

Effect of Different Photoactivation Techniques on the Bond Strength of a Dental Composite

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Using the push-out test, this study evaluated the bond strength of the composite resin Z250 (3M/ESPE) photoactivated with the XL2500 (3M/ESPE) quartz-tungsten-halogen light-curing unit with different curing protocols: soft-start (150 mW/cm² for 2 s (S2), 3 s (S3), 5 s (S5), 10 s (S10) or 15 s (S15), followed by 700 mW/cm² for 15 s; pulse-delay (150 mW/cm² for 2 s (P2), 3 s (P3), 5 s (P5), 10 s (P10) or 15 s (P15), with a 1-min delay, followed by 700 mW/cm² for 15 s. After storage at 37°C ± 1 for 24 h ± 1, the specimens were ground, polished and subjected to a push-out test in a universal test machine (Instron) with a cell load of 500 N at a crosshead speed of 0.5 mm/min. The data were analyzed statistically by ANOVA and Tukey's test at 5% significance level. There were no statistically significant differences (p>0.05) among the groups photoactivated using the soft-start mode. For the pulse-delay mode, P5 promoted the highest bond strength (p<0.05). Groups photoactivated with the pulse-delay mode (except for P2 and P15) presented significantly higher bond strength than those photoactivated with the soft-start. It may be concluded that the influence of initial exposure time was curing method-dependent, with the best results obtained using the pulse-delay method with 5 s in the first photoactivation cycle.

Key Words: pulse-delay, soft-start, bond strength.

INTRODUCTION

The composite resins are the most used direct restorative materials in dentistry (1). However, the primary problem of dental composites is still the polymerization shrinkage. Shrinkage stress at the adhesive interface tends to destroy the bond between the composite resin and the tooth structure (2). The magnitude of shrinkage stress is due to polymerization reaction speed (3). The operator can minimize shrinkage stress and gap formation at interface during photoactivation using different photoactivation methods (4) or incremental techniques (5). To decrease the speed

of the polymerization reaction, and consequently the shrinkage stress, techniques that suggest low irradiance during the initial period of photoactivation have been proposed (3-6). Soft-start polymerization technique adopts an initially low light intensity followed by a final cure with high light intensity. A slower rate of monomer-to-polymer conversion allows for better flow of the material, which, in turn, decreases contraction stresses, leading to better marginal adaptation (6,7). Complementing photoactivation with high irradiance allows for composites achieving the maximum degree of conversion (6). In the pulse-delay photoactivation technique, there is a short period (about 1-3 min)

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between initial exposition and final cure. This procedure can reduce the incidence of cavo-surface marginal gap and enamel fracture (8). Another advantage of these photoactivation methods is that the degree of cure is similar to conventional method (9).

However, there are still doubts as to the best photoactivation method and light exposure time to promote adequate bond strength between composite resins and dental structure. The aim of this study was to evaluate the bond strength of a composite resin photoactivated using soft-start and pulse-delay modes, with different initial light exposure times during the first photoactivation cycle.

MATERIAL AND METHODS

One hundred 3-mm-thick round metallic molds with a central cavity with 6 mm of lower diameter and 9 mm of the upper diameter were used (Fig. 1).

The molds were internally sandblasted with 40- μ m particle aluminum oxide using a micro-etcher (Danville Eng. Inc., San Ramon, CA, USA), and then etched with 37% phosphoric acid for 15 s, water rinsed and dried. A bonding agent silane (Ceramic Primer; 3M ESPE, St. Paul, MN, USA) was applied and air dried for 5 s. The adhesive system (Single Bond 2, 3M ESPE) was applied, air-thinned for 20 s and photoactivated for 10 s.

A Mylar strip was positioned on a glass slab. The composite resin Z250 (3M ESPE; shade A3) was

bulk inserted into the mold and another Mylar strip was seated onto the composite surface, and pressed manually using a microscope slide to remove composite excesses.

