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Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations

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ABSTRACT

Objectives. To evaluate push out force, hardness and cross-link density (CLD) of composite restorations photo-activated by different methods.

Methods. Z250 (3M ESPE) and XL2500 halogen unit (3M ESPE) were used. For push out force and hardness tests, conical restorations were made in bovine incisors. For CLD evaluation, cylindrical specimens were prepared. Different activation methods were tested: high-intensity continuous (HIC), low-intensity continuous (LIC), soft-start (SS) or pulse-delay (PD), with constant radiant exposure. Knoop readings were performed on bottom and top surfaces. Data were submitted to two-way ANOVA and Tukey's test ($\alpha = 0.05$). Push out force data were submitted to ANOVA and Tukey's test ($\alpha = 0.05$). Failure modes were classified under magnification (40×). CLD was estimated by hardness readings before and after storage in ethanol. Data were submitted to RM-ANOVA and Tukey's test ($\alpha = 0.05$).

Results. No significant differences in top hardness (KHN, N/mm^2) were observed for HIC (598), LIC (564), SS (585) and PD (573). LIC presented significantly lower bottom hardness (520) than HIC (574), SS (562) and PD (572). Push out force (N) for SS (246) and PD (238) were similar, but significantly higher compared to LIC (198) and HIC (193). For HIC and LIC, only adhesive and mixed failures were observed. For SS and PD, cohesive failures also occurred. After storage, HIC and LIC presented significantly lower softening than PD. HIC also presented lower softening than SS, and similar results were observed for SS and PD.

Significance. Different activation methods can interfere with push out force, hardness and CLD of composite restorations.

1. Introduction

Light-cured resin composites are increasingly popular in restorative dentistry due to their advantages such as esthetics, easy handling and capability to establish bond with tooth structures. However, one of the inherent shortcomings of these materials is the significant volumetric shrinkage which takes place during constrained polymerization [1]. This process strains the bond between tooth and filling, leading to the generation of stress at the bonding interface [2], and potentially causing marginal gap formation, post-operative sensitivity and pulp irritation.

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In order to attenuate the stress generation during the polymerization process, different light-activation approaches have been proposed, such as low-intensity continuous (LIC), soft-start (SS) and pulse-delay (PD). The main goal of these methods is to increase the time for the composite to flow during the earlier stages of the polymerization and to enable a certain degree of polymer chain relaxation before reaching the rubbery stage [1,3]. Indeed, previous investigators have described improved marginal adaptation and increased bond strength [3–5] in comparison to the standard high-intensity continuous (HIC) method. However, a recent study has reported that the degree of conversion (DC) for these methods might be lower when compared to the conventional method [1].

In addition to shrinkage stress, studies evaluating different curing strategies generally concentrate on the conversion of double bonds. However, the DC, although an important factor, does not give a complete characterization of the network structure, as polymers with similar DC might present distinct cross-link density (CLD) due to differences in the linearity of the chains [6,7]. The CLD plays a major role in the properties of the polymer, as highly cross-linked materials generally present increased fracture strength and wear resistance [8,9]; thus, it is a crucial factor for investigation. Therefore, the aim of this study was to investigate the influence of different light-curing methods on the push out force, hardness and CLD of resin composite restorations.

2. Materials and methods

2.1. Restorative procedures

Bovine incisors were obtained, cleaned and stored in 0.5% chloramine-T solution at 4 °C, for a week. After removal of root portions, buccal faces were wet-ground with 400-, 600- and 1200-grit SiC abrasive papers to obtain a flat surface in enamel. Standardized conical cavities (approximately 2 mm top diameter × 1.5 mm bottom diameter × 2 mm in height) were then prepared, using #3131 diamond burs (KG Sorensen, Barueri, SP, Brazil) at high-speed, under air–water cooling. A custom-made preparation device allowed standardization of the cavity dimensions. The burs were replaced after every five preparations. In order to expose the bottom surface of the cavities, the lingual faces were ground following the same procedure described for flattening the buccal aspects. Following these procedures, a cavity with a C-factor magnitude of 2.2 was obtained.

