

Influence of post-curing method on cure efficiency and roughness of direct resin composites in indirect restorations

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Objective: To evaluate the influence of different post-curing methods on cure efficiency and surface roughness of direct composite resins in indirect restorations compared to the conventional light-curing protocol. **Material and Methods:** Two direct composite resins, one nanofilled (Filtek Z350, 3M/ESPE) and one micro-hybrid (Amelogen, Ultradent), were used to obtain specimens ($d = 4$ mm, 2-mm thick) via different curing methods ($n = 10$). All specimens were light-cured at 48 J/cm^2 (conventional-control), then subjected to a post-curing method:

microwave post-curing or autoclave post-curing techniques. Both degrees of conversion and surface roughness were evaluated by means of Fourier transform infrared spectroscopy and a roughness meter, respectively. Data were submitted to two-way ANOVA and Tukey test ($\alpha = 0.05$) for multiple comparisons. **Results:** Post-curing methods increased the degree of conversion for both composites (micro-hybrid: $\sim 75\%$ and nanofilled: $\sim 60\%$) in comparison to the conventional light-curing method (micro-hybrid: $\pm 69\%$ and nanofilled: $\sim 56\%$). The curing method

had no statistical effect on surface roughness, although the micro-hybrid composite presented higher roughness than the nanofilled composite (~ 0.16 and ~ 0.13 , respectively). **Conclusion:** Within the limitations of this study, the following conclusion can be drawn: the microwave and autoclave techniques showed to be efficient post-curing methods for improving cure efficiency without compromising surface roughness, as compared to the conventional light-curing protocol. **Keywords:** Light-curing. Permanent dental restoration.

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Introduction

Post-cured direct resin composites seem to have similar or superior physical-mechanical properties compared to indirect resin composites, thus they are also appropriate to perform indirect restorations in some clinical situations.¹⁻⁴ Clinical follow-ups comparing indirect restorations performed with direct and indirect composite resins also demonstrated acceptable clinical management of this alternative technique compared to ceramics.⁵

Although increasing light-curing exposure can lead to improvements in physical-mechanical properties of resin materials,⁶⁻⁷ these improvements depend on the composition content of each composite.⁸⁻⁹ Post-curing techniques seem to be more efficient to improve physical-mechanical properties of direct resin composites, regardless of composite composition or light-curing protocol.³⁻⁴

Although many post-curing techniques are described in literature, not all of these post-curing techniques have been tested scientifically or shown to be effective. For example, post-heating direct composite resins in an oven has been shown not to be an efficient post-curing method for the improvement of physical-mechanical properties.⁶

Another important property of indirect restorations is the esthetical appearance, which can be affected by differences in surface roughness caused by polymer volatilization at higher temperatures.¹⁰ Differences in roughness cause different light reflection, thus enabling the color to appear glossier on smooth surfaces or more dull to rougher surfaces,

causing perceptible mismatching between the gloss of the restoration and the natural teeth.¹¹ Also, rougher surfaces are more susceptible to staining and bacterial plaque retention, likely reducing the longevity of the restoration.¹²⁻¹³

This study aims to evaluate the influence of the microwave and autoclave cycle techniques as post-curing methods on degree of conversion and roughness of direct resin composites. The tested hypothesis of this study is that the post-curing methods tested will increase the cure efficiency without compromising surface roughness of direct resin composites compared to conventional light-curing protocol.

Materials and methods

Table 1 shows the composition of each resin composite used to produce specimens ($d=4\text{mm}$, 2mm thick; $n=10$) using the different curing methods evaluated in this study. All specimens were light cured at 48 J/cm^2 (Blue-phase G2, IvoclarVivadent, Schaan, Liechtenstein), then associated with no post-curing method (conventional), microwave post-curing for 4 minutes at high potency, (MEF41, 1000mW, Eletrolux, Stockholm, Sweden), or autoclave cycle for 40 minutes at $121\text{ }^\circ\text{C}$.

Table 1: Material, product, manufacturer and composition of each resin composite.

COMPOSITE	PRODUCT	MANUFACTURER	COMPOSITION
Nanofilled	Filtek Z350 XT EA1	3M ESPE, St. Paul, MN, EUA	Similar mixture of Bis-GMA, Bis-EMA, UDMA and TEGDMA with nanoparticles of non-agglomerated silica and zirconia/silica nanoclusters (59.5 vol%)
Microhybrid	Amelogen EA1	Ultradent Inc., South Jordan, UT, EUA	Mixture of BisGMA (<60wt%) and TEGDMA (<40%) with silica dioxide and silicates particles (52vol%)

Bis-GMA (Bisphenol A glycidyl methacrylate); Bis-EMA (ethoxylated bis-phenol A methacrylate); UDMA (urethane dimethacrylate); TEGDMA (triethylene-glycol di-methacrylate).

