Influence of additional polymerization in the microhardness of direct composite resins

Influência da polimerização adicional na microdureza de resinas compostas diretas

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Abstract

Objective: To evaluate different methods of additional polymerization in the Vickers' microhardness of three composite resins. **Material and Methods:** With assistance of a metallic matrix (3 x 4mm), 30 specimens (sps) of each direct resin composite (drc) were prepared, entitled: Filtek Z250(3M ESPE, Saint Paul/MN-USA)(Gr1), W3D(Wilcos, Petrópolis/RJ-Brazil)(Gr2), and EstheticX (Dentsply/York/PA-USA)(Gr3). All sps were photopolimerized, by the incremental technique, during 40 seconds (Gr1 - n=10: control). After that, 20 samples of each group were divided and half of the samples (n=10) were submitted to the additional polymerization during 15 minutes to 127ºC in autoclave (Gr2) and the other half in stove (Gr3). After all sps were stored in distilled water (37ºC/24 hours). The microhardness was accomplished in digital machine (Future-Tech, FM 700 Model), with 50kg of load. The data obtained (Kg/mm^2) were analyzed utilizing the ANOVA Test (2 factors) and Tukey test ($p<0.05$). **Results:** The values of mean ± standard deviations obtained were: Gr1a(84,2±5,1), Gr1b (71,6±6,7), Gr1c (89,6±7,4), Gr2a (101,1±6,0), Gr2b (96,4±9,0), Gr2c (112,6±7,3), Gr3a (74,0±6,5), Gr3b (66,0±12,7) e Gr3c (81,7±6,5). The polymerization in stove produced a higher microhardness than the other kinds of polymerization ($p<0.05$) and the microhardness of the resins statistically differed among them ($p<0.05$). **Conclusion:** The additional polymerization of the Filtek Z250 in the stove produced microhardness statistically higher than the others.

Uniterms

Composite resins; hardness; polymerization.

Introduction

Restorations in composite resins manufactured by the indirect technique have been used in cases of very extensive cavities, once they allow a better outline, a more refined polish, reduction of the polymerization contraction effect and smaller marginal infiltration, when compared to the use of composite resins made by the direct technique[1]. According to Lacy[2], although the composite resins of direct use have excellent mechanical and optical properties, the problems inherent to the direct technique as polymerization contraction and sensitivity of technique, the use of this system to posterior reconstructions should be limited to small cavities.

In addition, the manufacturing of composite resin restorations by the indirect technique allows the obtainment of a higher conversion degree of these resins by the possibility of making the photopolymerization on all surfaces of the restoration. Thus, there are no areas with lower quantity of light, as it happens frequently in direct restorations where the quantity of residual monomer varies from 25% to 60% of non-reacted material or with deficient crossed reactions[3].

It is known that the reduction of the conversion degree can affect the biocompatibility of the resins because of the excess of the non-reacted monomers besides it is directly related to the commitment of the mechanical properties of the resin materials[4,5]. In such case, complemental methods of polymerization have been suggested to optimize the polymerization degree of the composite resins, such as: heat, pressure and heat/pressure.

Great part of the studies that evaluated the effect of the complemental polymerization, as much by heat as by pressure, used composite resins that present chemical composition differents of the lately commercialized resins. With the evolution of the resin materials, resins made with UDMA (urethane dimethacrylate) and Bis-EMA(6)[1] (Bisphenol A polyethylene glycol diether dimethacrylate) were produced, and they presented a molecular weight bigger than the conventional Bis-GMA and TEGDMA. Consequently, they present smaller quantity of double connections by unity of weight, that make this material present higher conversion degree when exposed to light, lower contraction of polymerization and less water absorption.

Thus, the present study proposed to evaluate the influence of the complementary polymerization with heat and heat/pressure in the Vickers's microhardness of the direct composite resins, where the polymerization with light was the control group.

Material and method

With assistance of a metallic matrix (5mm of diameter and 2mm of thickness), 30 sps of each drc were manufactured: Gr1- Filtek Z250 (3M ESPE/ Saint Paul/MN-USA); Gr2- W3D (Wilcos/ Petrópolis/RJ-Brazil) and Gr3- Esthet-X (Dentsply/ York/PA-USA). The material was inserted in the matrix with a metallic spatula, and photopolimerized by the incremental technique (1,5mm of thickness), during 40s in each increment, with a light cure equipment (XL Curing 3000 – 3M Dental Products/USA), with 500mW/cm^2 of light intensity. After the photopolymerization, all the sps were marked in the inferior edge, to identify the upper surface.