The adhesive system Single Bond 2 (3M ESPE, St. Paul, MN, USA, lot code 5EW) was then applied to the cavities, according to the manufacturer's instructions. Absorbent paper was used to remove excess dentin moisture. The specimens were placed onto a glass slab and the restorative procedures were carried out using the resin composite Filtek Z250 (3M ESPE, shade A2, lot code 5CG), which was bulk inserted into the cavity from its wider side. Different photo-activation procedures, as described in Table 1, were tested. For each method, 10 specimens were prepared.

Table 1 – Description of the photo-activation methods

Photo-activation method	Exposure protocol
High-intensity continuous	935 mW/cm^2 for 20 s
Low-intensity continuous	150 mW/cm^2 for 125 s
Soft-start	150 mW/cm^2 for 10 s + 935 mW/cm^2 for 18 s
Pulse-delay	150 mW/cm^2 for 5 s + 935 mW/cm^2 for 19 s

Prior to the curing procedures, the output power of the halogen curing unit XL2500 (3M ESPE) was measured with a calibrated power meter (Ophir Optronics, Danvers, MA, USA) and the diameter of the light guide tip with a digital caliper (Mitutoyo, Tokyo, Japan). Light irradiance (mW/cm^2) was computed as the ratio of the output power and the area of the tip. Different curing times were used in order to maintain a total radiant exposure of approximately 18.5 J/cm^2 for all samples. Irradiance at high light intensity (935 mW/cm^2) was carried out with the light guide tip positioned directly onto the restoration, which had been previously covered with a polyester strip. To produce an output of 150 mW/cm^2, a standard black acrylic cylinder separator was used to allow positioning the light guide tip 1.2 cm away from the restoration surface, and the irradiance was confirmed with the power meter. Additionally, the light spectrum profile emitted by the curing unit was analyzed with a computer-controlled spectrometer (USB 2000, Ocean Optics, Dunedin, FL, USA).

2.2. Hardness assessment

After light-curing procedures, the specimens were stored in distilled water at 37 °C, for 24 h. Thereafter, both the top and bottom surfaces were wet-polished with 1200-grit SiC paper to obtain a planar surface. Knoop hardness measurements were taken on both surfaces using an indenter (HMV-2, Shimadzu, Tokyo, Japan), under a load of 490 N (equivalent to 50 gf) for 15 s. Five readings were performed for each surface. The Knoop hardness number (KHN, N/mm^2) for each surface was recorded as the average of the five indentations. Data were submitted to two-way ANOVA (photo-activation method vs. surface) followed by Tukey's test ($\alpha = 0.05$).

2.3. Push out test

The push out test was performed in a universal testing machine (model 4411, Instron, Canton, MA, USA). An acrylic device, with a central orifice, was adapted on the base of the machine. Each specimen was positioned in the device with the top of its cavity against the acrylic surface. The bottom surface of the restoration was then loaded with a 1 mm diameter cylindrical plunger, at a cross-head speed of 0.5 mm/min until failure of the tooth-composite bonding in the lateral walls of the cavity. The plunger tip was positioned touching only the filling material, without stressing the surrounding walls. The load required for failure was recorded by the testing machine. This value was used to calculate the push out force (POF, N) for each specimen, as follows:

$$\mathrm{POF} = \left(\frac{Lg}{A_1}\right) A_2$$

where L is the load recorded by the testing machine (kgf), g the acceleration of gravity (9.8 m/s^2), A_1 the specific area of the specimen (mm^2) and A_2 is the adjusted area of the specimen (mm^2) using the theoretical dimensions of the cavity (2 mm × 1.5 mm × 2 mm). The push out force was recorded in N to avoid comparisons with traditional bond strength evaluations, such as tensile and shearing tests. Data were submitted to one-way ANOVA and Tukey's test ($\alpha = 0.05$).

