

Influence of two methods of additional activation on composite resins surface hardness

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Abstract

Aim: To evaluate the influence of two methods of additional activation on the surface hardness of composite resins. **Methods:** Two types of composites were tested: Filtek P60 and Filtek P350. For each material, 48 specimens were prepared and divided into four groups: Group 1 (control) - conventional activation, using a halogen light for 40 s; Group 2 - conventional activation and additional activation with a halogen lamp for 60 s; Group 3 - conventional activation and additional activation with an autoclave at 127°C for 6 min at 1.7 kg/cm³ pressure; and Group 4 - conventional activation and additional activation with an autoclave at 134 °C for 15 min at 2.1 kg /cm³ pressure. The use of autoclave has been suggested for being a standard equipment at dental offices, and thus, even at locations far from dental laboratories, it would be possible to have simple techniques that allow access to indirect restorations at lower costs. Data obtained in the study were analyzed statistically by analysis of variance followed by Tukey's test at a 5% level of significance. **Results:** For Z350, there was a significant increase in hardness for all groups of additional activation (Groups 2, 3 and 4), compared with the control group. For P60, a significant increase in surface hardness was found compared with the control group for the groups that used additional activation with an autoclave (Groups 3 and 4). **Conclusions:** Additional activation with an autoclave increased the surface hardness of the tested resins to a greater degree than additional activation with a halogen light.

Keywords: polymerization, composite resins, hardness.

Introduction

Technological evolution in dentistry is driven by the constant attempt to improve materials and techniques in line with the market's demands. Such advances mean that it is increasingly possible for clinicians to produce outcomes that combine function with good aesthetics. At the moment, the most widely used restorative materials in dentistry are composite resins, mainly because they adhere well to the tooth structure, have suitable mechanical properties and are available in a wide range of shades and translucencies that produce good aesthetic results¹.

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Since their introduction as restorative materials for posterior teeth, composite resins have significantly improved in terms of their physical and mechanical characteristics. The composition of their organic and inorganic matrix has changed, and there are currently various types of resins that can be used in posterior teeth, such as microhybrid resins and composites with nanoparticles²⁻³. Composite resins are usually recommended for direct restorations in posterior teeth when cavities are small and medium in size. To restore large cavities and extensively destroyed teeth, dentists generally opt for amalgam fillings or indirect laboratory-made resin, porcelain or metallic restorations⁴⁻⁵. Amalgam can be used for direct restorations, but it is limited by its unaesthetic outcome. The main limiting factor of indirect restorations is their high cost.

One viable alternative (published in the norms of the manufacturers of resins indicated for use in posterior teeth) is the use of these resins in indirect restorations such as crowns, veneers, inlays, onlays and so on. However, these indirect restorations require additional costs and special equipment for activation. Various studies have shown that by means of simple technical changes, such as additional activation, direct-use resins can achieve mechanical properties that are similar to laboratory-made restorations⁶⁻⁸.

Different methods of extraoral additional activation, including activation by light, dry heat and autoclaving, were proposed in order to improve physical and mechanical properties, and enable the use of direct-use composite resins in indirect restorations. The purpose of this additional treatment is to broaden the indication of resins and the clinical longevity of restorations⁷⁻⁹. However, there are few studies that investigate which of these are the best method. The autoclave has been suggested because it is a standard equipment at dental offices. Therefore, even at locations far from dental laboratories, it would be possible to develop simple techniques that could allow access to indirect restorations at lower costs.

The surface hardness of composite resins depends mainly on their microstructure and composition, but there is a correlation with the degree of conversion of monomers^{2,10}. The increase in hardness is related to a higher degree of conversion and improved mechanical properties of polymeric materials. Thus, the study of the microhardness of materials consists of an indirect method to evaluate the effect of different treatments on the properties of dental composites¹⁰⁻¹². The Vickers hardness test is commonly used to investigate improvement in material's mechanical properties^{2,13-16}. Using this test as a parameter, the aim of this study was to evaluate the microhardness of two direct resin composites after additional activation with light or using an autoclave.