For each type of composite resin, 10 sps were submitted to only the photopolymerization (a), 10 passed by complemental polymerization with heat/pressure (Autoclave – Cristófoli/Campo Mourão-PR/Brazil) (b) and 10 passed by complemental polymerization with heat (Stove – Odontobrás/ Ribeirão Preto-SP/ Brazil) (c).

The sps of the groups Gr1b, Gr2b and Gr3b were taken to the autoclave to 127ºC for 15min. On the other hand, the groups Gr1c, Gr2c and Gr3c were submitted only to heat, where they were kept in the temperature of 127ºC during 15 min. Following, all the samples were stored in distilled water for 24 hours to 37°C.

With assistance of a silicon matrix, the sps were included in acrylic resin activated chemically, with the side, where the microhardness reading would be accomplished, facing the ground. This procedure was accomplished with the purpose to facilitate the polish and the surface reading of the sps. The sps were regularized in politrix (Strues, Model DP 10, Panambra Ind. & Tec. S.A., São Paulo-SP/Brazil), using the water sandpapers nº 600, 800, 1200 and 2000.

After, the sps were taken to the microhardness (FM 700 – Future-Tech, Equilam, Tokyo/Japan) and submitted to a load of 50kg, for analysis of the microhardness. Each sample was submitted to three readings, and this way it was obtained the penetration value, which was converted to hardness values according to a pre established list.

The data were analyzed statistically by the tests ANOVA (2 factors) and Tukey, with a significance level of 5%.

Results

The means and standard deviations of the microhardness data (Kg/mm^2) are presented in the Table 1 and graphically represented in the Figure 1.

By the ANOVA Test it was verified that the main factors resin and polymerization presented statistical significance ($p<0.05$). It was also observed that there wasn't interaction effect between these two variables. Thus, the relationship between these variables are not dependent.

By the Tukey Test (5%) it was verified that the resins differed between themselves ($p<0.05$). The polymerization in the stove (94.65±14.95Kg/mm^2) presented microhardness values higher than the other polymerizations: light (86.41±12.72Kg/mm^2) and autoclave (77.94±16.45Kg/mm^2).

When the nine mean values of the experimental conditions (Table 2) are compared, through the multiple comparison test of the Tukey Test (5%), it verifies that the condition of higher microhardness was observed in the composite resin Filtek Z250, with secondary polymerization in stove ($p<0.05$).

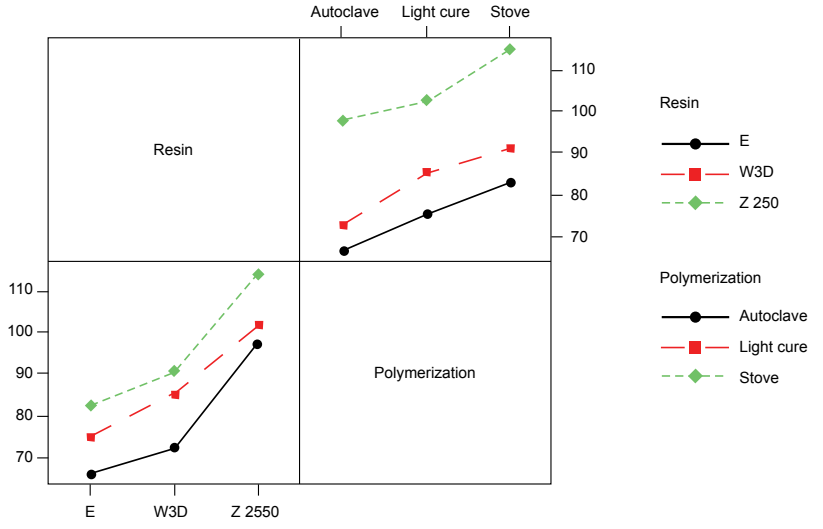

Figure 1 – Means graphic of microhardness values according to the experimental conditions established by the main factors: resin and polymerization.

