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ORIGINAL ARTICLE

Thermo-mechanical degradation of composite restoration photoactivated by modulated methods—a SEM study of marginal and internal gap formation

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Abstract

Objective. To evaluate the influence of thermal-mechanical degradation on superficial and internal gap formation of composite restorations photoactivated using modulated methods. ***Materials and methods.*** An experimental composite was prepared using a resin matrix containing 65wt% Bis-GMA and 35wt% TEGDMA. Camphorquinone (0.5wt%) and dimethylaminoethyl-methacrylate (0.5wt%) were dissolved in the resin as a photo-initiator system and 65wt.% silanized glass fillers were added to the matrix. Ground buccal surfaces of bovine lower incisors were used to make 160 preparations (3 mm × 3 mm × 2 mm in depth). An adhesive system (Adper Single Bond 2) was applied and the specimens were assigned into 16 groups ($n = 10$), according to the photoactivation method [high intensity (HI), low intensity (LI), soft-start (SS) and pulse-delay (PD)] and the degradation protocol [(control/no degradation; thermal cycling (TC); mechanical loading (ML); thermo-mechanical loading (TC+ML)]. Marginal and internal interfaces of bonded restorations were replicated in epoxy resin and analyzed by SEM. Gaps were expressed as a percentage of the total length of the margins. Data were submitted to 2-way ANOVA and Tukey's test ($\alpha = 0.05$). ***Results.*** For the control group no significance was noted among the photoactivation methods. TC had no effect in gap formation. ML and TC+ML increased the incidence of superficial gaps for both HI and SS groups as well as increased the internal gaps for all groups. ***Conclusion.*** Although photoactivation methods do not influence gap formation at first, composite restoration photoactivated by low intensity or modulated methods showed improved resistance to thermo-mechanical degradation. Mechanical loading is determinant for interfacial degradation of composite restorations, while thermal cycling has no effect on gap formation.

Key Words: *gap formation, mechanical loading, modulated photoactivation, thermal cycling*

Introduction

Although composite restorations have become popular because of their esthetic characteristics, some drawbacks inherent to the polymerization reaction still need to be overcome. As the material polymerizes, an increase in the stiffness accompanied by volumetric changes results in stress at the preparation walls that challenges the integrity of the bonding between the composite restoration and the tooth tissues. The adaptation of the restorative material to the preparation walls is crucial for the long-term performance of all bonded restoration [1]. Therefore, restorative techniques that allow improved marginal sealing associated with an adequate degree of conversion and mechanical properties are of special interest [2]. In this way, modulated photoactivation methods, such as soft-start and pulse delay, have been proposed [3,4].

The idea behind the use of modulated photoactivation methods is to modify the polymerization kinetics by modulating the power density during the

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photoactivation of composites [5,6]. The soft-start method employs an initial light activation at lower power density (usually from 80–150 mW/cm^2) followed by a final period at higher power density (usually from 400–1000 mW/cm^2). The final light exposure is claimed to provide enough energy density to the material, thus reaching an adequate degree of conversion [3,7].

The pulse delay photoactivation method is similar to the soft-start technique, since it also employs an initial light exposure at low power density followed by a final exposure at a higher power density. The major difference is the lag period or a hiatus that is proposed in the pulse delay technique [4]. The initial pulse with a low energy density starts the polymerization reaction. Also, in the lag period, without light exposure, the polymerization process occurs slowly. At the final stage, a longer exposure (10–20 s) at higher intensity would guarantee adequate mechanical properties [4]. Although improved mechanical properties have been reported, a difference in the polymer quality has been pointed out [8,9]. It has been claimed that composites photoactivated using the pulse delay technique exhibit a higher susceptibility to softening in 100% ethanol instead of achieving a comparable degree of conversion when compared to that of the standard continuous technique [8,9]. In this way, it has been hypothesized that PD technique results in a polymer structure with lower cross-linking density and that slow start polymerization techniques are associated with relatively few centers of polymer growth which may favor the formation of a more linear polymer structure [10]. On the other hand, it has also been reported that the modulated photoactivation methods can decrease the intensity of shrinkage stress [11], increase the bond strength of restorations [7,11,12] and also decrease the internal gap formation of composite restorations [3,13]. Nevertheless, the majority of studies claim advantages only when immediate results were evaluated.

