EFFECT OF POST-CURING TREATMENT ON MECHANICAL PROPERTIES OF COMPOSITE RESINS

Verónica A. Almeida-Chetti, Ricardo L. Macchi, María E. Iglesias

Dental Materials Department. School of Dentistry. University of Buenos Aires. Argentina.

ABSTRACT

The aim of this study is to assess the effect of additional curing procedures on the flexural strength and modulus of elasticity of indirect and direct composite materials. Twenty-four rectangular prism-shaped 2 mm x 2 mm x 25 mm samples of Belleglass, Premisa (Kerr), Adoro and Heliomolar (Ivoclar Vivadent) were prepared. Each composite was packed in an ad-hoc stainless steel device with a Teflon® instrument. A mylar strip and a glass slab were placed on top to obtain a flat surface. Polymerization was activated for 20 seconds with a halogen unit (Astralis 10, Ivoclar - Vivadent) with soft start regime and an output with a 350 to 1200 mw/cm² range at four different points according to the diameter of the end of the guide.

The specimens obtained were then randomly divided into two different groups: with and without additional treatment. In the group with additional treatment, the samples adorro were submitted to 25 minutes in Lumamat 100 (Ivoclar Vivadent) and the rest to 20 minutes in BelleGlass HP (Kerr). After the curing procedures, all samples were treated with sandpapers of decreasing grain size under water flow, and stored in distilled water for 24 h. Flexural strength was measured according to the ISO 4049[20] recommendations and elastic modulus was determined following the procedures of ANSI/ADA standard No. 27.

Statistical differences were found among the different materials and curing procedures employed (P< 0.01). The elastic modulus was significantly higher after the additional curing treatment for all materials except Premisa. Further work is needed to determine the association between the actual monomers present in the matrix and the effect of additional curing processes on the mechanical properties of both direct and indirect composites, and its clinical relevance.

Key words: *Polymerization; Composite resins; Flexural strength; Elastic modulus*

EFECTO DE UN PROCEDIMIENTO DE CURADO ADICIONAL EN LAS PROPIEDADES MECÁNICAS DE COMPOSITES

RESUMEN

El objetivo de este trabajo fue evaluar el efecto del curado adicional sobre la resistencia flexural y el módulo elástico de composites directos e indirectos. Se obtuvieron veinticuatro probetas prismáticas rectangulares de 2mm de lado y 25mm de largo, con los composites Belleglass, Premisa (Kerr), Adoro y Heliomolar (Ivoclar Vivadent). Cada uno de los materiales fue insertado con un instrumento de Teflón® en un dispositivo ad-hoc de acero inoxidable, sobre el material se colocó una cinta de acetato y una placa de vidrio, y luego se ejerció presión uniforme sobre el conjunto con el fin de obtener una superficie lisa y plana.

En todos los casos, la superficie de las probetas fue dividida en cuatro secciones de igual largo al diámetro de salida de la fibra óptica para asegurar la llegada de energía de activación a toda la masa de material; en cada una de ellas se activó la polimerización durante 20 segundos, con una lámpara halógena (Astralis 10, Ivoclar - Vivadent). Las muestras obtenidas fueron asignadas al azar a dos grupos: con y sin curado adicional. En el grupo con curado adicional, los especímenes de Adoro recibieron un curado adicional de 25 minutos en el dispositivo Lumamat 100 (Ivoclar Vivadent), mientras que el resto de los materiales fueron sometidos a 20 minutos en BelleGlass HP (Kerr). Luego de dichos procedimientos, se eliminaron los excesos con papel abrasivo de granulometría creciente bajo flujo de agua y se almacenaron en agua destilada a 37º C durante 24 horas. La resistencia flexural fue valorada en base a las recomendaciones de la norma ISO 4049, mientras que el módulo elástico fue determinado según la norma Nº 27 (ANSI/ADA).

Se encontraron diferencias significativas entre los diferentes materiales y procedimientos de curado empleados (P<0,01). El módulo elástico fue significativamente más elevado luego del procedimiento de curado adicional en todos los materiales, excepto en Premisa. Se necesitan más estudios para determinar la relación de los monómeros presentes en la matriz con el efecto de los procedimientos de curado adicional sobre las propiedades mecánicas de composites directos e indirectos y su relevancia clínica.