The composite resin was photoactivated with a quartz-tungsten-halogen light-curing unit (XL2500; 3M ESPE) using soft-start and pulse-delay photoactivation methods (Table 1). The tip of the light guide of the curing units was moved away from composite resin surface to reduce irradiances, and acrylic resin (JET; Artigos Odontológicos Clássico, São Paulo, SP, Brazil) spacers were interposed between the composite surface and the tip of the curing units to standardize the photoactivation distance.

After photoactivation, the specimens were stored

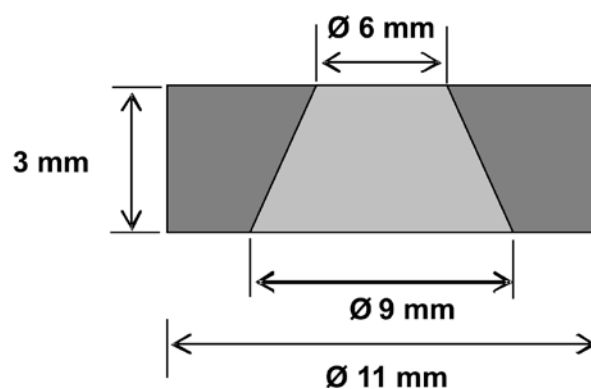


Figure 1. Schematic illustration of the metallic mold.

Table 1. Photoactivation methods.

Method	Group	Time (s)	Irradiance (mW/cm ²)	Delay (min)	Light exposure (s)	Irradiance (mW/cm ²)	Energy density Total (J/cm ²)
Pulse-delay	P2	2	150	2	15	700	10.80
	P3	3	150	2	15	700	10.95
	P5	5	150	2	15	700	11.25
	P10	10	150	2	15	700	12.00
	P15	15	150	2	15	700	12.75
Soft-start	S2	2	150	---	15	700	10.80
	S3	3	150	---	15	700	10.95
	S5	5	150	---	15	700	11.25
	S10	10	150	---	15	700	12.00
	S15	15	150	---	15	700	12.75

in incubator at $37^{\circ}\text{C} \pm 1$ for $24 \text{ h} \pm 1$, in a dark and dry container, before the push-out test. The top and bottom surfaces of restorations were ground using 400 grit sandpapers (Carborundum; Saint-Gobain Abrasivos Ltda, Cruz de Rebouças, Igarapu, PE, Brazil) on an automated polisher under water cooling to remove the composite resin excess, promoting a correct positioning of specimens while testing.

For the push-out test an acrylic resin apparatus containing a central hole was attached in a universal testing machine (Instron Corp; Model 4411, Canton, MA, USA). A sphere-shaped rod attached to a compression load cell (500 N) was used to load the specimens until failure at a crosshead speed of 0.5 mm/min (Fig. 2). The push-out bond strength was determined by computing the quotient of maximum load (N) and adhesion area (equation for calculation of truncated cones, with values expressed in mm^2). The data were analyzed statistically significant by two-way ANOVA (*photoactivation mode* and *exposure time*) followed by Tukey's test at the 5% significance level.

RESULTS

Table 2 displays the mean and standard deviations for push-out bond strength. For pulse-delay photoactivation method group, P5 presented significantly higher bond strength than the other groups ($p < 0.05$). Group P2 presented the lowest push-out bond strength, differing significantly ($p < 0.05$) from P5 and

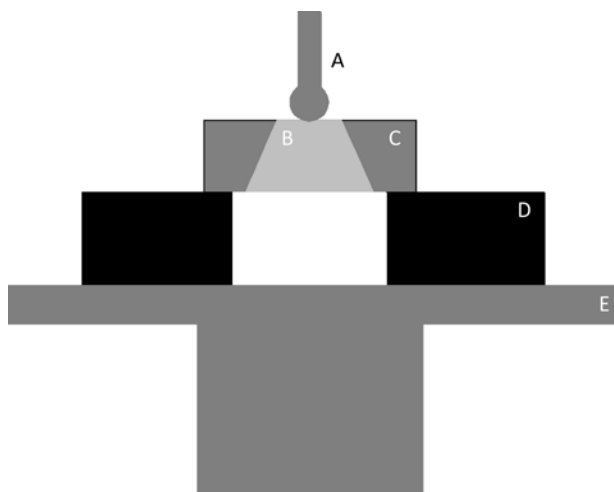


Figure 2. Schematic illustration of the metallic mold for push-out test. A: Sphere-shaped rod; B: Specimen; C: Metallic mold; D: Acrylic resin apparatus; E: Universal testing machine.

