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# Can the pulse-delay photoactivation technique substitute the conventional technique? – evaluation by microhardness tests

*A técnica de fotoativação pulso-espera pode substituir a técnica convencional? – avaliação através de ensaios de microdureza* 

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#### Resumo

Introdução: A contração de polimerização é uma propriedade inerente às resinas compostas a qual pode ser responsável por eventos como infiltração marginal, sensibilidade pós operatória e trincas na estrutura dental. Com o intuito de minimizar tais efeitos adversos, técnicas de polimerização alternativas podem ser utilizadas. Objetivo: Avaliar a eficácia da técnica pulso-espera na ativação de resinas compostas através da microdureza Vickers. Material e método: Trinta e cinco corpos de prova foram confeccionados e distribuídos em sete grupos: De acordo com o grupo, os corpos de prova tinham espessura de 2 ou 4 mm, inseridos como único incremento ou incrementos de 1 mm, e foram fotoativados com 500 mW/cm² durante 40 s (fotoativação convencional) ou com ativação de 250 mW/cm<sup>2</sup> durante os primeiros 3 s, seguido de intervalo de 1 min para a ativação de cada incremento, e espera de 5 min para ativação final por 40 s (técnica pulso-espera). A microdureza foi medida na superfície oposta à fotoativação, exceto para o grupo controle que foi medido na superfície irradiada. **Resultado**: A média (VHN) das durezas encontradas foram: G1-  $86,5 \pm 2,0$ ; G2-  $52,8 \pm 2,3$ ; G3-  $92,3 \pm 1,4$ ; G4-  $86,9 \pm 1,7$ ; G5-  $91,2 \pm 2,4$ ; G6-66,2 ± 1,7; Controle-101,3 ± 2,7. O teste ANOVA para dois fatores de variação, espessura do incremento e técnica de fotoativação (F = 404,79) e o teste de Tukey (T = 4,56) evidenciaram diferenças significativas entre os grupos (p < 0,01), exceto entre os grupos 3 e 5. **Conclusão:** Os resultados mostraram que, com 2 mm de profundidade, todas as técnicas de inserção e fotoativação empregadas apresentaram polimerização adequada. No entanto, a 4 mm de profundidade, apenas a técnica incremental com ativação convencional apresentou polimerização satisfatória.

Descritores: Resinas compostas; dureza; polimerização; fotoativação.

## Abstract

**Introduction:** The polymerization shrinkage is an inherent property of the resins which may be responsible for events such as microleakage, postoperative sensitivity and microcracks in the dental structure. In order to minimize such adverse effects, alternative polymerization techniques can be used. **Objective:** Evaluate the efficacy of the pulse-delay technique in the activation of composites by Vickers microhardness. **Material and method**: Thirty-five samples were created and divided into seven groups: According to the group, the specimens had thickness of 2 or 4 mm, could be inserted as single increment or 1mm increments, and were photoactivated with 500 mW/cm<sup>2</sup> for 40 s (conventional photoactivation) or received activation of 250 mW/cm<sup>2</sup> during the initial 3 s, with 1 min delay for activation of each increment, and 5 min delay to final activation for 40 s (pulse-delay technique). Vickers microhardness was measured on the bottom surface, except for the control group which was measured at the top surface. **Result**: The hardness (VHN) found were: G1- 86.5 ± 2.0; G2- 52.8 ± 2.3; G3- 92.3 ± 1.4; G4- 86.9 ± 1.7; G5- 91.2 ± 2.4; G6- 66.2 ± 1.7; Control- 101.3 ± 2.7. ANOVA for two variation factors, increment thickness and photoactivation technique (F = 404.79) and Tukey test (T = 4.56) showed significant differences among groups (p < 0.01), except between groups 3 and 5. **Conclusion:** Results showed that, with 2 mm in depth, all insertion/ photoactivation technique semployed presented suitable polymerization. However, at 4 mm in depth, only incremental technique with conventional polymerization showed to be efficient .

Descriptors: Composite resins; hardness; polymerization; photoactivation.

#### INTRODUCTION

Control of the binomial degree of polymerization versus polymerization contraction is a difficulty associated with direct composition resin restorations. When a composite resin polymerizes, monomers are cross-linked in long chains. In forming this link, the monomer molecules come close to one another, and result in a lower volume at the end of the reaction. This is the polymerization contraction inherent to resins. The greater the degree of conversion of monomers into polymers, the greater the polymerization contraction<sup>1-3</sup>. High levels of energy irradiance increase the degree of conversion of composites. The irradiance absorbed by the composite is directly related to the initial generation of free radicals of dimethacrylate monomers<sup>4</sup>.

In an attempt to obtain better mechanical properties, increasing the degree of conversion of resin, polymerization contraction is undesirably increased. Its detrimental consequences are: post-operative sensitivity, bacterial infiltration in the tooth-restoration interface, cavity recurrence and formation of cracks in the dental remnant<sup>5</sup>.