Palabras clave: *Polimerización; Resinas compuestas; Resistencia flexural; Módulo elástico*

INTRODUCTION

One of the major goals of restorative dentistry is to develop restorative procedures that can succeed regardless of the ability of the operator and the hazards of clinical management, in order to achieve more predictable success.

The indirect technique for composite restorations was first conceived as a way to optimize the mechanical and chemical properties of polymerized materials and to overcome some major problems inherent to direct techniques, such as the difficulty to achieve marginal adaptation and interproximal anatomy as well as the adverse effects of polymerization shrinkage, including both volumetric changes and the development of stress [1].

The materials, initially marketed as laboratory composites, were first introduced in the 1980s as an alternative to the other esthetic materials available for indirect restorations: dental ceramics and acrylic resin. Although indirect composites generally contain the same components as direct composites, the former are marketed in association to extra-oral curing devices which offer different combinations of light and/or pressure or heat. These units are intended to provide post-curing treatment in order to obtain an optimized polymer and thus an improved restoration [1].

Although there is no solid evidence regarding the extent to which these post-curing treatments significantly improve the properties of the resulting polymers, their effect is believed to be independent of the composition of the composite treated [2-9].

Some studies found higher values of flexural strength (FS) [10,11] and elastic modulus (E)[4,9,12] in resin composites commercialized as direct materials but nevertheless submitted to post-curing treatments.

Although the higher values for mechanical properties which have been found in some studies may be due to an increase in the degree of conversion[13], the multiple variables involved in the different monomers present in each brand formulation and the fact that these brands promote the usage of their own post-curing devices make it extremely difficult to ascertain the effect produced purely by the treatments. [4,6] This may also explain why some studies have found evidence of improvement in mechanical properties regardless of the brand or the post-curing unit employed [2-10,12,14-16], whereas others have found no significant difference [2,12,17,18].

In terms of the temperature reached by the devices and its effect on the result obtained, many studies have found that post-cure treatments with temperatures over 100° C were associated with improved physical and mechanical properties [2-9] due to an increased mobility of the monomers resulting in a higher degree of conversion of the matrix [12,19,20].

The controlled atmosphere inside the curing unit [8], especially under high nitrogen pressure,[9,17] was also found to result in fewer air bubbles trapped within the mass. Absence of available oxygen to create inhibited layers produced a more dense resin matrix with fewer residual monomers and thus, better mechanical properties [21].

The aim of this study is to assess the effect of additional curing procedures on the flexural strength and modulus of elasticity of indirect and direct composite materials.

MATERIALS AND METHODS

The different materials and devices employed are listed in Tables 1 and 2 respectively. Flexural strength was assessed according to the ISO 4049 [22] recommendations and the elastic modulus was determined following the procedures of ANSI/ADA standard No. 27 [23].

Twenty-four rectangular prism-shaped 2 mm x 2 mm x 25 mm samples of the indirect composites Belleglass (Kerr-457996) and Adoro (Ivoclar Vivadent-G12179) and the direct materials Premisa (Kerr-417541) and Heliomolar (Ivoclar Vivadent-D59447) were prepared. Each composite was packed in an *ad-hoc* stainless steel device with a Teflon® instrument. A mylar strip and a glass slab were placed on top of the material in order to distribute the pressure uniformly and obtain a flat surface. Polymerization was activated for 20 seconds with a halogen unit (Astralis 10, Ivoclar - Vivadent) with a soft start regime and an output with a 350 to 1200 mw/cm^2 range at four different points determined according to the diameter of the end of the guide, to ensure that sufficient energy reached the whole mass.

The specimens were randomly divided into two groups: with and without additional treatment. In the group with additional treatment, the samples of those materials originally intended for post-curing treatments were submitted to the type of treatment and device recommended by the manufacturer: 25 minutes in Lumamat 100 (Ivoclar Vivadent) for Adoro (Ivoclar Vivadent) specimens, and 20 minutes in BelleGlass HP (Kerr) for BelleGlass (Kerr) specimens. A twenty-minute post-cure treatment in Belleglass HP was employed for both direct composites.

After the curing procedures, all samples were treated with sandpapers of decreasing grain size under water flow, and stored in distilled water for 24 h.