However, even if the marginal integrity could be well established during and immediately after the restorative procedure, marginal degradation/gap formation may occur with time due to the chemical, thermal and mechanical stress that occur at the adhesive interface [14–17]. Mechanical loading also causes deformations at the bonded interface leading to microcracks or plastic deformation, resulting in temporary or permanent gaps [14,18]. Also, thermal variations in the oral cavity induce mechanical stresses due to differential thermal changes in dental structure/composite, which induce crack propagation throughout the bonded interfaces [14,15,17,19].

Considering that the influence of modulated photoactivation methods to the long-term maintenance of interfaces of bonded restorations are not conclusive and also the importance of the thermal and the mechanical loading in the longevity of composite restorations, hypotheses were delineated to evaluate these factors. Hence, the aim of this study was to evaluate the effect of thermal-mechanical degradation in the marginal and internal adaptation of composite restorations photoactivated by modulated methods. The results were compared to that of obtained using a continuous photoactivation method. The research hypotheses tested were: (1) the thermal stresses will negatively affect the marginal and internal adaptation of restorations; (2) the mechanical stresses will negatively affect the marginal and internal adaptation of restorations; (3) the synergic effects of both thermal and mechanical stresses will cause an increase in the interfacial degradation of restorations; (4) restorations photoactivated using modulated methods will exhibit improved marginal and internal adaptation compared to that with a conventional (continuous exposure) method; and (5) restorations photoactivated using modulated methods will resist more the thermal-mechanical degradation than restorations photocured using the conventional continuous light activation method.

Materials and methods

Experimental design

In this *in vitro* study, marginal and internal adaptation of restorations of an experimental dimethacrylate-based resin composite were evaluated according to the factors: (1) Photoactivation method at four levels: High intensity, Low intensity, Soft Start and Pulse Delay; and (2) Degradation protocol at four levels: Control–no degradation, Thermal Cycling, Mechanical Loading and Thermo-Mechanical Loading. Sixteen groups (n = 10) were obtained by the product among factors under study.

Preparation of the experimental composite

A monomer blend containing 65wt% of 2,2-bis [4-(2-hydroxy-3-methacrylyloxypropoxy)phenyl]propane (BisGMA, Sigma-Aldrich Inc., St Louis, MO) and 35wt% of triethylene glycol dimethacrylate (TEGDMA, Sigma-Aldrich Inc.) was prepared. Then, 0.5wt% camphorquinone (CQ, Sigma-Aldrich Inc.) and 0.5wt% *N,N'*-dimethyl aminoethyl methacrylate (DMAEMA, Sigma-Aldrich Inc.) that characterizes the light-curing initiator system, were also dissolved in the matrix. Also, the inhibitor BHT (butylated hydroxytoluene, Sigma-Aldrich Inc.) was added to the organic matrix in a concentration of 0.1wt% in order to avoid spontaneous polymerization of the monomers. The organic matrix was reinforced with silanized barium aluminum silicate glass fillers (BaAlSi, average size: 0.5 μm) and silica (SiO_2, average size: 0.04 μm). The fillers were incrementally added and homogeneously mixed to a 65wt%

Table I. Photoactivation methods used in this study.

Method*	Light exposure protocol	Energy dose
High intensity (HI)	40 s at 700 mW/cm^2	28 J/cm^2
Low intensity[†] (LI)	187 s at 150 mW/cm^2	28 J/cm^2
Soft-Start[†] (SS)	10 s at 150 mW/cm^2 + 38 s at 700 mW/cm^2	28 J/cm^2
Pulse Delay[†] (PD)	10 s at 150 mW/cm^2 + 3 min light off + 38 s at 700 mW/cm^2	28 J/cm^2

*The halogen lamp XL 2500 (3M ESPE, St Paul, MN) was used to photo-activate specimens.

[†] Light intensity reduction was obtained by increasing distance between light tip and composite top surface through a standard spacer.

loading. Considering this filler content, 80wt% were BaAlSi and 20wt% were SiO_2. The manipulation of the experimental composite was carried out under filtered orange light.

