the main effect of stress reduction of a restorative material, Clearfil Lustre, will occur within the first 10 seconds of curing, where the light intensity was 250mW/cm^2. Yoshikawa and others (2001) showed that the best adaptation of resin composite to the cavity walls occurred with the use of a low initial light intensity of 270 mW/cm^2 for 10 seconds followed by a high intensity light of 600 mW/cm^2 for 50 seconds. Moreover, the microhardness results of their study showed more uniform polymerization of the resin composite in this curing condition. Aguiar, Ajudart and Lovadino (2002) found a lower quantitative leakage means using the step-cure polymerization method compared to a conventional light curing method when 200 mW/cm^2 was used for the initial intensity and a bulk placement technique was used. An appropriate cure of the composite cannot be achieved with intensities lower than 233 mW/cm^2 in a 1-mm thick layer (Rueggeberg & others, 1993). The intention of the initial irradiation is not to promote a thorough cure of the composite. However, the intensity must have a sufficient penetration capacity so that the initiators of deeper materials are activated, leading to a slow but homogeneous cure (Feilzer & others, 1987; Goracci, Casa de Martinis & Mori, 1996). This allows most of the polymerization contraction to occur during the initial flowable stage of material polymerization, permitting the resin to flow within itself and preventing it from pulling away from the cavity walls (Ciucchi & others, 1997).

The results of this study may not be valid for other types of composites. When *in vitro* evaluations are performed to predict the longevity of resin composite, it is important to evaluate the optimal irradiation conditions into cavity designs. Further research is required to confirm whether the step-cure polymerization method has a potential to reduce shrinkage stress in other resin composite filling materials.

CONCLUSIONS

Within the limits of this study, it can be concluded that:

1. Bond strengths to cavity walls are lower when produced in the cavity and are dependent on the light curing method.
2. Step-cure polymerization using an initial light intensity of 150 and 300 mW/cm^2 did not improve TBS relative to the continuous irradiation control group with a high C-factor.

3. The continuous exposure method used in this study resulted in the lowest microtensile bond strength.
4. Step-cure polymerization using an initial light intensity of 200 or 250 mW/cm^2 followed by full intensity provides an increase in resin/dentin bond strength in box-shaped cavities without statistical differences in the restorations bonded to a flat cavity.

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