Mechanical tests

To assess flexural strength and elastic modulus, a three-point bending test under compressive load was carried out in a universal testing machine (Instron 1100 Mas. USA) at a cross head speed of 1 mm/min. The specimens were measured with a

0.1 mm accuracy caliper (Mauser, Germany) and placed on two cylindrical supports (2 mm in diameter) with a distance of 20 mm between centers. Flexural strength (σ) and elastic modulus (E) were calculated with the following equations:

$$\sigma \text{ (MPa)} = \frac{3\,Fl}{2\,bh^2}$$

$$E \text{ (MPa)} = \frac{F_1 l^3}{4\,bh^3 d}$$

F: maximum load (N)
l: distance between supports (mm)
b: width of the specimen (mm)
h: height of the specimen (mm)
F_1: a defined load increase under the proportional limit (N)
b: deflection of the specimen produced by F_1 (mm)

The data were analyzed by ANOVA, and Tukey's test was applied for multiple comparisons (P<0.05).

RESULTS

Statistical differences were found among the different materials and curing procedures employed (P<0.01) (Tables 1 and 2).

Flexural strength of indirect and direct composites, each cured with the protocol recommended by the manufacturer (photoactivation for Premisa and Heliomolar, and photoactivation plus additional curing treatment for Belleglass and Adoro), was compared by Tukey's test. Belleglass had the highest statistically significant FS (P<0.01), and there was no significant difference among the rest of composites tested (Fig 1).

Additional curing procedures entailed a significant increase in FS for all materials (P<0.01). When the FS of the different composites was compared after additional cure, statistical differences were found among the materials (P<0.01), except between Belleglass and Heliomolar, and between Heliomolar and Premisa (Tukey).

The elastic modulus was different according to the curing protocol employed; significant statistical differences were found among materials and treatments. The elastic modulus of indirect and direct composites each cured with the protocol recommended by the manufacturer (photocure for Premisa and Heliomolar, and photoactivation plus additional curing treatment for Belleglass y Adoro), was compared by Tukey's test. Statistical differences were found among the study composites (P<0.05) with the exception of Adoro and Heliomolar. (Fig. 2)

Table 1: Flexural strength - ANOVA.

Source	Sum of squares	df	Mean	F	p
Composite	7212 . 554	3	2404 . 185	23 . 207	.000
Additional Curing	26735 . 024	1	26735 . 024	258 . 067	.000
Interaction	7823 . 435	3	2607 . 812	25 . 173	.000
Error	4143 . 896	40	103 . 597		
Total	45914 . 909	47			

Table 2: Modulus of elasticity - ANOVA.

Source	Sum of squares	df	Mean	F	p
Composite	66 . 480	3	22 . 160	87 . 645	.000
Additional Curing	140 . 220	1	140 . 220	554 . 578	.000
Interaction	100 . 995	3	33 . 665	133 . 147	.000
Error	10 . 114	40	. 253		
Total	317 . 809	47			

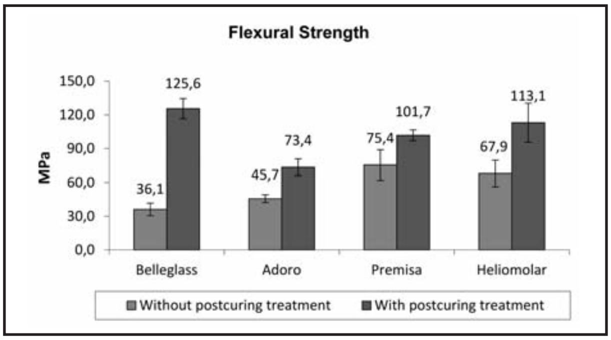

Fig.1: Flexural strength of direct and indirect composites without and with post-curing treatment.

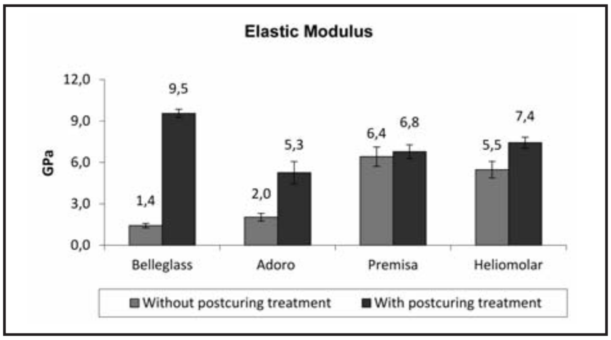

Fig.2: Modulus of elasticity of direct and indirect composites without and with post-curing treatment.