Samples preparation

One hundred and sixty lower bovine incisors, free from cracks or any other kind of structural defects, were selected under ×20 magnification. The teeth were disinfected in a 0.5% chloramine solution for 15 days and frozen stored for no longer than 1 month in a 0.9% saline solution. The crowns were cut off at the cement–enamel junction using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil). Buccal surfaces were ground and flattened under water cooling using #180 grit SIC paper (Saint-Gobain Abrasives, Igaraçu, PE, Brazil), in order to expose a flat enamel surface at least 6 mm in diameter. The crowns were sectioned in fragments of 6 mm × 8 mm using a double-faced diamond disk (KG Sorensen) in order to allow the fixation of the specimen in the standard preparation device. The thickness of enamel after being ground was ~ 0.5 mm.

Preparations were made in the ground bucal surfaces of each tooth with a diamond bur (ref. 2143, KG Sorensen) with a high speed water-cooled handpiece (Kavo SA, Joinville, SC, Brazil) using a standard preparation device. The diamond bur was replaced after every five preparations. The preparation presented a square form (3 mm × 3 mm × 2 mm in depth). In this geometry, the cavity volume was 18 mm^3 and the C-factor was 3.6.

After preparation, the specimens were randomly assigned into 16 groups, according to the photoactivation method (Table I) and the degradation protocol (Table II). Adhesive system Adper Single Bond 2 (3M ESPE, St Paul, MN, EUA, lot number 8RB) was applied to preparation walls in accordance with the manufacturer's recommendations. Preparation walls were previously etched using a 35% phosphoric acid gel (Scothbond Etchant, 3M ESPE, lot number N187625) for 15 s, water-rinsed for 10 s and blotted dry by using absorbent paper. Two layers of the adhesive were consecutively applied with a 5 s interval in between, carefully dried for 15 s in order to remove the solvent and then photoactivated for 10 s using a quartz-tungsten-halogen light-curing unit (XL 2500, 3M ESPE) operating at 700 mW/cm^2. Light intensity was monitored using a handheld radiometer (Model 100, Demetron Research Corp., Danbury, CT) throughout the experiment to ensure that a consistent intensity was maintained. Experimental composite was bulk inserted into the preparation and photoactivated according to the experimental groups (Table I). Photoactivation methods, light exposure and energy doses used are listed in Table I. Specimens were stored in distilled water at 37°C for 24 h and then finished and polished using Soflex discs (3M ESPE). Then, specimens were subjected to the degradation protocols, as described in Table II. Degradation procedures were performed using a thermal fatigue simulator device (MSCT-3, Elquip, São Carlos, SP, Brazil) and a mechanical fatigue simulator (ER 37000, Erios, São Paulo, SP, Brazil).

Table II. Degradation protocols used in this study.

Degradation protocol	Description
(1) Control	Specimens were not subjected to any degradation protocol
(2) Thermal cycling[a]	10,000 thermal cycles; each cycle at the temperatures of 5°C, 37°C and 55°C during 30 s [19]
(3) Mechanical loading[b]	100,000 mechanical cycles, frequency of 4 Hz at 60 N [14,25]
(4) Thermo-mechanical loading	Protocols (2) + (3)

[a] Performed through thermal fatigue simulator device.

[b] Performed through mechanical fatigue simulator device.

Marginal and internal gaps assessment

Marginal and internal gaps were assessed by using Scanning Electron Microscopy (SEM) in which the replica technique was used [20]. An impression of the superficial margins was taken of all the restored teeth using a polyvinylsiloxane material (Aquasil, Dentsply DeTrey, Konstanz, Germany) and positive epoxy resin replicas were obtained (Buehler, Lake Buff, IL). Each replica was then mounted on a metallic stub, gold-sputtered (Balzers-SCD 050 Sputter Coater, Liechtenstein) and observed under SEM (JEOL, JSM-5600LV, Scanning Electron Microscope, Japan). Replicas were visualized at ×25 and ×200 magnifications. The classification of the margins were made at ×200 magnification directly on the microscope monitor and the measurements were made by using a multi-point measuring device

Table III. Percentage of marginal gaps for photoactivation methods and degradation protocols tested in this study.

