

# Effect of Post-Cure Heating on the Flexural Strength of Two Indirect Resin Composites.

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**Abstract** - The aim of this study was to evaluate whether a post-cure heat-treatment may improve the flexural strength of two indirect resin-based composites. Tested factors were: material (Gradia Indirect, Gradia Forte), mass (opaqus dentin, dentin, enamel) and curing mode (light, light and heat). A three-point bending test appliance was developed according to ISO 4049/2000. Three-Way ANOVA and 2-Parameter Weibull cumulative distribution function were performed. Factors material and curing mode were significant ( $p < 0.001$ ), while the mass type was not ( $p = 0.181$ ). A post-cure heat treatment may be useful for enhancing the flexural strength of both materials.

**KEY WORDS:** Composite, Post-cure, Post-curing, Heat, Heating, Post-heating.

## INTRODUCTION

Recently, among the materials available for the aesthetic treatment of anterior and posterior teeth, a growing interest has been directed toward the use of indirect resin composites<sup>1</sup>. Aesthetics needs, reduced costs of simplified laboratory procedures and concerns about the presence of metals, have contributed to expand the applications of resin composites, originally developed as restorative materials, to full coverage crowns and small fixed partial dentures<sup>2,3</sup>. In spite of a similar chemical composition, indirect resin composites are known to possess superior physical and mechanical properties than in situ-cured resin restorations<sup>4</sup>. The clinical behaviour of indirect composites may be further improved by performing a post-curing heat treatment, which is supposed to increase the degree of monomer conversion<sup>5</sup>. Fracture toughness<sup>6</sup>, flexural strength and modulus<sup>6,7</sup>, surface hardness<sup>8,9</sup>, marginal integrity<sup>10</sup> and biocompatibility<sup>11</sup>, are some of the properties that seemed to benefit from post-curing treatment. A new indirect resin composite, classified as MFR (i.e., Microparticle Filled Resin) nano-hybrid type (Gradia Forte), was recently introduced on the market. For this material, a dry heat-curing process following the standard light-curing is recommended by the manufacturer.

The aim of this study was to evaluate whether a post-curing heat treatment may have a beneficial effect on the flexural strength of two indirect resin-based materials. The null hypotheses investigated were that there is no difference in the composite flexural strength: (a) when a dry heat-curing is performed as an additional post-curing treatment, (b) regardless of the composite material used, and (c) irrespective of the selected mass.

## MATERIALS AND METHODS

Two indirect resin composites (Gradia Indirect; Gradia Forte) were used in this study (Table 1). For each material, three masses were selected: opaqus dentin (OD), dentin (D) and enamel (E). Twenty bar-shaped composite specimens were made of each mass, using a vinylpolysiloxane-based mould (Elite H-D+) with dimensions of 25 mm length, 2.1 mm height, and 2.1 mm width. The mould was filled by layering two increments, of about 1 mm in thickness. Each increment was light-cured for 30 s. A polyethylene strip was positioned and pressed against the last increment of composite with a glass slide in order to obtain a flat surface. All the specimens were light-cured in a light-curing unit (Labolight IV-II) for 3 min. In addition, ten specimens for each mass were also heat-cured in a heat-curing device (Petit Oven PO-I) for 15 min at 110 °C.

Specimens were wet-ground with 600 and 1200 FEPA metallographic paper, until the dimensions of  $2.0 \pm 0.1$  mm in height and  $2.0 \pm 0.1$  mm in width were obtained, as required by the normative ISO 4049/2000<sup>12</sup>, and then stored in distilled water for 24 h. Afterwards, a three-point bending test was performed by using a new device developed to run the test, as specified by the indications reported in ISO 4049/2000<sup>12</sup>. The appliance consisted of two HSS/Cobalt rods (diameter: 2 mm) mounted parallel with 20 mm between centers (support span: 20 mm). Each specimen was loaded at its center with a cylindrical-ended striker (diameter: 2 mm), with a crosshead speed of 0.75 mm/min until failure in a universal testing machine (Triaxial Tester T400 Digital). The load at failure was recorded and the flexural strength ( $\sigma$ ) was calculated in Megapascals (MPa) using the following equation:

$$\sigma = \frac{3Fl}{2bb^2}$$

where  $F$  is the load at failure (N),  $l$  is the span between the supports (mm),  $b$  is the specimen width (mm), and  $b$  is the specimen height (mm).