Material and methods

Specimen preparation

To obtain the resin specimens, a circular split-ring matrix with an outer diameter of 2 cm and an inner diameter of 1 cm was used. It was held in position by a circular metal

matrix with a 2-cm diameter perforation and 3-cm outer diameter (Figure 1 and 2).



Fig. 1: Circular split-ring metal matrices.



Fig. 2: Metal matrices assembled.

The composite resins used were: 1) Filtek P60 (3M ESPE, St. Paul, MN, USA) – a hybrid composite, for direct and indirect restorations in posterior teeth and 2) Filtek Z350 (3M ESPE) – a nanoparticulate composite, for direct anterior and posterior restorations and for indirect inlay, onlay and veneer restorations (Table 1). P60 was used in this study to an extensively researched material, with a large number of published papers, and because it is a material with an indication for use in posterior teeth, region usually requires partial indirect restorations. Z350 composite was selected due to its recent launch in the dental market and for representing the newest class of composite resins with an indication for anterior and posterior teeth.

The matrix was placed on a glass plate and the inner space was filled with one of the studied composite resins in a single increment. A polyester strip was placed on the top surface and activated for 60 s using a Kondertech activation device, model CL-K200 (Kondertech, São Carlos, SP, Brazil) having a light intensity of 500 mW/cm². The light intensity was checked with a digital radiometer (Dabi-Atlante, São Paulo, SP, Brazil). Ninety-six specimens were prepared, 48 for each restorative material, divided into the following groups: Group 1 (control) – conventional activation, using a

Table 1: Specifications of composite resins used

	Material	Classification	Indication Composition	Manufacturer
P60	Microhybrid	Posterior	BisGMA, UDMA, BisEMA	3M ESPE
Z350	Nanoparticulate	Anterior and posterior	BisGMA,UDMA, TEGDMA, BisEMA	3M ESPE

halogen light for 60 s; Group 2 – conventional activation and additional activation with a halogen light for an additional 60 s; Group 3 – conventional activation and additional activation with an autoclave at 127°C for 6 min and 1.7 kg/cm³ pressure; Group 4 – conventional activation and additional activation with an autoclave at 134°C for 15 mins at 2.1 kg/cm³ pressure.

Hardness Test

After they had been prepared, the specimens were stored in distilled water in a bacteriological oven at 37°C for 7 days in a light proof container. The Vickers microhardness tests were carried out with the aid of a MMT-3 Microhardness Tester (Buehler, Lake Bluff, IL, USA) under a load of 50 gF for 30 s. Three microhardness impressions were carried out per sample, one in the center and two at the periphery, as follows: traced an imaginary line dividing the sample in half, a central impression was made and the other between the center and right and left edges.

Statistical analysis

The data obtained from the means were subjected to analysis of variance (ANOVA) followed by Tukey's test at a 5% level of significance.

Results

The tested resins had different means of hardness, according to the type of material and the type of treatment of the samples, as shown in Table 2.

Discussion

The results of this study indicate that additional activation methods significantly improved the microhardness of the composite resins evaluated. These results are consistent with the study of Dickerson and Hastings¹⁷ (1995), which reported polymerization rates of approximately 50% to 60% for self-activated resins and 55% to 65% for photoactivated resins, and reported that these resins reached a degree of

conversion of 80-85% when subjected to a temperature of 125°C.

The use of an autoclave for additional activation (groups 3 and 4) significantly increased the microhardness compared with the control group, for both materials tested, probably because the autoclave generates greater amounts of energy in the form of heat and pressure, which increases the conversion rate. Other studies that evaluated the influence of heat treatment also found improvements in the mechanical properties of materials, which may be related to a reduction in the amount of residual double carbon bonds in the polymer¹⁸⁻²¹.