	Photoactivation methods			
Degradation protocols	HI	LI	SS	PD
No degradation	0.0^{bA}	0.0^{aA}	0.0^{bA}	0.0^{aA}
Thermal cycling	8.5^{bA}	0.0^{aA}	0.0^{bA}	0.0^{aA}
Mechanical loading	62.9^{aA}	7.8^{aBC}	19.5^{aB}	0.0^{aC}
Thermo-mechanical loading	50.1^{aA}	0.3^{aB}	3.7^{abB}	8.2^{aB}

HI, high intensity; LI, low intensity; SS, soft-start; PD, pulse-delay. Different uppercase letters in the rows and lowercase letters in the columns indicate statistically significant differences ($p < 0.05$).

that allowed the observation of the entire perimeter of the restoration at ×25. The perimeter of the restorations was measured. Marginal gap formation was calculated and expressed as the percentage of the measured perimeter of each specimen.

Following, the teeth were mesio-distally sectioned to obtain three sections in a cutting machine (Isomet 1000, Buehler) using a slow rotating diamond disc under water cooling. Internal margins of sectioned slices were replicated and evaluated according to the procedures described above. Means of the three slices were calculated for statistical analysis.

Statistical analysis

The results of marginal and internal adaptation were expressed as the percentage of gap at the margins relative to the total length measured. Data were subjected to 2-way ANOVA and Tukey's test at a significance level of 0.05. All statistical analysis was executed using Assistat Beta 7.5 software.

Results

Marginal adaptation

According to ANOVA, significant differences were noted between the photoactivation methods, the degradation protocols and the interaction between photoactivation methods vs degradation protocols was also significant ($p < 0.05$). Marginal adaptation results, expressed in percentage of marginal gaps, are listed in Table III. No difference among photoactivation methods was observed for control, unstressed groups ($p > 0.05$). For groups submitted to both mechanical loading and thermo-mechanical loading, when the conventional, high intensity activation mode was used, the highest incidence of gaps was observed. It was significantly higher than that exhibited by the groups photoactivated using low intensity, soft-start and pulse-delay.

Table IV. Percentage of internal marginal gaps for photoactivation methods and degradation protocols tested in this study.

	Photoactivation methods			
Degradation protocols	HI	LI	SS	PD
No degradation	37.5^{bA}	32.2^{bA}	22.8^{bA}	35.2^{bA}
Thermal cycling	51.4^{bA}	40.2^{bAB}	31.2^{bB}	47.5^{abA}
Mechanical loading	69.0^{aA}	61.3^{aAB}	50.0^{aB}	57.5^{aAB}
Thermo-mechanical loading	60.9^{abA}	61.2^{aA}	63.0^{aA}	61.8^{aA}

HI, high intensity; LI, low intensity; SS, soft-start; PD, pulse-delay. Different uppercase letters in the rows and lowercase letters in the columns indicate statistically significant differences ($p < 0.05$).

Perfect marginal sealing was observed in the control groups (no degradation). For groups submitted to thermal degradation, only the group photoactivated using high intensity showed a gap in the outer enamel margins, even though no significant difference was observed among high intensity and the other photoactivation methods. Also, no significant difference was observed among the control groups (no degradation) and those submitted to thermal degradation.

A significant increase in the gap formation was observed for groups photoactivated using high intensity when submitted to mechanical loading and thermo-mechanical loading degradation protocols. Marginal adaptation of the groups photoactivated using Low Intensity and Pulse Delay were not affected by any degradation protocol.

Internal adaptation

According to ANOVA, as observed for marginal adaptation data, there was a statistically significant difference between the photoactivation methods, the degradation protocols and the interaction between photoactivation methods vs degradation protocols was also significant ($p < 0.05$). Internal adaptation results, expressed in gap%, are listed in Table IV.

The mechanical loading and the thermo-mechanical loading caused a significant increase in the internal gap formation for all groups, regardless of the photoactivation method when compared to the no degradation groups. For the groups submitted to thermal cycling, a significant increase in the internal gap formation was only observed for the group photoactivated using the soft start technique when compared to the control (no degradation group).

Regarding the photoactivation methods, no difference was observed when no degradation was performed (control). The soft-start photoactivation method provided the lowest percentage of internal gap formation for groups submitted to thermal cycling or mechanical loading stresses.