After testing, the fractured specimens were examined under magnification (40×). Their modes of failure were classified as follows: adhesive failure, cohesive failure within the composite or mixed failure involving adhesive, dentin and composite. Additionally, representative fractured specimens were coated with gold and examined under SEM (JSM 5600LV, Jeol Inc., Peabody, MA, USA).

2.4. *Cross-link density evaluation*

Standardized cylindrical specimens were obtained by placing the composite into a brass mold (2 mm inner diameter × 2 mm thick). The bottom and top surfaces were covered with a transparent polyester strip and the different curing protocols were used to cure the material. For each method, 10 specimens were prepared. Samples were dry-stored for 24 h in light-proof containers, at 37 °C, and then were wet-polished with 1200-grit SiC paper, in order to obtain a smooth, planar surface.

Hardness measurements were taken on the irradiated surface, following the same procedure previously described. Readings were performed at five locations, and the average value of the five readings was recorded as the initial Knoop hardness number (KHN$_1$) for each specimen. Thereafter, the specimens were soaked in absolute ethanol for 24 h, at room temperature, and hardness was again determined (KHN$_2$). The CLD was estimated by the softening effect promoted by the ethanol, i.e., by the decrease in hardness. Data were submitted to repeated measures ANOVA, followed by Tukey's test ($\alpha = 0.05$).

3. Results

Results for the Knoop hardness assessment are summarized in Table 2. For top hardness, irrespective of the light-curing method, no significant differences were detected. On the other hand, significant differences were detected on the bottom surface for LIC compared with HIC and PD ($p < 0.05$), but not in comparison with SS.

Means for the push out force test are shown in Table 3. SS and PD displayed similar outcomes, but both presented significantly higher push out force values in comparison to HIC and LIC ($p < 0.05$). Fig. 1 depicts the percentage of failure modes for the push out test, and Fig. 2 shows SEM images of representative fractured specimens. For HIC and LIC, adhesive and mixed failures were observed in similar percentages, but cohesive failures within the composite were not detected. For SS and PD, mixed failure was the mostly observed mode, but cohesive failures were also detected.

Table 2 – Means (standard deviations) for top and bottom hardness (KHN, N/mm^2)

Photo-activation method	Top	Bottom
High-intensity continuous	598 (49) A,a	574 (32) A,a
Low-intensity continuous	564 (63) A,a	520 (32) B,b
Soft-start	585 (35) A,a	562 (33) A,ab
Pulse-delay	573 (19) A,a	572 (27) A,a

Means followed by different capital letters in the same line, and small letters in the same column, were significantly different ($p < 0.05$).

Table 3 – Means (standard deviations) for push out force

Photo-activation method	Push out force (N)
High-intensity continuous	193 (37) b
Low-intensity continuous	198 (22) b
Soft-start	246 (21) a
Pulse-delay	238 (31) a

Means followed by different letters were significantly different ($p < 0.05$).

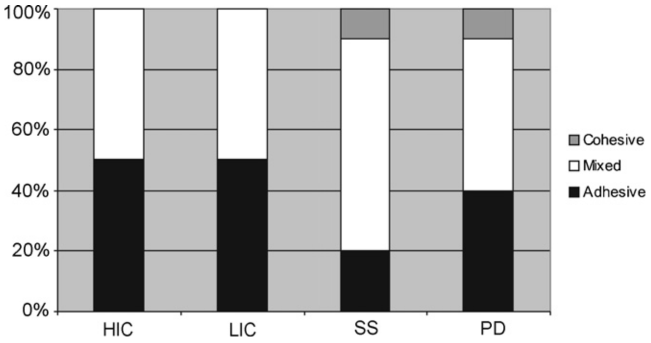

Fig. 1 – Percentage of failure modes in the push out force test.