Cure Efficiency

Cure efficiency for each resin composite was measured using Fourier transform infrared spectroscopy (FT-IR) coupled to an attenuated total reflectance (ATR) (Spectrum 100, Perkin-Elmer, MA, USA). Each sample was placed in a Teflon mold ($d=4\text{mm}$, 2mm thick). Unpolymerized blends were scanned, photo-activated with 48J/cm^2 , and treated to conventional (no post-curing method), microwave, or autoclave cycle post-curing methods. The polymerized samples were scanned, and unconverted carbon double bonds were quantified by calculating the area ratio derived from the aliphatic C=C (vinyl) absorption (1638 cm^{-1}) to the aromatic C=C absorption (1608 cm^{-1}) signals for both polymerized and unpolymerized samples ($n=10$). The cure efficiency for each resin was calculated as the degree of conversion (DC), according to the follow equation:

$\text{DC (\%)} = \{1 - (X_a/Y_a)/(X_b/Y_b)\} \times 100$, where, X_a (polymerized) and X_b (unpolymerized) represent the areas under the polymerizable aliphatic double bond bands, and Y_a (polymerized) and Y_b (unpolymerized) represent the areas under the aromatic double bond bands.

Surface roughness

The surface roughness was determined by a roughness meter (Mitutoyo, Kanagawa, Japan) and characterized by the average height parameter, Ra (mm). Three measurements passing through the center of the specimen were performed to calculate the average.

Statistical analysis

Data were submitted to two-way analysis of variance (ANOVA) and Tukey's test ($p = 0.05$) for multiple comparisons.

Table 2: For each of the dental composite resins, the mean degree of conversion value (%) \pm standard error is provided.

COMPOSITE	CURING MODE		
	Conventional	Autoclave	Microwave
Microhybrid	69,2 (3,5) Ab	74,5 (5,3) Aa	75,7 (4,1) Aa
Nanofilled	56,1 (2,1) Bb	60,6 (6,5) Ba	62,1 (5,1) Ba

Means followed by different capital letters in the same line and small letters in the same column were significantly different ($p < 0.05$).

Table 3: For each of the dental composite resins, the mean surface roughness value (Ra) \pm standard error is provided.

COMPOSITE	CURING MODE		
	Conventional	Autoclave	Microwave
Microhybrid	0,17 (0,1) Aa	0,16 (0,0) Aa	0,12 (0,0) Aa
Nanofilled	0,13 (0,1) Ba	0,13 (0,0) Ba	0,12 (0,0) Ba

Means followed by different capital letters in the same line and small letters in the same column were significantly different ($p < 0.05$).

Results

Tables 2 and 3 show the mean degree of conversion and surface roughness values (%) \pm standard error, respectively. The post-curing methods evaluated, microwave and autoclave, increased the cure efficiency for both composites (microhybrid: 75.7% (± 4.1) and 74.5% (± 5.3); nanofilled: 62.1% (± 5.1) and 60.6% (± 6.5), respectively) in comparison to the conventional photo-curing method (microhybrid:

69.2 (± 3.5) and nanofilled: 56.1% (± 2.1)). No statistical differences were found among the surface roughness of any curing protocol regardless of the resin composite evaluated; the microhybrid composite presented higher roughness than the nanofilled.

Discussion

The tested hypothesis that the post-curing methods would improve cure efficiency without compromising the surface roughness of direct resin composites compared to the conventional light-curing protocol was accepted. As observed in Table 2, the degree of conversion for both direct composite resins increased with post-curing techniques when compared to conventional light-curing without compromising surface roughness (Table 3).

As previously suggested, different post-curing techniques can improve physical-mechanical properties of direct composite resins regardless of composite resin composition.³⁻⁴ Although post-heating in an oven had been shown not to be an efficient post-curing method for improving physical-mechanical properties of direct composite resins,⁶ the microwave and autoclave techniques were efficient post-curing methods that improved degree of conversion of direct composite resins.

These outcomes can be explained by considering thermal stability. Elevated temperature below 60°C can improve the mechanical properties considerably due to increased cross-linking of the resin system, creating a more stable polymer;¹⁵ but temperatures above the thermogravimetric temperature of polymer components causes volatilization from low molecular weight monomers to high molecular weight monomers, thus causing

consecutive degradation of the organic matrix in resin composites.¹⁰ The temperature used on the post-curing methods evaluated in this study showed to be efficient to improve the monomer polymerization without causing significant polymer volatilization which would affect surface roughness.

Differences between both composites were observed. The nanofilled composite showed lower surface roughness and lower degree of conversion compared to the microhybrid, regardless of the post-curing method performed. While the precise composition of commercial materials is not fully released, differences in filler content can affect roughness and cure efficiency and may explain differences between the microhybrid and nanofilled. Nanofilled composite resins usually show smoother surfaces than hybrid composite resins because of the difference in the size of the filler particles, as can be observed in Table 1.¹⁶

Moreover, the size and amount of the filler can also affect the light transmittance through the composite, thus affecting monomer conversion.¹⁶⁻¹⁷ With smaller filler particles, higher filler loadings can be achieved. However, higher filler loadings, in turn, decrease light transmittance through the composite during

photo activation due to attenuation of light flux caused by increased light scattering.¹⁷

Light scattering is typically maximized when the filler particle size is close to that of the incoming light, which would be approximately 400-500 nm for the curing light used. This wavelength is approximately 10x larger than the size of the nanofillers used in the nano-filled composite evaluated, which would suggest minimal scattering. However, nanofillers tend to agglomerate, thereby producing particles with larger effective particle sizes, thus enhancing light scattering as they approach or exceed the wavelength of the curing light.¹⁸ Thus, the higher filler loading of the nano-filled composite explains its lower degree of conversion in comparison to the microhybrid.

Conclusions

Within the limitations of this in vitro study, the following conclusion can be drawn: the microwave and autoclave techniques showed to be efficient post-curing methods for improving cure efficiency without compromising surface roughness compared to the conventional light-curing protocol.

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