In another study of the behavior of resins and additional activation, Bagis and Rueggeberg⁶ (2000) reported that heat treatment increases the conversion rate, and this increase would not be possible if it was only photoactivated. Table 2 confirms this statement, and even the use of additional photoactivation time did not result in a greater hardness of Filtek P60.

Perhaps this difference is due to the composition of the resins. Trujillo, Newman and Stansbury²² (2004) reported that exposure of composites to additional heat treatment, limited to a biologically compatible time period, significantly affects the kinetics of activation, and increases the conversion rate of resins and improves their properties.

The increase in hardness can be explained by the fact that the temperatures used in the heat treatment were close to the glass transition temperature, which increases the kinetic energy of the resinous monomers and the quantity of free radicals. The greater mobility within the polymer chain enables new reactions of the activated radicals and a greater number of crosslinks in the organic matrix. The continuation of the activation process leads to greater stability and hardness of the composite^{7-8,21,23}.

With Filtek Z350 resin, as well as the additional conversion using heat and pressure, the longer light exposure (exceeding the amount recommended by the manufacturer) affected significantly the surface hardness. This fact suggests that, although the microhardness of this material is suitable for dental needs, it is possible to produce a higher conversion of residual monomers through greater exposure to halogen

Table 2: Mean and standard deviation of Vickers hardness of experimental groups according to the type of composite resin.

	Filtek P60		FiltekZ350	
	Mean	Standard Deviation	Mean	Standard Deviation
Group 1	49.9 (A)	3.1	40.2 (a)	3.6
Group 2	51.6 (A)	3.9	44.7 (b)	4.1
Group 3	55.6 (B)	1.8	46.2 (bc)	2.0
Group 4	56.1 (B)	3.6	48.4 (c)	2.9

Different letters indicate a statistically significant difference ($\alpha=0.05$) according to the t-Test.

light and thereby increase the restoration's durability²⁴.

Another advantage is the greater biocompatibility of the material. Increased activation results in a significant decrease in the amount of non-activated monomers and, consequently, lower levels of leachable materials that promote oral cytotoxicity⁶. The use of heat also causes a similar effect: it results in more monomers linked to the polymer chain and some of the unreacted monomers are volatilized during the heating process²⁵.

The effect of additional activation and the different microhardness values of the resinous materials depend mainly on their composition. BIS-GMA has a low degree of conversion because of its characteristics of high molecular weight, high viscosity and low flexibility. The addition of diluent monomers with higher flexibility, such as EGDMA or TEGDMA, enhances Bis-GMA's mobility and its polymerization conversion rate. Another alternative to Bis-GMA is the monomer UDMA, which has a molecular weight similar to Bis-GMA, but a lower viscosity^{18,26}.

It has been shown that the monomer TEGDMA creates a thicker polymer chain, but it is the most flexible and has a greater rate of water absorption. Bis-GMA forms a more rigid chain and absorbs less water; however, it absorbs more water than the UDMA/Bis-EMA combination. Hydrolysis of intermolecular bonds weakens the polymer. In UDMA-based composites, hydrogen bonds increase the conversion rate and improve mechanical properties. When TEGDMA is replaced by UDMA and/or BIS-EMA (during co-polymerization with BIS-GMA), the absorption of water is decreased. Such characteristics influence the conversion rate and the mechanical properties of composites²⁷⁻²⁸. The abovementioned information explains the different behaviors between the Z350 and P60 composites after additional activation: Z350 contains BIS-GMA, UDMA, TEGDMA and BIS-EMA, while P60 contains BIS-GMA, UDMA and BIS-EMA.

From the above discussion, it is possible to state that additional activation by means of thermal treatments improved the hardness of the composite resins tested, regardless of their composition. The use of an autoclave as an additional method of activation is very effective and provides better results. By using a routinely found equipment at dental offices, such as autoclave, it is possible to develop a simple, low cost technique, especially in cases of difficult access to special equipment for the production of laboratory restorations.

It can be concluded that the additional activation using an autoclave increased the surface hardness of the tested resins more than additional activation with halogen light.

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