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Effect of the photoactivation method on composite resin cure

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ABSTRACT | For composite resins to obtain good properties, they must present a high degree of conversion and minimal polymerization contraction. To minimize this contraction, alternative photoactivation techniques have been suggested. The objective of this study was to compare the hardness of a photoactivated composite resin using the pulse-delay, soft start and conventional techniques, in thicknesses of 1, 2 and 3 mm in the irradiated surface and in the opposite surface. Photoactivation was performed with halogen light for 20 seconds in the conventional and soft start techniques. In the pulse-delay technique, each increment of 1 mm was photoactivated for 3 seconds with a final photoactivation of 40 seconds. The samples were stored in an oven at 37°C for one week and submitted to the Vickers microhardness test. The results were submitted to ANOVA and the Tukey's test with a level of significance of 5%. It was concluded that the hardness was higher with photoactivation of 40 seconds, and for the techniques that had the photoactivation time of 20 seconds (conventional and soft start), there was no difference in terms of hardness. Therefore, the hardness is not influenced by the technique, but rather, by the polymerization time.

DESCRIPTORS | Hardness; Polymerization; Composite Resin.

RESUMO | **Efeito do método de fotoativação na polimerização da resina composta** • Para que as resinas compostas obtenham boas propriedades devem apresentar um alto grau de conversão e o mínimo de contração de polimerização. Para minimizar essa contração, técnicas alternativas de fotoativação têm sido sugeridas. O objetivo deste estudo foi comparar a dureza de uma resina composta, na superfície irradiada e na superfície oposta, fotoativada pelas técnicas pulso-espera, soft start e convencional em espessuras de 1, 2 e 3 mm. A fotoativação foi realizada com fotoativadores de luz halógena por 20 segundos nas técnicas convencional e soft start. Na técnica do pulso-espera cada incremento de 1 mm foi fotoativado por 3 segundos com fotoativação final de 40 segundos. Os corpos de prova foram armazenados em estufa a 37°C por uma semana e submetidos ao teste de microdureza Vickers. Os resultados foram submetidos à ANOVA e ao teste de Tukey com nível de significância de 5%. Foi concluído que a dureza foi maior com a fotoativação por 40 segundos e para as técnicas que tiveram o tempo de fotoativação de 20 segundos (convencional e soft start) não houve diferença quanto à dureza. Dessa forma, a dureza não é influenciada pela técnica, mas sim pelo tempo de polimerização.

DESCRITORES | Dureza; Polimerização; Resina Composta.

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INTRODUCTION

Composite resins have been widely used in dental clinics, as the main material for direct esthetic restorations in a wide range of dental procedures, as a luting agent for indirect restorations.

The activation of the polymerization process can be induced by heat, chemical reaction, or photochemical reaction. The system of photoactivation by visible light is the most common in today's composite resins, and has the advantage of enabling greater control of the work time, but the profound limitation of light penetration requires it to be inserted into the cavity in incremental stages.[1]

Photopolymerization composite resins contain a photoinitiator, of which camphorquinone is the most common, which when activated by blue light, transforms into a free radical that breaks the carbon double bonds of the monomers present in the matrix, initiating the polymerization process.[1]

The higher the proportion of monomers that converts into polymers, the higher the degree of conversion, and consequently, the better the mechanical and biological properties of the resin. The hardness is directly correlated with the degree of conversion, and can be used as a method to determine the degree of polymerization of resins.[2-4]

A high degree of conversion is important to achieve good properties in the material. This conversion percentage also influences the contraction of polymerization of the resin, which is one of the main problems associated with this material, as it leads to the formation of tensions on the interface between the tooth and the restoration. These tensions can result in postoperative sensitivity, marginal infiltration, and cracks in the tooth enamel. In an attempt to minimize the effects caused by the contraction of polymerization, some alternative photoactivation techniques (pulse-delay and soft start) have been suggested. This way, considering the constant development of dental materials and the improvement of techniques for evaluation of their properties, studies are always necessary to consolidate new concepts and assess their likely clinical performance, studying possible alternatives to improve them. The objective of these techniques is to release the tensions caused by contraction on the free surface.[5-7]

The techniques used in this study were the soft start, pulse-delay and continuous conventional techniques. The soft start technique has low initial intensity in the first few seconds, followed by full intensity.[8] The pulse-delay technique begins with slow polymerization, unleashed after an initial pulse, followed by a waiting period, before final activation with high intensity to complete the polymerization.[9-10] There are few studies evaluating the effectiveness of these techniques on the irradiated opposite surface. Therefore, to evaluate the hardness in the opposite surface can be an alternative to define real effectiveness of the techniques to activate a composite resin.