In order to control the stress generated by polymerization contraction, other photoactivation techniques such as soft-start, ramp, pulse and pulse-delay are suggested. These techniques all use initial low-intensity irradiation, thus reducing the speed of the monomer/polymer conversion reaction. The reaction proceeds slowly, allowing stress relief through the flow of the molecules on the non-adhered surface during the pre-gel phase. The idea is for the maximum flow to occur before a high intensity of light can be used to complete the polymerization reaction<sup>3,4</sup>. In the pulse-delay technique, after an initial pulse, which triggers polymerization, there is a waiting period before considerably slow polymerization occurs, followed by the performance of final activation with high intensity<sup>6,7</sup>.

Other studies<sup>7-11</sup>, however, show that alternative photoactivation techniques, although mitigating the effects of polymerization contraction, provide inferior mechanical results for composite resin restorations, due to unsatisfactory polymerization.

The surface hardness analysis has been used as an indirect method of evaluation of the degree of polymerization of composite resins<sup>12</sup>.

The aim of this study was to evaluate whether the pulse-delay technique is capable of substituting the conventional photoactivation technique while maintaining acceptable values of hardness. In this context, the purpose is to analyze in vitro in-depth Vickers microhardness of a hybrid composite for posterior teeth photoactivated with the conventional technique and the pulse-delay alternative, in the techniques of incremental insertion and in single portion, in the thicknesses of 2 and 4 mm.

# MATERIAL AND METHOD

Thirty-five cylindrical specimens were prepared with the assistance of black, cylindrical polypropylene matrixes, 1 mm thick with an internal orifice in a diameter of 4 mm. All specimens

were prepared by a unique calibrated operator for technique failure did not represent a limitation of the study. These matrixes were fitted into a matrix holder of dull brass forming an assembly resting on a glass slide to allow surface smoothness and levelness of the composite (Filtek P60<sup>R</sup> 3M ESPE Dental Products - St. Paul, MN, USA). The glass slide was placed on a sheet of black pasteboard producing a negative effect of reflection on the lower surface.

Specimens were randomly distributed into 7 groups (n = 5) according to: the insertion technique (single-portion and incremental), thickness (2 and 4 mm) and photoactivation (conventional and pulse-delay), forming six experimental groups and one control group (Table 1). The mechanical test was of Vickers microhardness.

For the insertion of the composite in the incremental technique, the 1 mm matrixes were overlapped to attain the specimen thickness compatible with the group in question, which could be 2 or 4 mm thick. The distance between the irradiation source and the surface of the specimen was thus standardized in 0 mm.

The height of the matrix holders corresponded to 0.5 mm below the height of the specimen, in order to avoid interference in the adaptation of the glass plate that was resting on the surface of the composite resin to be irradiated.

The irradiating tip was leaned against the glass plate and positioned in the center of the specimen for the activations.

In the single increment insertion with conventional photoactivation groups, the irradiation time was 40 s and the intensity was 500 mW/cm<sup>2</sup>, offering energy density of 20 J/cm<sup>2</sup>. In the incremental insertion with conventional photoactivation technique groups, the increments had a thickness of 1 mm and each increment received irradiation for 40 s at 500 mW/cm2. The energy density for the groups with thickness of 2 mm was 40 J/cm<sup>2</sup> while for the 4mm groups it was 80 J/cm<sup>2</sup>. In the insertion/ increment pulse-delay photoactivation technique groups, each 1 mm increment received an irradiation pulse of 3 s at 250 mW/cm<sup>2</sup>. The waiting time between each pulse was 1 min. The next increment was inserted during this time. After a waiting time of 5 min final activation was performed for 40 s at 500 mW/cm<sup>2</sup>. The energy density used was 21.5 J/cm<sup>2</sup> in the 2 mm specimens and 23 J/cm<sup>2</sup> in the 4 mm specimens. The specimens from the control group were prepared in single portion, with thickness of 1 mm, and photoactivated for 160 s at 500 mW/cm<sup>2</sup>, with the offering of energy density of 80 J/cm<sup>2</sup> (Table 1). This methodology used, including intensity of the radiation source was based on literature<sup>13,14</sup>.

Once prepared the specimens were containerized in a completely sealed container with total absence of light or air renewal, and were stored in a incubator at 37 °C for 7 days.