The elastic modulus was significantly higher after the additional curing treatment in all materials except Premisa. When the E of the different composites was compared after additional cure, statistical differences were found among the materials ($P<0.01$), except between Premisa and Heliomolar (Tukey). Fig. 2

DISCUSSION

Composite resin properties are related to the composition of the organic matrix, the quantity and quality of the filler, and the way polymerization is activated.

In order to improve the physical and mechanical properties of indirect composites (second generation laboratory composites), post-curing treatments are employed using different devices combining visible light, heat and/or pressure. Nevertheless, since the inception of the first indirect composites, differing results have been reported regarding their benefits compared to those of direct materials.

Most indirect composites differ from direct composites in the kind of indicator included in their composition; e.g., materials designed for treatment with devices that employ heat can include thermosensitive initiators in addition to photosensitive initiators. However, direct composites do not contain initiators sensitive to heat and evidence has been found to support the idea of improvement in some mechanical properties when these materials are submitted to post-curing treatments, especially those with temperature [10,11,15,17].

Post-curing treatments which involve devices that generate temperatures of about 100º C entail an improvement of the mechanical properties of composites.[2-9] This study evaluated the effect of two post-curing treatments on the FS and E of two indirect composites: Adoro (temperature reached by Lumamat 100, 107º C) and Belleglass (temperature reached by Belleglass HP, 135º C). Although statistical differences were found in both cases, Belleglass showed overall greater improvement, possibly due to two different factors which may allow a higher conversion degree: the controlled oxygen-free atmosphere and the higher temperature employed in Belleglass HP [12,19,20].

The differences in the characteristics, temperatures, atmosphere and pressure employed in each curing device may contribute to the high variability found among the results in the literature reviewed.[4,6] Therefore, in order to gain in standardization in this study, the same device (Belleglass HP) was applied to treat both direct composites.

In agreement with other studies, flexural strength increased in both Heliomolar and Premisa [4,10-12], but modulus of elasticity only did in Heliomolar [4,9,12], Some authors suggest that both E and FS are affected directly by the volume of inorganic filler, but also by the type and distribution of the monomers that constitute the matrix and the way in which they are affected by exposure to temperature. However, there is lack of information about the effect of temperature on the different monomers systems. There is some evidence that higher amounts of UDMA (urethane dimethacrylate) may be related to a greater increase in FS and E after post-cure,[14] which is consistent with the behavior of Heliomolar in this study. There is also some agreement that a higher proportion of TEGMA (triethylene glycol dimethacrylate) results in a higher degree of conversion [13], which may explain the increase in FS in Premisa. The increase in FS after the post-cure treatments may thus be due to the behavior of two different monomers present in each direct material. The lack of improvement in E for Premisa may be explained by its higher content of inorganic filler, which might have made the effect of post-curing in the organic matrix less noticeable [2,12,18].

Although some authors have reported statistically lower FS values in indirect than in direct composites [10,11], in this study no significant difference was found in FS between Belleglass and Heliomolar after they were submitted to post-cure treatment. On the other hand, Belleglass had the highest elastic modulus, whereas Adoro, also an indirect composite, had the lowest. Finally, there was no significant difference in E or FS between the direct materials.

Even though FS and E increased in both direct and indirect materials, the accretion was greater in Belleglass, probably due to the thermosensitive initiators in its composition, which may have helped to attain higher conversion rates and thus a more cross-linked matrix. Although direct composites lack this kind of initiator, both properties improved in Heliomolar, while FS improved in Premisa.

Although the mechanism by which the post-curing procedures affect the organic matrix and thus the final properties of the composites are still unclear in the literature, the differences in the kind and proportion of co-monomers present in each formulation may explain the somewhat erratic behavior of the study materials. Further research is needed to determine the actual composition of commercial composites - considering that this information is not always available with the necessary detail - and how it relates to the effect of additional curing processes on the mechanical properties of both direct and indirect composites and its clinical relevance.

ACKNOWLEDGEMENTS

This study was supported by a grant from University of Buenos Aires (code 20020090100178).

The authors wish to acknowledge the generous support of Dr. Fernando Maravankin with the supply of BelleGlass HP device.