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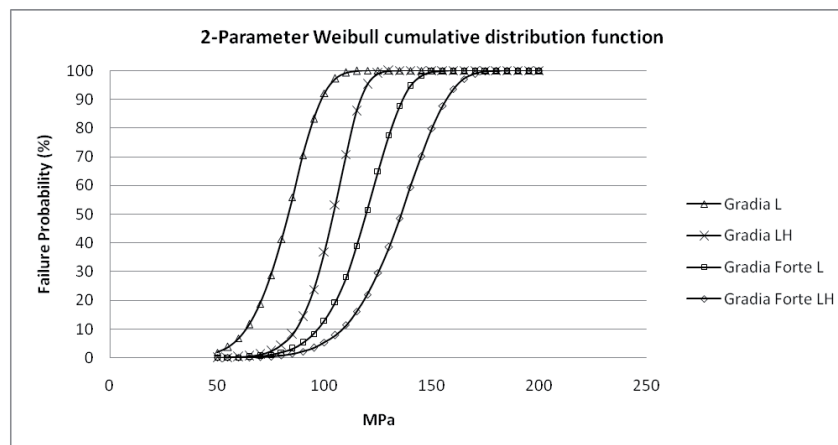
**Table 1.** Manufacturer, batch number and composition of the materials used in this study.

	<i>Gradia Indirect</i>	<i>Gradia Forte</i>
Manufacturer	GC Corp., Tokyo, Japan	GC Corp., Tokyo, Japan
Batch N.	0612121	0610111
<b>Composition (% in weight)</b>		
Urethane based methacrylate	21	20
Multifunctional methacrylate	4	4
Silica nano-fillers	5	4
Fine particle glass fillers	49	69
Prepolymerized fillers	21	3
Photoinitiator	Trace	Trace
Pigment	Trace	Trace

**Table 2.** Means and standard deviations (S.D.) of the flexural strength measured in the tested groups.

Group	Material	Curing Mode	Mass	N	Mean (MPa)	S.D. (MPa)
1	Gradia Indirect	L	OD	10	71.1	8.1
2			D	10	84.1	10.8
3			E	10	90.5	13.6
4			OD	10	97.0	4.9
5	Gradia Forte	LH	D	10	101.5	16.6
6			E	10	109.9	5.9
7			OD	10	128.9	12.5
8			D	10	112.4	11.4
9	Gradia Forte	L	E	10	112.5	15.3
10			OD	10	135.0	17.9
11			D	10	129.9	18.5
12			E	10	136.8	20.9

**L:** Light curing; **LH:** Light curing + heat treatment; **OD:** Opaqus dentin; **D:** Dentin; **E:** Enamel.

**Figure 1.** 2-parameter Weibull cumulative distribution function. **L:** Light curing; **LH:** Light curing + heat treatment.

As the data were normally distributed (Kolmogorov-Smirnov test), and groups exhibited homogeneous variance (Levene test), a three-way ANOVA was employed for examining the effect of “material”, “mass” and “curing mode” (i.e., independent variables) on the composite flexural strength (i.e., dependent variable). The analyses were processed by the SPSS 12.0 software with significance set at the 95% probability level. A 2-Parameter Weibull cumulative distribution function was also performed in order to determine the failure probability at specific loads. The equation used was:

$$F(\chi; \kappa, \lambda) = 1 - e^{-\left(\frac{\chi}{\lambda}\right)^\kappa},$$

where  $\chi$  are the investigated values,  $\kappa$  is the Scale Parameter (Characteristic Life) and  $\lambda$  is the Shape Parameter.

## RESULTS

The means and standard deviations of the flexural strength for all the tested groups are presented in Table 2. The three-way ANOVA revealed that factors material and curing mode were significant ( $p < 0.001$ ), while factor mass was not ( $p = 0.181$ ). Only the interaction between material and mass was statistically significant ( $p = 0.001$ ).

The statistical analysis pointed out significant differences between the tested resin-based composites, with Gradia Forte recording superior flexural strength than Gradia Indirect. For both materials, post-cure heat treatment resulted in statistically higher values than light-cured groups. When Gradia Indirect was only light-cured, flexural strength ranged between  $71.1 \pm 8.1$  MPa (L-OD) and  $90.5 \pm 13.6$  MPa (L-E), while when it was also heat-cured results ranging between  $97.0 \pm 4.9$  MPa (LH-OD) and  $109.9 \pm 5.9$  MPa (LH-E) were obtained. Flexural strength between  $112.4 \pm 11.4$  MPa (L-D) and  $128.9 \pm 12.5$  MPa (L-OD) and between  $129.9 \pm 18.5$  MPa (LH-D) and  $136.8 \pm 20.9$  MPa (LH-E) were achieved, respectively, for light-cured and light/heat-treated Gradia Forte.