P10 groups. No statistically significant differences ($p > 0.05$) were found among groups photoactivated using soft-start methods. Comparison between photoactivation methods showed that P3, P5, and P10 presented significantly higher bond strength ($p < 0.05$) than S3, S5, and S10, respectively. However, there were no significant differences between P2 and P15 compared to S2 and S15, respectively ($p > 0.05$).

DISCUSSION

During photoactivation, free radicals are generated to initiate the polymerization reaction of the composite resin. Both free radical generation and polymerization rate are influenced by the irradiance emitted from the light source (10). Photoactivation techniques that supply low irradiance during the initial period of photoactivation have been proposed to decrease polymerization rate and consequently the shrinkage stress (6). Thus, soft-start and pulse-delay light modulation techniques were evaluated in this study.

The results showed that pulse-delay methods yielded significantly higher bond strength than soft-start mode. Both soft-start and pulse-delay method adopt an initial low irradiance followed by a final cure with high irradiance. However, the pulse-delay method adopt after the first polymerization cycle a period in "dark" (1 min) before the second polymerization cycle. Pfeifer et al. (11) found a significant decrease on shrinkage stress (19%) after 1 min delay, and attributed this results to either a lower degree of conversion or to a lower elastic modulus during the initial periods of polymerization. The reduction on the polymerization rate, and consequently in the stress rate could be related to improved marginal

Table 2. Push-out bond strength (MPa) means and standard deviations of Filtek Z250 composite resin photoactivated using different methods and exposure times.

	Pulse-delay (P)		Soft-start (S)
P2	5.40 (1.89) c,A	S2	4.16 (0.78) a,A
P3	6.38 (1.16) bc,A	S3	4.21 (1.16) a,B
P5	34.20 (8.47) a,A	S5	5.24 (0.95) a,B
P10	7.80 (2.33) b,A	S10	4.06 (0.63) a,B
P15	6.12 (1.73) bc,A	S15	5.26 (1.19) a,A

Different lowercase letters in columns and uppercase letters in rows indicated statistically significant difference at 5%.

adaptation and bond strength. Cunha et al. (12) reported that the initial pulse limited the contraction force produced when the second, higher irradiance exposure was applied. Therefore, the maximum stress rate generated when stepped methods are used to photoactivate composite resin is reduced. This could lead to less gap formation and improved bond strength.

In the delay period, little amount of free radicals and double bonds conversion are produced and the composite resin has more time to molecular rearrangement and stress relief. The lower initial conversion may produce polymeric chains with greater mobility allowing more dark-cure and stress relief (13). Lim et al. (14) showed that a constant stress (1 MPa) was exerted by composites during the pause in the two-step curing routine. However, the slow rate of polymerization during the delay period may prolong the low modulus phase, without significant reduction in the degree of conversion (14).

For the soft-start method the activation of some free radicals during the first photoactivation cycle occurs and initiates the polymerization reaction. During the first polymerization cycle, the degree of conversion reaches about 50% of that obtained after the second light exposure. Immediately after the first polymerization cycle the specimens are photoactivated using the maximum irradiance emitted from the light source. The high irradiance after the first cycle does not allow that the stress be relieved internally (15). Thus, the stress produced by fast polymerization reaction in the second polymerization cycle added to stresses produced in the first cycle are directed to bond interface, leading to gap formation and reducing the bond strength as compared to pulse-delay method (16).