Table 4 – Results for the cross-link density evaluation

Photo-activation method	KHN$_1$	KHN$_2$
High-intensity continuous	639 (27) A,a	426 (18) B,a
Low-intensity continuous	626 (45) A,a	423 (28) B,ab
Soft-start	606 (23) A,a	390 (19) B,bc
Pulse-delay	606 (14) A,a	359 (27) B,c

Means followed by different capital letters in the same line, and small letters in the same column, were significantly different ($p < 0.05$).

The outcomes for the CLD evaluation are displayed in Table 4. For the initial hardness testing (KHN$_1$), no significant differences were observed between all of the activation methods. After ethanol storage, both HIC and LIC presented significantly lower softening effects than PD ($p < 0.05$). However, only HIC presented significantly lower softening when compared to SS ($p < 0.05$). Similar softening outcomes were observed for SS and PD.

4. Discussion

In the present study, the push out force of a restorative composite in a high C-factor cavity was assessed, with development of the polymerization shrinkage stress directly on the

Fig. 2 – SEM images of representative fractures specimens. (A) Adhesive mode of failure: note the scratch lines on dentin, caused by bur action during the cavity preparation procedures. (B) Mixed failure involving adhesive, composite and dentin: the predominant mode of failure in the study. (C) Cohesive failure within the composite: this mode was observed only for specimens activated by soft-start and pulse-delay methods.

bonding interface [10]. The outcomes reveal that both continuous irradiation methods yielded significantly lower push out force values than SS and PD, which is in line with a previous investigation [5]. The probable explanation for this result is due to differences in the polymerization process, as modulated curing protocols have been demonstrated to decrease the rate of polymerization and, thus, the stress generation at the bonding interface [1]. These methods rely on prolonging the viscous-elastic stage of curing, allowing more time for the composite to flow before reaching the rubbery stage [2]. In fact, a previous study [4] showed that SS and PD methods were able to reduce the formation of internal gaps in composite fillings. This might also be related to the present results.

Nonetheless, according to a recent study [1], the reduced shrinkage stress observed for two-step curing approaches could be a result of lower double bond conversion than standard protocols. Therefore, in the present investigation, hardness was assessed to estimate DC. The present findings show that, for top hardness, no significant differences were observed among the curing methods. This corroborates with the assumption that similar DC can be obtained by different activation strategies, as long as the total radiant exposure is kept constant [11].

However, when assessing the bottom hardness, LIC yielded significantly lower values when compared to all of the other methods, except for SS, which is probably a result of a lower DC. The irradiance intensity is a critical factor for the in-depth cure of composites, since the incident light is attenuated with increasing distance from the irradiated surface, as a result of absorption and scattering effects [12]. Indeed, Rueggeberg [13] reported that about only 9% of the light energy hitting the top surface of the composite is available at 2 mm depth. Therefore, during continuous activation at 150 mW/cm^2, an intensity reaching the bottom layer around 15 mW/cm^2 might be expected. Also, this low light energy would be distributed along the incident electromagnetic spectrum, between 390 and 520 nm (Fig. 3). Therefore, as camphorquinone presents an optimal spectral absorbance range between 450 and 490 nm, with an absorbance peak at 468 nm [13], the energy available between 450 and 490 nm might have been insufficient to excite the photo-initiator to the same level than during activation with higher light intensity, leading to poorer polymerization. Indeed, Watts [12] stated that a minimum threshold of light irradiance reaching a specified depth is required to activate effective polymerization.

Another factor that might be considered is heat generation during the photo-activation procedures. High light intensities result in a high temperature increase within the composite [1,14], which can account for greater double bond conversion, even at the bottom layer, due to increased monomer mobility in the environment and also increased reaction rate parameters [14]. Additionally, the light guide tip was positioned distant from the cavity surface for producing 150 mW/cm^2, in order to approximate the clinical situation. This could also be related to less heat generation. Moreover, the lower bottom hardness for LIC could explain the lower push out forces observed for this method, as the DC is directly related to the bond strength between composite and adhesive [15].