CORRESPONDENCE

Dr. Verónica Almeida Chetti.
M. T. de Alvear 2142
C1122AAH Buenos Aires. Argentina.
veroalch@yahoo.com.ar

REFERENCES

1. Touati B, Aidan N. Second Generation Laboratory Composite Resins for Indirect Restorations. J Esthet Dent 1997; 9:108-118.
2. Wendt SL Jr. The effect of heat used as a secondary cure upon the physical properties of three composite resins. I. Diametral tensile strength, compressive strength, and marginal dimensional stability. Quintessence Int 1987;18: 265-271.
3. Wendt SL Jr. The effect of heat used as secondary cure upon the physical properties of three composite resins. II. Wear, hardness, and color stability. Quintessence Int 1987;18: 351-356.
4. Peutzfeldt A, Asmussen E. Mechanical properties of three composite resins for the inlay/onlay technique. J Prosthet Dent 1991;66:322-324.
5. Khan AM, Satou N, Shintani H, Taira M, Wakasa K, Yamaki M. Effects of post-curing by heat on the mechanical properties of visible-light cured inlay composites. J Oral Rehabil 1993;20:605-614.
6. Eldiwany M, Powers JM, George LA. Mechanical properties of direct and post-cured composites. Am J Dent 1993; 6:222-224.
7. Loza-Herrero MA, Rueggeberg FA. Time-temperature profiles of post-cure composite ovens. Gen Dent 1998;46:79-83.
8. Soares CJ, Pizi ECG, Fonseca RB, Martins LRM. Mechanical properties of light-cured composites polymerizec with several additional post-curing methods. Oper Dent 2005; 30-3:389-394.
9. Gonçalves Mota E, Mitsuo Silva Oshima H, Henrique Burnett Jr. L, Spohr AM, Nogarett Pibernat de Carvalho LM. Influencia de la polimerización adicional en las propiedades de una resina Ormocer®. Av Odontoestomatol 2006; 22: 271-277.
10. Santana IL, Lodovici E, Matos JR, Medeiros IS, Miyazaki CL, Rodrigues-Filho LE. Effect of experimental heat treatment on mechanical properties of resin composites. Braz Dent J 2009; 20:205-210.
11. Miyazaki CL, Medeiros IS, Santana IL, Matos Jdo R, Rodrigues Filho LE. Heat treatment of a direct composite resin: influence on flexural strength. Braz Oral Res 2009; 23:241-247.
12. Peutzfeldt A, Asmussen E. The effect of postcuring on quantity of remaining double bonds, mechanical properties, and in vitro wear of two resin composites. J Dent 2000; 28:447-452.
13. Asmussen E, Peutzfeldt A. Influence of UEDMA BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater 1998;14:51-56.
14. Asmussen E, Peutzfeldt A. Mechanical properties of heat treated restorative resins for use in the inlay/onlay technique. Scand J Dent Res 1990; 98:564-567.
15. Borba M, Della Bona A, Cecchetti D. Flexural strength and hardness of direct and indirect composites. Braz Oral Res 2009; 23:5-10.
16. Reinhardt JW, Boyer DB, Stephens NH. Effects of secondary curing on indirect posterior composite resins. Oper Dent 1994; 19:217-220.
17. César PF, Miranda Jr. WG, Braga RR. Influence of shade and storage time on the flexural strength, flexural modulus, and hardness of composites used for indirect restorations. J Prosthet Dent 2001; 86: 289-296.
18. Kildal KK, Ruyter IE. How different curing methods affect mechanical properties of composites for inlays when tested in dry and wet conditions. Eur J Oral Sci 1997;105:353-361.
19. Kildal KK, Ruyter IE. How different curing methods affect the degree of conversion of resin-based inlay/onlay materials. Acta Odontol Scand 1994; 52:315-322.
20. Bağis YH, Rueggeberg FA. Effect of post-cure temperature and heat duration on monomer conversion of photo-activated dental resin composite. Dent Mater 1997; 13:228-32.
21. Materiales Dentales. 4ª edición. Macchi RL. 2007. Panamericana Publisher.
22. ISO – Standars. ISO 4049 Dentistry - Polymer-based filling, restorative and luting materials. International Organization for Standardization. Switzerland. 2000.
23. American National Standard/American Dental Association. Specification Nº 27 for resin based filling material. 1993.