The 2-Parameter Weibull distribution curve is presented in Figure 1. The lowest probability of failure was recorded for light/heat-cured Gradia Forte ( $\kappa = 141.74$ ;  $\lambda = 8.32$ ), followed by light-cured Gradia Forte ( $\kappa = 124.36$ ;  $\lambda = 9.06$ ) and light/heat-cured Gradia Indirect ( $\kappa = 107.79$ ;  $\lambda = 10.35$ ). The highest probability of failure was found in flexural specimens made of light-cured Gradia Indirect ( $\kappa = 87.49$ ;  $\lambda = 7.02$ ).

## DISCUSSION

The three-point bending test was used to evaluate the mechanical behaviour of the resin composites by measuring their flexural strength. This is basically a monotonic test, as a static load is applied to the specimen up to failure, and it is not entirely representative of the fatigue resistance and the clinical behaviour of the tested material<sup>13</sup>. However, the three-point bending test is considered as a reliable method to evaluate the flexural strength<sup>14</sup>, it is recommended in the ISO 4049/2000 specification<sup>12</sup> and it is commonly used for comparative purposes<sup>14-17</sup>.

Post-curing heat treatment had a significant influence on the composite flexural strength of the tested indirect composites. Hence, the first null hypothesis was rejected. In light-cured, methacrylate-based composites, 30 to 45% of the resin monomers remain unreacted in the form of pendant C=C bonds<sup>18-20</sup>. Several studies have shown that a significant positive correlation exists between the degree of conversion of residual monomers into polymer and the physical and biological behaviour of the resin-based material<sup>6</sup>. The exposure of a previously cured composite restoration to high extra-oral temperatures may induce an additional activation of the polymerization reaction<sup>21</sup>. As the reactive potential of residual resin monomers is compromised by the presence of oxygen and water and it gradually decays over time after polymerization<sup>22, 23</sup>, the earlier heat is applied after initial photo-curing and the higher is the overall conversion value<sup>24, 25</sup>. It has been shown that if post-cure heating is performed within 6 hours of initial light-curing, the degree of cure and the overall material's properties may still be improved<sup>25</sup>. The extent of polymerization was found to raise linearly with the increase of post-cure temperature, which was even more beneficial than heat treatment duration on monomer conversion<sup>11, 21</sup>. Temperature is thought to accelerate the mobility of the dimethacrylate resin monomers and to result in a more homogenous polymer matrix<sup>26</sup>. Three are the mechanisms proposed by Bagis and Rueggeberg<sup>5</sup> to explain the lower content of residual, potentially leachable monomers after post-cure heating: the covalent bonding of C=C bonds to the polymer network, the decrease in diffusion channel size when the higher degree of conversion is associated with a superior cross-link density in the polymer, and/or the volatilization of the resin monomers promoted by the temperature increase.

In this study, post-cure heating at 110°C for 15 minutes led to a significant improvement of the composite flexural strength. This is in line with a previous investigation<sup>6</sup>, showing a major enhancement of several physical properties when indirect resin composites were post-cured at 80-120°C for 15 minutes. The increase in composite flexural strength is presumably related to the higher degree of monomer conversion achieved at these temperatures<sup>5</sup>. In the present investigation, the beneficial effect of heat-treatment for Gradia Forte, as claimed by the manufacturer, was confirmed. A further finding was that heating also resulted a valuable post-curing method for Gradia Indirect, for which no post-curing method after light-curing is usually recommended and has so far been proposed.

Significant differences in composite flexural strength were detected among the two tested materials, thus leading to the rejection of the second null hypothesis. It is known that the mechanical properties of resin-based composites are mainly dependant upon their microstructure and composition, such as the composition of the matrix<sup>27-29</sup>, the type, quantity, size of fillers and their interface with the resin matrix<sup>16, 29-31</sup>. Varying the matrix composition and the relative amounts of UDMA, UEDMA, BisGMA and TEGDMA has a significant effect on the physical properties of the resin<sup>28, 32</sup>. However, fillers are considered more influent on the final mechanical properties of the resin-based material<sup>33</sup>. Rodrigues *et al.*<sup>16</sup> found a significant positive correlation between the filler weight and flexural strength/modulus of elasticity. The bonding between the matrix and the filler