Discussion

The dimensional stability of the tooth/restoration interface is challenged from the very beginning of

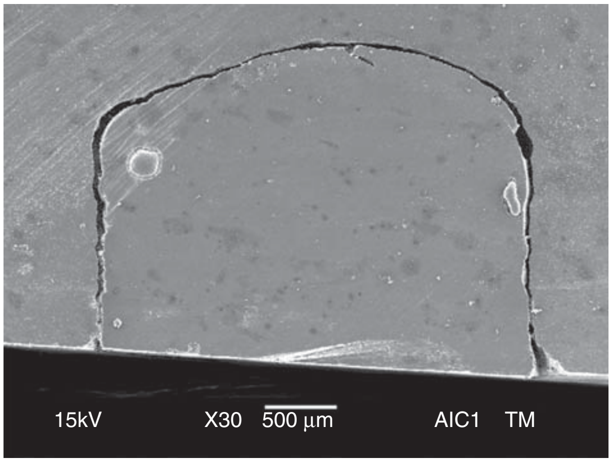

Figure 1. Complete debonding of restoration after thermo-mechanical loading.

the polymerization reaction in composite restorations. Gap formation has been considered one of the first signs of interface failure in bonded restorations [21,22]. In this way, the evaluation of the marginal/interfacial adaptation comprises a relevant issue. The origin of gaps has been related to three main factors: polymerization shrinkage stress [23], bonding failure during the adhesive procedure [24] and external stresses, such as mechanical and thermal stresses [25,26].

In this study, specimens were submitted to 10,000 thermal cycles, which is equivalent to 1 year in the oral environment, according to Gale and Darvel [19]. Extreme temperatures of 5 and 55°C were selected because of the temperature of consuming cold and hot beverages, respectively. An intermediary temperature (37°C) was included due to body temperature, in order to avoid thermal shock with the extreme temperatures, since it does not occur often in clinical conditions.

The hypothesis (1) that the thermal stresses would affect the marginal and internal adaptation of restorations was rejected since no evidence of gap formation/propagation due to thermal stress was noted. Hence, gap formation/propagation in TC groups cannot be attributed to thermal stress, but to the polymerization shrinkage or failure in the adhesive procedure. These results corroborate with those obtained by Wendt et al. [27] and de Smith et al. [28] that found no significant difference when the experimental groups were submitted to thermal cycling in comparison to the control, untreated groups. A similar behavior of shrinkage/expansion of restorative composite and tooth structure face to the thermal variation can explain this result.

Conversely, the effect of mechanical loading was clearly observed in this study, confirming the hypothesis (2) that mechanical loading negatively affects marginal adaptation of restorations. Reasons that explain these differences rely on the elastic modulus of the compounds of the restorations. Different plastic/elastic deformation degrees occur at the interface, creating microcracks between the preparation floor and the adhesive layer [17]—known to be a stress concentration area. Deformation varies between 0.1–1 μm/kg [29] depending on the elastic modulus. In addition, mechanical loading is related to fatigue, which may cause catastrophic failures in bonded restorations. In fatigue failures, the crack begins in one defect (gap, bobble or void) and progressively continues until a total failure occurs (Figure 1). Marginal fractures are indicative of fatigue failure. In this condition, materials and interfaces often fail after repeated sub-catastrophic loadings, with low stress. Results of the present study, regarding mechanical loading, certainly jeopardized the marginal quality, corroborating with several studies in which were found a decrease in the bond strength or an increase in the gap formation in composite restorations submitted to mechanical loading [30,31]. In the present study, the specimens were submitted to 100,000 mechanical cycles in a wet environment (frequency of 4 Hz and load of 60 N). This degradation protocol was selected based on previous studies [14,15,25] that pointed out that this protocol was effective in reducing the marginal quality of composite restorations.

A synergic effect caused by the association of thermal and mechanical cycling when evaluating the interfacial degradation has been shown [14]. However, this was not observed in the present study; the association of thermal and mechanical cycling produced no significant increase in the gap formation when compared to that produced only when mechanical loading was performed, rejecting hypothesis (3). In this way, the fact that thermal cycling caused no significant increase in the gap formation, also allows one to conclude that the mechanical loading is the main responsible factor to the interfacial degradation, when external stresses were considered. These findings highlight the importance to evaluate the efficacy of the restorative procedures under external stress, especially when using the chewing simulation.