During the polymerization, a composite's viscoelastic behavior changes from viscous (pre-gel phase) to predominantly elastic (post-gel phase), and its capacity to accommodate the reduction in volume through flow decreases accordingly.[11] Several authors have reported effectiveness of alternative photoactivation techniques in the reduction of tension caused by contraction of composite resins.[12-15] Some studies reported a decrease of approximately 28% in tension obtained with pulse-delay methods.[13,15-16] Resin samples photo activated by alternative techniques showed degree of conversion and hardness values similar to samples cured by continuous high intensity irradiation.[15]

The objective of this study was to compare the Vickers hardness of a photoactivated composite resin by the soft start, pulse-delay and continuous conventional techniques, in thicknesses of 1, 2 and 3 mm in the irradiated surface and in the opposite surface.

MATERIALS AND METHODS

The microhardness of both surfaces (irradiated or opposite) of composite resin samples of different thicknesses (in three levels: 1, 2 or 3 mm), photoactivated by different techniques (in three levels: pulse-delay, soft start and continuous conventional) was evaluated. Forty-five samples were prepared, distributed in 9 groups with 5 samples in each, according to the thickness of the matrix and the photoactivation technique.

For the preparation of the samples, the composite resin Filtek™ Z350 (3M ESPE, St Paul, Minnesota, USA), color $A^{3,5}$, was inserted in circular matrices of black polypropylene with heights of 1, 2 and 3 mm. Each matrix was placed on a glass slide, to obtain a smooth, flat surface, and black card was placed over this slide, to prevent reflection of light from underneath. Another glass slide was placed on the surface of the resin, to flatten it and make it parallel with the horizontal plane.

The resin was activated with halogen light (Degulux SoftStart®, Degussa-Hulls, Buehler, Dusseldorf, Germany) and the photoactivation time and intensity varied according to the technique. For the pulse-delay technique, 1 mm portions were inserted, one on top of the other, totalling the thickness of the matrix (1, 2 or 3 mm). After insertion of each portion, photoactivation was applied for 3 seconds, and after the last portion, the sample was photoactivated for 40 seconds. The total energies supplied for each thickness were: 1 mm=21500 J, 2 mm=23000 J and 3 mm=24500 J. In the soft start method, the samples were photoactivated for 20 seconds, the first 10 seconds at intensity of 100 mW/cm² and the other 10 seconds at 500 mW/cm² (6000 J) for each thickness of the matrix. In the continuous conventional technique, the polymerization time was 20 seconds, with intensity of 500 mW/cm² (10000 J) for each thickness of the matrix. In all the groups, the photoactivation was performed with the tip of the photoactivator perpendicular to the surface of the matrix, and leaning on it. The opposite surface to the irradiated surface was identified with a marker pen; each sample was kept dry and was stored in a black receptacle at 37°C for seven days.

After this seven-day period, five Vickers microhardness indentations were made, with load of 25 gf for 30 seconds, on each surface of each sample; one in the center and four at the edges, with distances of 100 µm between them. A microhardness tester (HMV-2000, Shimadzu Co., Kyoto, Japan) was used for this purpose, using the software program CAMS-WIN. The values obtained from the opposite and irradiated surface were compared with the hardness values obtained on the irradiated side, in each sample.

RESULTS

The results obtained consisted of 450 microhardness values (9 groups with n=5), resulting from the cross-referencing of three techniques, three different thicknesses, two surfaces, five repetitions and five measurements of each sample.

For the statistical analysis, the averages of five measurements for each sample were calculated, resulting in 90 values corresponding to the 18 groups studied. These 18 groups of values were submitted to ANOVA and the Tukey's test (homogeneous averages), with a level of significance of 5%. ANOVA showed that there was a statistically significant difference between the groups ($p<0.05$) and by the Tukey's test, the averages of the 18 groups were compared between them (Table 1).

Based on these comparisons, it can be noted that on the irradiated side, the continuous conventional and soft start techniques caused the same hardness in all three thicknesses (1, 2 and 3 mm). The pulse-delay technique presented statistically higher results than the continuous conventional and soft start techniques at thicknesses of 1 and 2 mm. For the thickness of 3 mm in soft start

Table 1 | Average of hardness values found on each surface; 1, 2 and 3 are the thicknesses of the matrices

Irradiated surface			Opposite surface			
Thickness (mm)	Continuous Conventional	Soft start	Pulse-delay	Continuous Conventional	Soft start	Pulse-delay
1	77.66a	76.78a	94.90b,c	75.90a	79.68a	96.95b
2	75.92a,d	77.07a	95.53b,c	67.35d	50.95e	75.71a
3	77.338a	88.40a,c	96.48b,c	39.52f	31.55f	66.51d

technique has not presented difference comparing with the technique pulse-delay in all thicknesses. In the 1mm thickness, all the techniques presented equal hardness values between the irradiated side and the opposite side for each technique. In the 2 mm thickness, the continuous conventional technique presented no difference between the results for the irradiated side and the opposite side. In the 3 mm thickness, the results showed that all the techniques present statistically different values between the two sides, with higher values on the irradiated surfaces.