When soft-start was used, the initial light exposure time did not influence the bond strength. However, specimens photoactivated with the pulse-delay method with short (2 and 3 s) and long (10 and 15 s) initial light-exposure times showed significantly lower bond strength than those photoactivated for 5 s. These findings are in accordance with those of previous studies that used the pulse-delay method with different light exposure time to photoactivate composite resin (17). During the initial stages of polymerization reaction, contraction may be compensated for by flow within the material and contraction strain will produce contraction stress only after the development of a certain modulus of elasticity (about 50%) (14). As the polymerization in the second cycle occurs at high speed, there is more shrinkage stress,

leading to lower bond strength values (18).

The degree of conversion of composite resins depends on the energy density supplied during the photoactivation (19). P2 and P3 groups used 0.3 and 0.45 J/cm², respectively, in the first polymerization cycle. This amount of energy may have been insufficient to generate adequate amount of free radical and to polymerize adequately the composite resin during the first cycle. Thus, most of polymerization may have occurred in the second cycle that uses high irradiance, concentrating more stress at the bonding interface. The low energy density during the first cycle of polymerization may also explain the results found when comparing P2 and S2 groups, which showed no difference between them.

However, the groups that used longer initial light exposure times during the first photoactivation cycle (10 and 15 s) also presented lower bond strength than P5 group. When composite resin is photoactivated with low irradiance a more linear polymeric chain can be formed. Materials with linear polymer chains are more susceptible to the effects of plasticizers, reducing the resistance values. Moreover, P10 and P15 were photoactivated with the highest energy densities (12 and 12.75 J/cm², respectively). Previous studies reported that the higher the density of energy supplied during light curing, the greater the degree of conversion and polymerization shrinkage (20). Therefore, more stress can be directed to the bonding interface, leading to a reduction on the bond strength. Besides, the increase of photoactivation time (P15 and S15) leads to a rapid increase on elastic modulus, reducing the composite resin flow. Therefore, stresses cannot be released into the material, leading to a possible rupture of the resin-dentin bond, even if a delay period is used after the first curing cycle. Prolonged curing time can reduce the bond strength of restorations, even when modulated photoactivation methods are used.

The results showed that great differences between P5 and the others groups were found. These results could be associated to absence or reduction on gaps formation between the composite resin and the metallic mold. Gaps are sites of stress concentration and are associated with an accentuated decrease in bond strength (P2, P3, P10, and P15 groups). However, the absence or reduction of the gaps allows tension to be distributed across the bond interface, increasing the bond strength (P5 group).

Within the limitations of this study, it may be concluded that the exposure time during the first photoactivation cycle affected the bond strength of

the pulse-delay method, with the highest results for P5 (5 s light exposure time). However, soft-start was not influenced by the exposure time variations. The pulse-delay method was proven more effective than the soft-start method.

RESUMO

O objetivo deste estudo foi avaliar a resistência de união (push-out) da resina composta Filtek Z250 (3M/ESPE), fotoativada com luz de lâmpada halógena XL2500 (3M/ESPE), utilizando diferentes protocolos de fotoativação: soft-start (150 mW/cm² por 2 (S2), 3 (S3), 5 (S5), 10 (S10) ou 15 s (S15), seguido por 700 mW/cm² por 15 s; e pulse-delay (150 mW/cm² por 2 (P2), 3 (P3), 5 (P5), 10 (P10) ou 15 s (P15), com 1 min de espera, seguido de 700 mW/cm² por 15 s. As amostras foram armazenadas a 37°C ± 1 por 24 h ± 1, lixadas, polidas e submetidas ao ensaio de resistência de união (push-out) em máquina de ensaio universal (Instron) com velocidade de 0,5 mm/min. Os dados foram submetidos à Análise de Variância e teste de Tukey (5%). Os resultados mostraram que não houve diferença significativa entre os grupos fotoativados pelo método soft-start (p>0,05). Para o método pulse-delay, o grupo P5 apresentou a maior resistência de união, significativamente superior aos outros grupos. Os grupos fotoativados pelo método pulse-delay (exceto P2 e P15) apresentaram resistência de união significativamente superior ao método soft-start. Pode-se concluir que a influência do tempo de exposição inicial foi método-dependente, sendo os melhores resultados apresentados pelo método pulse-delay fotoativado com exposição inicial de 5 s.

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