Several studies have shown that the photoactivation method can interfere in the polymerization kinetics [5,6,11]. Thus, the speed at which the polymerization reaction occurs depends upon the light intensity [32]; and the degree of conversion is a function of energy dose [33]. A common idea is that high light intensity is important to achieve improved mechanical properties. However, this increases the polymerization speed, which leads to the development of a rigidity network, decreasing the possibility of accommodation of polymeric chains to the preparation walls [34]. In this situation, a fast increase in the elastic modulus accompanied by a volumetric shrinkage generates stress at the bonding interface, which causes an immediate failure in the adaptation of composite restorations [23]. However, this was not observed in this study. In the control groups and TC conditions, a perfect marginal sealing was observed in the majority of the specimens. In this way, the research hypothesis (4) must be rejected. Restorations photoactivated using modulated methods exhibited no improved marginal and better internal adaptation than those photoactivated using a conventional, continuous photoactivation method, at least at first, considering groups not subjected to any degradation protocol.

However, when specimens were submitted to mechanical loading and thermo-mechanical cycling, a significant increase in the marginal and internal gaps was observed, but this increase in the gaps depended on the photoactivation method applied. Specimens photoactivated using modulated and low power density methods showed better performance after mechanical loading and thermo-mechanical cycling, allowing the research hypothesis (5) to be accepted. In this sense, it can be supposed that, in the HI group, although a good sealing can be immediately achieved (control, unstressed group), the adhesive interface is in a stress condition (due to high shrinkage stress) after the photoactivation procedure, enabling more gap to be formed/propagated when specimens are subjected to mechanical loading.

The dental literature regarding the influence of modulated photoactivation methods in the gap formation is still controversial. Reasons that explain this controversy rely on the fact that the gap formation is of multi-factorial cause. In this way, it can be considered that the efficacy of the modulated photoactivation in reducing stress and increasing marginal adaptation is material composition-dependent [6].

Positive effects of modulated photoactivation methods were observed in the present study only when the specimens were subjected to mechanical loading and thermo-mechanical loading. In these conditions, SS and PD groups, as well as the LI group, showed lower incidence of gaps than the HI group. Pulse delay technique allowed the best performance of the composite restorations regarding marginal adaptation, even after mechanical loading all specimens exhibited perfect sealing. In addition, the soft start technique allowed the best performance of restorations in terms of internal adaptation. Low intensity photoactivation mode groups showed intermediate results, but similar to those observed when modulated methods were applied considering the majority of conditions.

Polymerization kinetics is altered by modulated methods [4,7,11,35]. A short delay in the shrinkage development is observed. Additionally, a decrease in the shrinkage stress and an increased marginal quality in bonded restorations photoactivated using the soft start method have been demonstrated [3,13,36,37]. The decrease in the shrinkage stress using the pulse delay method has been related to a decrease in the polymerization speed, allowing a slow development of the elastic modulus and stress relief due to a molecular rearrangements, polymer flow and deformation [4]. Considering both the literature and the results of the present study, it can be stated that the restorations photoactivated using modulated methods may exhibit a higher resistance to mechanical degradation due to a more favorable development of shrinkage stress. Also, it is important to state that the mechanical properties are not jeopardized by the modulated method as long as the energy dose is kept constant [38]. Alonso et al. [39], using the same restorative material and the same photoactivation methods of this study, observed that both degree of conversion and hardness were not affected when using low power density or modulated photoactivation methods.

In this way, it can be suggested that restorations photoactivated using modulated methods are less susceptible to mechanical degradation and should be recommended in an attempt to increase the durability of resin composite restorations.

Conclusions

Within the limitation of this study, it is possible to conclude that:

1. Composite restorations photoactivated using low intensity or modulated methods exhibit improved resistance to thermo-mechanical degradation;
2. Mechanical loading is the determinant factor for interfacial degradation of composite restorations, whereas thermal cycling has no effect on gap formation; and
3. The association of thermal and mechanical stresses produces no synergic effect. The degradation effect is similar to that caused when only the mechanical loading is performed.

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