DISCUSSION

The composite resin polymerization process occurs by the conversion of molecules of monomers in a polymer chain, accompanied by the connection of these molecules, occupying a small volume than at the start. This reduction in total volume of the material is known as polymerization contraction. Although composite resin is considered the best material for direct esthetic restorations, polymerization contraction is one of the factors that most contributes to the failure of the restorations,[16-19] as polymerization contraction of a composite resin generates tensions and deformities in the interface between the tooth and the restoration.[20-21] According to Ferracane and Mitchem[20] the low contraction of a composite resin promotes the lowest stress on the interfacial bond, and this resulted in a smaller marginal gap formation and lower leakage.

In order to control the stresses generated by polymerization contraction, other photoactivation techniques, like the soft start, ramp, pulse, and pulse-delay techniques, are suggested.[7,18,21-23] These techniques all use low-intensity initial radiation, thereby reducing the speed of reaction of conversion of monomers into polymers. The reaction takes place slowly, reducing stresses through the flow of molecules on the non-adherent surface during the pre-gel phase. The idea is that maximum flow will occur before a high intensity light can be used to complete the polymerization reaction. Various authors have reported on the efficacy of soft start photoactivation or pulse-delay methods in reducing contraction tension of composite resins.[7,18,21-23] In this study, the pulse-delay and soft start techniques were used. In the pulse-delay technique, after an initial pulse, which unleashes the polymerization; this is followed by a delay, so that polymerization occurs very slowly, then a high-intensity final activation is carried out.[7,9-10] In the soft start technique, photoactivation is initiated with several seconds of low irradiance, passing immediately to maximum irradiance (6000 J).[7,18,21] The results of this study showed that the groups presented the same hardness on the irradiated surface, except for the pulse-delay groups, which present higher values. This variation may be related to the higher total quantity of energy emitted in the pulse-delay method (21500 to 24500 J), while the soft start technique presented 6000 J, and the conventional technique presented 10000 J.

Witzel et al.[23] and Cunha et al.[24] compared four methods of photoactivation (continuous conventional light, soft start, and two forms of activation with the pulse-delay technique) with different potencies (80 mW and 150 mW). The pulse-delay photoactivation methods reduced the contraction tension, without compromising the degree of composite resin conversion. However, other studies[9,25-28] show that alternative photoactivation techniques, despite lessening the effects of polymerization contraction, provide poorer mechanical results for composite resin restorations, due to unsatisfactory polymerization.

However, it should be emphasized that in the pulse-delay technique, a time of 40 seconds was used,[28-29] while in the other two techniques, a time of 20 seconds was used, according to the manufacturer's instructions. Considering that the photoactivation time is directly related to the hardness and degree of conversion of the composite resins,[5] and the observation that after 40 seconds the resin presented greater hardness than after 20 seconds, it is presumed that this resin reached a higher degree of polymerization, i.e. only 20 seconds was not enough time for the resin to reach its maximum polymerization capacity, and therefore, maximum hardness. The analysis of surface hardness has been used as an indirect method to evaluate the degree of polymerization of composite resins.[30] In the present study, the Vickers microhardness test was used, to evaluate the mechanical behavior of composite resins because it is associated with the degree of polymerization of the resins, particularly at greater depths. As observed (Table 1), at a 2 mm depth, only the continuous conventional group did not present any difference in hardness between the irradiated surface and the opposite surface, but at a depth of 3 mm, all the groups presented lower hardness values on the side opposite the irradiated side. Thus, it can be stated that the hardness of a composite resin decreases as the depth increases.

Rueggeberg et al.,[31] by using the convential photoactivation method, also found no difference in microhardness for thicknesses of up to 2 mm of composite resin, and De Araújo et al.[32] found an inversely proportional relationship between hardness and thickness of the composite resin layer, with thicknesses greater than 2 mm also presenting low values for microhardness.

Corroborating these results, Camargo et al.[7] when comparing the hardness after 4 photoactivation methods (stepped, ramped, pulse-delay and continuous conventional) in 4 different thicknesses (0.1, 1, 2 and 4 mm), also observed that at thicknesses of up to 2 mm, all the techniques presented satisfactory polymerization. Statistically significant differences were also observed in a study by Dalli'Magro,[33] in which they observed a decrease in hardness after a 3-mm thickness, in all the groups, when compared with the hardness on the top.

CONCLUSION

Based on the methodology used and the results obtained, it can be concluded that the hardness was higher with photoactivation of 40 seconds (pulse--delay technique) and for the conventional and soft start techniques (20 seconds) there was no difference in terms of hardness. Therefore, hardness is not influenced by the technique, but rather, by the polymerization time.

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