The surface opposite to the light application was of interest for the measurement of the Vickers microhardness, except for the control group that had microhardness measured on the irradiated surface. The tests were conducted with the HMV-2000<sup>R</sup> (SHIMADZU- Kyoto, Japan) micro hardness tester with Vickers

Group	Insertion technique	Thickness of the specimen	Irradiation time	Waiting times	Intensity of the irradiation source	Energy density (J/cm <sup>2</sup> )
1	Single increment	2 mm	40 s	-	500 mW/cm <sup>2</sup>	20
2	Single increment	4 mm	40 s	-	500 mW/cm <sup>2</sup>	20
3	Incremental (1 mm per increment)	2 mm	40 s (per increment) Total 80 s	-	500 mW/cm <sup>2</sup>	40
4	Incremental (1 mm per increment)	4 mm	40 s (per increment) Total 160 s	-	500 mW/cm <sup>2</sup>	80
5	Incremental (1 mm per increment)	2 mm	3 s per increment + 40 s total final activation of 46 s	1 min per incre- ment 5 min for final activation	250 mW/cm <sup>2</sup> in the pulses and 500 mW/cm <sup>2</sup> in the final activation	21.5
6	Incremental (1 mm per increment)	4 mm	3 s per increment + 40 s total final activation of 52 s	1 min per incre- ment 5 min for final activation	250 mW/cm <sup>2</sup> in the pulses and 500 mW/cm <sup>2</sup> in the final activation	23
Control	Single increment	0 mm (irradiated surface)	160 s	-	500 mW/cm <sup>2</sup>	80

Table 1. Characteristics o	f the com	posite specimens	distributed	among the	groups of interest
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indenter, using 50 gf load and time of 45 s. Five indentations were made, one in the center of the specimen and the other four at a distance of 100  $\mu m$  above, below, to the right and to the left of the first indentation made.

Data were submitted to the analysis of variance (ANOVA) for two variation factors (increment thickness and photoactivation technique) and Tukey tests with significance level of 1%. To assess the degree of polymerization, an analysis of percentage of maximum hardness (PMH) was conducted by means of the relationship between the hardness obtained in the groups and that obtained on the surface of the control group, expressed as a percentage. The minimum PMH value considered was defined as 80%<sup>15</sup>.

## RESULT

For the data analysis the arithmetic means of hardness were calculated for each specimen. The ANOVA for two variation factors results showed significant differences (p < 0.01) among the groups (F = 404.79), except between groups 3 and 5. Tukey's critical value (T = 4.56) was calculated for contrast analysis among the mean values of the groups. The mean values of hardness for each group, with the respective standard deviations, and the significances among them, are presented in Table 2. The PMH analysis for the experimental groups can be observed in Table 3.

## DISCUSSION

An adequate degree of conversion of a composite resin is required as the mechanical properties of the future restoration will depend intimately on the polymerization attained, especially in depth<sup>6,10</sup>. A portion of the composite from the light source absorbs less energy when compared with portions closer to the radiating edge, because the light undergoes scattering and reflection<sup>16</sup>. However, it is known that the higher the degree of conversion of monomers into polymers, the greater the polymerization contraction<sup>17</sup>, which generates stress and deformations on the tooth-restoration interface<sup>18,19</sup>.

Alternative photoactivation techniques can be used to control the stress that occurs during polymerization contraction. Cunha et al.<sup>17</sup> observed that using four photoactivation methods (continuous light, soft-start and two forms of activation with the pulse-delay technique) with different powers (80 and 150 mW), they concluded pulse-delay photoactivation methods reduce the shrinkage stress without compromising the degree of conversion of composite resin<sup>17,20</sup>.

Yap et al.<sup>21</sup> verified the effect of the photoactivation techniques in pulse, pulse-delay and soft-start on post-gel polymerization contraction, in composite resin specimens. They perceived that in the initial stages after activation, the alternative techniques exhibited less polymerization shrinkage than the control technique of 400 mW/cm<sup>2</sup> for 40 s. However, when considering all the time intervals, the reduction of polymerization contraction was not significant in the alternative techniques in relation to the conventional technique<sup>21</sup>.

This study included the performance of a Vickers microhardness test, which is important to evaluate the mechanical behavior of composite resins as it is associated with their degree of polymerization, especially in depth<sup>22</sup>.

The influence of the incremental insertion pulse-delay photoactivation technique in the Vickers microhardness, when compared with the single increment with conventional photoactivation technique, can be observed in the comparisons between Groups 1 and 5 and groups 2 and 6 (Table 2). It is possible to perceive that, for the thickness of 2 mm, the mean microhardness of the single portion insertion group is

Table	2.	Mean	values	of	Vickers	microhardnes	is by	Group,	with
respec	tive	e stand	ard dev	iati	ons				

	Mean value of Vickers microhardness (VHN) (Standard deviation)
Group 1	$86.5 \pm 2.0^{\circ}$
Group 2	52.8 ± 2.3 <sup>e</sup>
Group 3	$92.3 \pm 1.4^{b}$
Group 4	$86.9 \pm 1.7^{\circ}$
Group 5	$91.2 \pm 2.4^{b}$
Group 6	$66.2 \pm 1.7^{d}$
Control group	$101.3 \pm 2.7^{a}$

Load: 50 gf - Time: 45 s.