particles also plays an important role in the behaviour of nano-filled composites<sup>27</sup>. In particular, if the particles are non-bonded with the resin matrix, the overall wear resistance of the material can be affected. The results obtained in this study indicated that for a given curing cycle, the two composites achieved statistically different flexural strength values. An explanation might reside in the different chemical composition of tested materials. Based on the information provided by the manufacturer, the two composites have a similar filler loading, ranging between 75 and 76% in weight (Table 1). Although the exact composition of the resin matrix is not specified, the greatest difference seems to involve the type of filler used (Table 1). Gradia Forte, which obtained better results, presents a larger percentage of fine particle glass filler (69% instead of 49%) and a smaller percentage of pre-polymerized fillers (3% instead of 21%).

No significant differences were found in specimens made by different masses (opaqus dentin, dentin and enamel), thus leading to the acceptance of the third null hypothesis.

From the information provided by the manufacturer the composition of the tested masses is the same for each material. Based on this information, the differences in opacity may be reasonably due to the pigments type/quality, but this is not specified by the manufacturer itself. Therefore, a possible difference in mechanical properties (not found in this study) of different masses should not be attributable to compositional differences. Shortall<sup>34</sup> investigated the influence of the material's shade on the depth of cure of a direct composite and a correlation between the opacity of the material and the depth of cure was found. Similar results have been shown by Tanoue *et al*<sup>35</sup>, which demonstrated that the depth of cure may be influenced by the shade letter (A, B, C or D) and the shade number (1 and 2, or 4). To prevent a possible influence of the shade on the results, in this study the specimens were made by using the same shade for the different masses tested (A3). The difference in opacity between opaqus dentin, dentin and enamel was clinically visible, and it was logical to expect a better polymerization in a more translucent mass (i.e. enamel), rendered in a higher flexural strength. The methods used in this study may have reduced this phenomenon. Specimens were fabricated according to the manufacturer's instructions, by layering and light-curing increments of about 1 mm in thickness. A final light-curing of 3 min was also performed. In these conditions the influence of the mass type on the composite flexural strength was not statistically relevant.

## MANUFACTURERS' DETAILS

- Elite H-D+: Zhermack Spa, Badia Polesine, Italy.
- Gradia Forte: GC, Tokyo, Japan.
- Gradia Indirect: GC, Tokyo, Japan.
- Labolight LV-II: GC Corp., Tokyo, Japan.
- Petit Oven PO-I: GC Corp., Tokyo, Japan.
- SPSS 12.0: SPSS, Chicago, IL, USA.
- Triaxial Tester T400 Digital: Controls, Milano, Italy.