Table 1 – Mean (± standard deviation) of the microhardness data (values in Kg/mm^2) obtained in 10 sps, according to experimental conditions

Resin	Polymerization		
	Light (Mean ± DP) (Kg/mm^2)	Autoclave (Mean ± DP) (Kg/mm^2)	Stove (Mean ± DP) (Kg/mm^2)
Filtek Z250	101.1 ± 6	96.4 ± 9	112.6 ± 7.3
W3D	84.2 ± 5.1	71.6 ± 6.7	89.6 ± 7.4
Esthet-X	74 ± 6.5	66 ± 12.7	81.7 ± 6.5

Table 2 – Arrangement of groups of same performance, after the Turkey Test (5%) for the nine experimental conditions of microhardness

Resin	Polymerization	Mean (Kg/mm²)	Homogeneous Groups *						
Filtek Z250	Stove	112.6	A						
Filtek Z250	Light	101.1		B					
Filtek Z250	Autoclave	96.4		B	C				
W3D	Stove	89.6			C	D			
W3D	Light	84.2				D	E		
W3D	Autoclave	71.6						F	G
Esthet-X	Stove	81.7				D	E	F	
Esthet-X	Light	74					E	F	G
Esthet-X	Autoclave	66							G

*mean values followed by the same letter don't differ themselves statistically

Discussion

The accomplishment of indirect restorations (inlay/onlay), using drcs, has been proposed with the goal of minimizing the tensile caused on the bond line when the resin is photopolimerized , allowing a better devolution of the shape lost by the dental element as well[6]. Thus, many authors[7-9] suggest the use of additional methods of polymerization in these materials, such as light, heat and/or pressure, in a try to improve the mechanical and aesthetical properties of the direct resins, and also amplifying the indication of these materials for indirect use.

Baush *et al.*[10] mentioned in their study that temperatures above 60ºC can improve the resins mechanical properties, once the samples thermic analysis revealed that between 60ºC and 70ºC an endothermic reaction on the free radicals happened, initiating the development of additional crossed chains, generating materials more strength.

However, in another study made by Wendt[11], the author evaluated the influence of the dry heat, as a secondary polymerization method, on the mechanical properties of three direct composite resins (Profile TLC – S.S. White; P 30 – 3M; Occlusin – Coe), changing the temperature (60º, 100º, 125º, 150º, 175º and 200ºC). After the analysis of the results, the authors concluded that the dry heat (125º C) improved the mechanical properties of resistance to wastage, hardness and color stability, of the composite resins. Additionally, temperatures above 125ºC, showed stability in the microhardness values. In a second study of the same author[12], it was varied the time of complemental polymerization in direct composite resins, using dry heat to 125ºC, changing just the time of secondary polymerization (2,5; 7,5, 10 and 15 minutes). The results demonstrated that independent of time, the secondary polymerization increased the strength to microhardness of the direct composite resins.

Thus, based in the studies of Wendt[11-12] and in recent studies of Soares et al.[9], we standardized the temperature and the time of secondary polymerization of the direct composite resins in our study to 127º C, as much in stove as in autoclave, during 15 minutes.

In our study, it was observed that the secondary polymerization of the drcs in stove (Gr1c-112.6±7.3, Gr2c-89.6±7.4; and Gr3c-81.7±6.5Kg/mm²) provided higher values of microhardness compared to other groups, being statistically higher than the groups polymerized in autoclave (Gr1b-96.4±9, Gr2b-71.6±6.7; and Gr3b- 66±12.7Kg/mm²), but when it was compared to the control groups (photopolimerized), it was significantly higher just for the Filtek Z250 resin.

Corroborating with our research, Adabo et al.[1], studied the effect of different techniques of polymerization (light, pressure/heat and light/heat) in the microhardness of the direct and indirect composite resins, used for *inlay* restorations: P-50 (3M/ESPE), Extilux posterior CVS (Kulzer) and Isosit (Ivolclar/Vivadent), this utilized as control. After the data analysis, the

authors observed that the complemental polymerization with light and heat, created higher microhardness values, equal among them, and significantly higher to the other conditions. Wendt and Leinfelder[13], evaluated the effect of the dry heat (125ºC, 10min) in the mechanical properties of microhardness and strength to wastage of a composite resin (Occlusin/Coe), and they also verified that this kind of complemental polymerization created an essential improvement of these properties.