Table 3. Analysis of the percentage of maximum hardness (PMH)

	PMH (%)
Group 1	85.4
Group 2	52.2
Group 3	91.1
Group 4	85.8
Group 5	90.0
Group 6	65.4

86.5 VHN and for the incremental insertion with pulse-delay photoactivation technique it is 91.2 VHN, with significant difference between the mean values (p < 0.01), and an increase of 5.4%. For the thickness of 4 mm, the hardness for the single increment group is 52.8 VHN, while for the group of incremental technique and pulse-delay it is 66.2 VHN. Therefore it is possible to observe an increase of 25.3%, with significant differences among means (p < 0.01). However, in these groups (2 and 6), PMH were unsatisfying. Statistically significant differences were also observed in the study of Dalli 'Magro et al. (2008) in which they observed a decrease in hardness from 3 mm in all the groups compared with hardness at the top<sup>23</sup>.

It is known that increases in the thickness of a composite resin specimen generally cause a decrease of hardness in their deeper portions<sup>20</sup> especially when this specimen is filled in a single portion or in increments above 2 mm. According to Gauer et al.<sup>24</sup>, Vickers hardness values are higher at the top (side facing the light) than at the base, regardless of the light source and of the type of photopolymerizer used<sup>24</sup>.

The incremental insertion with pulse-delay photoactivation technique was also compared with the incremental insertion with conventional photoactivation technique, which can be analyzed by comparing groups 3 and 5 and groups 4 and 6 (Table 2). In the thickness of 2 mm the microhardness obtained with the insertion/conventional incremental photoactivation technique

is 92.3 VHN, while with the insertion/incremental pulse-delay photoactivation technique it is 91.2 VHN, with no significant difference between the means. In the thickness of 4 mm there is significant difference (p < 0.01) between the mean values of microhardness, going from 86.9 VHN, in the incremental insertion with conventional photoactivation technique, to 66.2 VHN, in the incremental insertion with pulse-delay photoactivation technique. The reduction, using the pulse-delay technique, with 4 mm, in relation to the conventional technique, is 23.8%. Corroborating the results obtained, Camargo et al.<sup>1</sup> ratify the concept that 2 mm should be the ideal thickness of an increment to reach a good degree of conversion<sup>1</sup>.

For the thickness of 2 mm of composite resin, it can be considered that the incremental pulse-delay technique will become a more suitable choice than the incremental technique with conventional activation. The reason is that the techniques have equivalent in-depth polymerization. That is, a resin with adequate mechanical properties and good control of polymerization contraction is to be expected, yet we must emphasize that more clinical time is necessary for the use of this technique<sup>1-3</sup>. For 4 mm of thickness the incremental pulse-delay technique obtained as a result a inferior hardness to the incremental technique with conventional polymerization. This evidently limits the indication of this technique for clinical use. The fact is that the clinician often comes across cavities deeper than 2 mm, particularly in the proximal boxes of posterior teeth, which require more technical care for satisfactory polymerization<sup>25</sup>. In this case, the incremental insertion with conventional photoactivation technique appears more appropriate. In the overall analysis it would be possible to affirm that the incremental technique with conventional polymerization behaves in a superior way to the incremental pulse-delay technique for the in-depth polymerization of resin.

The percentage of maximum hardness (PMH) was the method chosen to evaluate the technique in terms of in-depth polymerization, gauged by Vickers microhardness (Table 3). The differences between the mean values of the control group and all the other groups proved to be statistically significant (p < 0.01). The PMH showed that groups 1, 3, 4 and 5 exhibited values above 80% (minimum limit determined as acceptable) <sup>15</sup>. They include only one group with specimens in a thickness of 4 mm, group 4, of incremental insertion with conventional photoactivation technique. The other groups that exceeded the PMH limit of 80% were all with specimens with thickness of 2 mm: group 1, group 3 and group 5. The single increment insertion group measuring 4 mm (group 2) and the incremental insertion pulse-delay photoactivation technique groups measuring 4 mm (group 6) did not reach the minimum limit of PMH.

Based on the PMH analysis it can be inferred that for considerable thicknesses such as 4 mm, only the incremental insertion with conventional photoactivation technique provides adequate in-depth polymerization results, while all the other techniques are inadequate. With 2 mm of thickness in the specimen, all the techniques cure the resin adequately.

### CONCLUSION

Even with the benefit promoted by the pulse-delay photoactivation technique with incremental insertion, as the

control of over polymerization shrinkage, this technique can substitute the conventional technique only for depths not exceeding 2 mm. At depths of 4 mm pulse-delay technique can not replace the conventional technique.

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# CONFLICTS OF INTERESTS

The authors declare no conflicts of interests.

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