## ADDRESS FOR CORRESPONDENCE

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## REFERENCES

1. ADA Council on Scientific Affairs. Direct and indirect restorative materials. *J Am Dent Assoc* 2003;**134**(4):463-72.
2. Ellakwa A, Thomas GD, Shortall AC, Marquis PM, Burke FJ. Fracture resistance of fiber-reinforced composite crown restorations. *Am J Dent* 2003;**16**(6):375-80.
3. Husein A, Berekally T. Indirect resin-bonded fibre-reinforced composite anterior bridge: a case report. *Aust Dent J* 2005;**50**(2):114-8.
4. Gregory WA, Berry S, Duke E, Dennison JB. Physical properties and repair bond strength of direct and indirect composite resins. *J Prosthet Dent* 1992;**68**(3):406-11.
5. Bagis YH, Rueggeberg FA. The effect of post-cure heating on residual, unreacted monomer in a commercial resin composite. *Dent Mater* 2000;**16**(4):244-7.
6. Takeshige F, Kinomoto Y, Torii M. Additional heat-curing of light-cured composite resin for inlay restoration. *J Osaka Univ Dent Sch* 1995;**35**(59-66).
7. Klymus ME, Shinkai RS, Mota EG, Oshima HM, Spohr AM, Burnett LH. Influence of the mechanical properties of composites for indirect dental restorations on pattern failure. *Stomatologija* 2007;**9**(2):56-60.
8. Hirabayashi S, Hood JA, Hirasawa T. The extent of polymerization of Class II light-cured composite resin restorations; effects of incremental placement technique, exposure time and heating for resin inlays. *Dent Mater J* 1993;**12**(2):159-70.
9. Marais JT, Dannheimer MF, Oosthuizen MP, Booysse A. The effect of post-curing on Vickers Hardness of four light-cure resin composite materials. *Sadj* 1999;**54**(3):123-5.
10. Wendt SL, Jr., Leinfelder KF. Clinical evaluation of a heat-treated resin composite inlay: 3-year results. *Am J Dent* 1992;**5**(5):258-62.
11. Bagis YH, Rueggeberg FA. Effect of post-cure temperature and heat duration on monomer conversion of photo-activated dental resin composite. *Dent Mater* 1997;**13**(4):228-32.
12. International Organization for Standardization. ISO 4049/2000 - Dentistry -- Polymer-based filling, restorative and luting materials. Geneva, Switzerland. 2000.
13. Scherrer SS, Wiskott AH, Coto-Hunziker V, Belser UC. Monotonic flexure and fatigue strength of composites for provisional and definitive restorations. *J Prosthet Dent* 2003;**89**(6):579-88.
14. Chung SM, Yap AU, Chandra SP, Lim CT. Flexural strength of dental composite restoratives: comparison of biaxial and three-point bending test. *J Biomed Mater Res B Appl Biomater* 2004;**71**(2):278-83.
15. Asmussen E, Peutzfeldt A. Flexural strength and modulus of a step-cured resin composite. *Acta Odontol Scand* 2004;**62**(2):87-90.
16. Rodrigues Junior SA, Zanchi CH, Carvalho RV, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. *Braz Oral Res* 2007;**21**(1):16-21.
17. Pereira CL, Demarco FF, Cenci MS, Osinaga PW, Piovesan EM. Flexural strength of composites: influences of polyethylene fiber reinforcement and type of composite. *Clin Oral Investig* 2003;**7**(2):116-9.
18. Asmussen E. Factors affecting the quantity of remaining double bonds in restorative resin polymers. *Scand J Dent Res* 1982;**90**(6):490-6.
19. Inoue K, Hayashi I. Residual monomer (Bis-GMA) of composite resins. *J Oral Rehabil* 1982;**9**(6):493-7.
20. Ruyter IE, Svendsen SA. Remaining methacrylate groups in composite restorative materials. *Acta Odontol Scand* 1978;**36**(2):75-82.
21. 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**(6):447-52.
22. Burtcher P. Stability of radicals in cured composite materials. *Dent Mater* 1993;**9**(4):218-21.
23. Lapcik L, Jr., Jancar J, Stasko A, Saha P. Electron paramagnetic resonance study of free-radical kinetics in ultraviolet-light cured dimethacrylate copolymers. *J Mater Sci Mater Med* 1998;**9**(5):257-62.

24. Wu W, Fanconi BM. Post-curing of dental restorative resin. *Polymer Engineering & Science* 1983;**23**(13):704-707.
25. Loza-Herrero MA, Rueggeberg FA, Caughman WF, Schuster GS, Lefebvre CA, Gardner FM. Effect of heating delay on conversion and strength of a post-cured resin composite. *J Dent Res* 1998;**77**(2):426-31.
26. Ruyter IE. Types of resin-based inlay materials and their properties. *Int Dent J* 1992;**42**(3):139-44.
27. Ferracane JL, Matsumoto H, Okabe T. Time-dependent deformation of composite resins--compositional considerations. *J Dent Res* 1985;**64**(11):1332-6.
28. Asmussen E, Peutzfeldt A. Influence of UEDMA BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. *Dent Mater* 1998;**14**(1):51-6.
29. Musanje L, Ferracane JL, Ferracane LL. Effects of resin formulation and nanofiller surface treatment on in *vitro* wear of experimental hybrid resin composite. *J Biomed Mater Res B Appl Biomater* 2006;**77**(1):120-5.
30. Calais JG, Soderholm KJ. Influence of filler type and water exposure on flexural strength of experimental composite resins. *J Dent Res* 1988;**67**(5):836-40.
31. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002;**87**(6):642-9.
32. Floyd CJ, Dickens SH. Network structure of Bis-GMA- and UDMA-based resin systems. *Dent Mater* 2006;**22**(12):1143-9.
33. Hervas-Garcia A, Martinez-Lozano MA, Cabanes-Vila J, Barjau-Escribano A, Fos-Galve P. Composite resins. A review of the materials and clinical indications. *Med Oral Patol Oral Cir Bucal* 2006;**11**(2):E215-20.
34. Shortall AC. How light source and product shade influence cure depth for a contemporary composite. *J Oral Rehabil* 2005;**32**(12):906-11.
35. Tanoue N, Koishi Y, Matsumura H, Atsuta M. Curing depth of different shades of a photo-activated prosthetic composite material. *J Oral Rehabil* 2001;**28**(7):618-23.