For Baush et al.[10], among the complemental methods, it is detached the application of heat, once that in lower temperature, the polymerization is processed slowly until the available peroxide groups are consumed, reducing the possibility of arrangement of extra crossed bonds, resulting in a material with long chains, with high mobility and heterogeneous structure. With the use of heat, the supply of exceed energy acts on the free radicals making that the arrangement of crossed chains predominate, creating more strength materials.

On the other hand, the studies made by Burke et al.[14] and Covington and McBride[7], the complemental polymerization of the drcs in autoclave (heat and pressure) improved the physical and mechanical properties of these materials.

In this study, the additional polymerization in autoclave reduced the microhardness values (Table 1). According to Adabo et al.[1], this occurs because the water under pressure (autoclave), makes the resin absorbs a great quantity of liquid, reducing its superficial hardness, possibly annulling the effect of the heat use or dissolving the monomers, reducing the effects of the complemental polymerization .

The higher exposition of the resin to the water, higher the swelling in the net of the crossed bonds of the polymers that reduce the frictional strengths among the polymer chains. It is possible that at the same time, the water causes hydrolyze in the interface matrix/filler and/or fissures on the polymer matrix, helping to reduce the resin properties. Once the polymer nets are saturated with water, the resin structure become steady, and it does not happen any additional reduction in the polymer properties. This limited reduction of the mechanical properties of the composite resins provides the evidence that the additional degradation, such as hydrolyze of the interface matrix/load or fissure on the polymer matrix, does not develop to a crescent degradation in case of the resin keeps humid. Thus, most authors[8,16-18] agree that the weakness of the polymer due to the water absorption is the main factor responsible by the decrease of their mechanical properties.

On the other hand, the microhardness of the composite resins does not depend just on the extension of the polymerization degree of the polymeric matrix, but it also depends on the quantity of filler of each resin (Chadwick et al.[19]. At the present study, the microhardness of the resin Filtek Z250 was the highest of the others resins, and it also presented the greatest number of load on its composition (60% vol.). Additionally, Reich et al.[20] related that the mechanical properties of indirect composites are highly influenced by their filler particle size distribution, the kind of filler particles, shape, and its silanization to the matrix.

Conclusion

Based in the results it is lawful to conclude that the additional polymerization of the resin Filtek Z250 with heat in the stove, created microhardness statistically higher than in the other groups studied.

Resumo

Objetivo: *Frente à possibilidade de se utilizar resinas compostas diretas (rcds) para procedimentos restauradores indiretos, o presente estudo propôs avaliar diferentes métodos de polimerização adicional na microdureza Vickers' de três resinas compostas de uso direto.* ***Materiais e Métodos:*** *Com o auxílio de uma matriz metálica (3,0 x 4,0mm), foram confeccionados 30 corpos-de-prova de cada resina denominadas: W3D(Wilcos/Brasil)(Gr1), Z250(3M ESPE/EUA) (Gr2) e Esthetic X(Dentisply/EUA)(Gr3). Todos os espécimes (sps) foram fotopolimerizados, pela técnica incremental, durante 40 segundos (Gr1 - n=10: controle). Em seguida, 20 amostras de cada grupo foram aleatoriamente divididas e metade das amostras (n=10) submetidas à polimerização adicional durante 15 minutos a 127ºC em autoclave (Gr2) e a outra parte em estufa (Gr3). Após todos os sps foram armazenados em água destilada a 37ºC durante 24 horas. O teste de microdureza foi realizado em microdurômetro (Future-Tech, Modelo FM 700), com força de 50 kg. Os dados obtidos (Kg/mm²) foram analisados utilizando o teste ANOVA (2 fatores) e o teste t (Tukey) (p<0.05).* ***Resultados:*** *Os valores de médias±desvios padrões obtidos foram: Gr1a(84,2±5,1), Gr1b (71,6±6,7), Gr1c (89,6±7,4), Gr2a (101,1±6,0), Gr2b (96,4±9,0), Gr2c (112,6±7,3), Gr3a (74,0±6,5), Gr3b (66,0±12,7) e Gr3c (81,7±6,5). Observou-se que a polimerização em estufa gerou maior microdureza que as demais polimerizações (p<0,05) e que a microdureza das resinas diferiu estatisticamente entre si (p<0,05).* ***Conclusão:*** *Dessa forma, é lícito concluir que a polimerização adicional da Z250 na estufa gerou microdureza estatisticamente superior aos demais grupos.*

Unitermos

Resinas compostas; dureza; polimerização.

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