Besides stress generation, the rate of cure can also interfere with properties of the final composite, like CLD. Therefore, the ethanol softening analysis was carried out to estimate the degree of cross-linking promoted by the different irradiance methods. The results demonstrated that SS and PD presented greater softening than HIC. This is probably explained by the fact that a slow polymerization start is generally

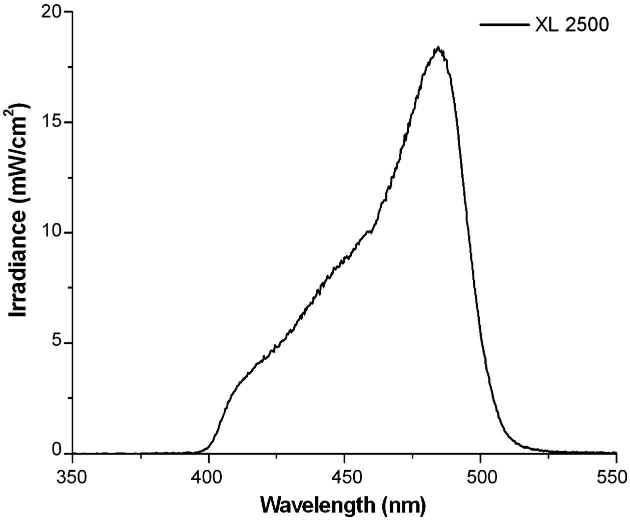

Fig. 3 – Light spectrum profile emitted by the halogen curing unit. An emission range between 390 and 520 nm, with a peak at 485 nm, is observed.

associated with relatively few centers of polymer growth, possibly resulting in a more linear final polymer structure. This is in opposition to irradiance at high intensity which generally initiates a multitude of growth centers and favors the formation of a polymer network with more cross-links [6,7]. Since solvent uptake and swelling are directly related to CLD, a polymer with fewer cross-links is more sensitive to the plasticizing action of solvents [8].

The outcomes of the failure analysis corroborate with the results observed in the push out, hardness and softening evaluations. For both continuous methods, half of the number of specimens showed adhesive failures. For HIC, this finding is probably related to a generation of high stress, leading to disruption of bonding to the cavity walls [8]. Conversely, although activation at a low intensity probably decreased the resulting shrinkage stress for LIC, the lower degree of conversion at the bottom layer, as observed in the hardness assessment, probably interfered with bonding between the adhesive and the restorative composite [15]. In this particular case, Tay et al. [16] demonstrated that, for conical-shaped cavities, different generations of shrinkage stress might occur due to different C-factor conditions in the same cavity. Therefore, dividing the cavity of the current study into two sections (top and bottom), the bottom area has a higher C-factor. The higher C-factor could lead to a generation of higher stress in this area and this, in association to the poor bonding between adhesive and composite, could have triggered an initial debonding. On the other hand, few adhesive failures were observed for SS and PD, probably due to the lower aforementioned stress generation. However, cohesive failures within the composite were observed only for these methods, which could be related to lower strength of the polymer as a function of lower CLD. Indeed, the softening effect observed for these methods was higher when compared to both continuous methods.

In summary, the present findings show that both two-step curing approaches, SS and PD, yielded significantly higher push out forces as compared to the continuous modes, probably due to lower stress generation at the tooth–filling interface. In the clinical situation, this could result in improved marginal and internal adaptation of restorations [4,5]. However, the present study raises a question about the polymer structures resulting from the two-step methods of activation. An increased susceptibility to softening was observed for the two-step methods, which could pose a risk to increased wear and degradation in the oral environment. Polymers with a high CLD may be advantageous not only because they may present enhanced mechanical properties, but also by being less susceptible to the softening of food substances and to enzymatic attack [6].

5. Conclusion

Different photo-activation methods can interfere with push out force, hardness and cross-link density of restorations. Pulse-delay and soft-start methods yielded higher push out force to the cavity walls, but with increased susceptibility to softening.

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