

Microhardness Evaluation of a Direct/Indirect Hybrid Composite Resin at Complementary Activation in Light or Heat

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Abstract. This investigation aims at determining the microhardness value of the hybrid direct/indirect composite resins submitted to conventional light curing and post-cured using two different methods, a light source from a laboratory light curing unit and a conventional oven heating, and the obtained results were compare to an indirect composite resin. A hybrid direct/indirect composite resin (Filtek P60TM – 3M-ESPE, St. Paul, USA), and an indirect composite resin (ArtglassTM – Heraeus Kulzer, Germany) were used. The Filtek P60TM was polymerized using: a conventional light curing for 80 seconds; the complementary polymerization methods were applied using a laboratory light curing for 360 seconds, and an oven curing at 125°C for 600 seconds. The ArtglassTM composite was polymerized using a laboratory light curing unit for 360 seconds. The post-curing method produced higher values of Vickers Hardness Number (HV) for both light and heat post-curing treatments, shown that increase in hardness is independent of the post-curing methods (laboratory light curing unit or heat curing using a conventional oven heat). Microhardness was determined by the Vickers indentation technique with a load of 50 gram-force for 15 seconds. The Filtek P60TM resin showed higher hardness values than ArtglassTM resin. The employment of post-curing using heat methods on Filtek P60TM resulted in similar mechanical properties (microhardness) that were comparable to laboratory light curing unit, and the laboratory light curing unit is much more expensive than oven heat.

Keywords: composite resin, complementary polymerization, post-curing, microhardness.

1. Introduction

Photo-activated composite materials were developed more than 15 years ago and have been widely used due to their improved physical and handling properties. However, when composites are used directly in posterior teeth several problems that deserve attention have been pointed out. One of the problems associations with the photo-activation system is lack of uniformity in the resin matrix conversion. Insufficient curing performance induces excessive wear or lost of anatomic form, staining, and discoloration of veneered restorations.

The adequate polymerization of composites is an important factor to ensure good clinical performance. The degree to which these materials are cured is proportional to the amount of light they are exposed to. A number of additional curing (post-curing) procedures have been proposed for overcoming insufficient material monomer conversion, as post-radiation with light curing, secondary heating, microwave, and pressure application during polymerization process (Tanoue, 1999 and 2000, Soares, 2005, Rueggeberg, 1988).

Indirect inlay/onlay techniques, using post-curing, have been introduced to enhance the mechanical and physical properties and improve the quality of restorations in posterior teeth (Asmussen, 1990 and 1991, Peutzfeldt, 1990). The most important aspect of indirect composite systems is the possibility of using high intensity light with laboratory light sources and other post-curing method (Tanoue, 1998 and 1999; Matsumura, 1999, Rueggeberg, 1997). The complementary curing increases the monomer conversion degree of the compose resin, resulting in a bigger breaking of the double carbon linking, providing an increase in the mechanical properties of the material (Rueggeberg, 1988).

The composition of resin-based inlay/onlay materials is principally the same as those composite resin-based direct filling materials. The use of external curing using light and heat results in a significantly higher degree of conversion (in the range 65% - 71%) than curing a direct technique (in the range 39% - 44%) (Kildal, 1997). These hypotheses suggest the use of direct filling composites in the manufacturing of indirect restorations.

Several mechanisms have been proposed to explain the further polymerization of post-cured treatment composites. The increase of the conversion degree may be an important factor that enhances the mechanical properties of composites (Park & Lee, 1996). Post-curing treatment results in increased segmental chain vibrational amplitude, allowing near radicals and methacrylate groups to collide, thus increasing monomer conversion (Rueggeberg, 1997). The use of the post-curing treatment, immediately after initial polymerization light is necessary to obtain this improvement (Baharav, 1997). Both the application of a laboratory light source or secondary heating after irradiation are known to be effective in improving certain properties of composites (Wendt Jr., 1987, Matsumura, 1999; Tanoue, 1999, Oertli, 2002).

Oertli et al (2002) showed that the ideal temperature of complementary activation for a composite resin is above of 100°C. Wendt Jr. (1987) showed that the excellent temperature of complementary activation of a composite resin so that it has an improvement of its physical properties is around 125°C. This temperature coinciding with the glass transition temperature of the majority of composite resins that can be an indicative of increase of the chain vibration amplitude of the resinous matrix. Wendt Jr. (1987) showed still that the wear rate reduce when the degree of conversion and the associated hardness was increased.

Microhardness tests are considered an efficient method to investigate the physical strength of a material, therefore, may be one appropriate indicative method to guide indirect composite application. The hardness of a material is a relative measure of its resistance of the material surface to indentation when a specific constant load is applied. Thus, the hardness may be described as a measure of the ability of a material to resist indentation or scratching (Miranda, 2003). The microhardness indentation, according to Xu *et al.* (2000), offers information that may be relevant to applications that involve localized, non-uniform deformation or point-contacts, such as occlusal contacts occurring on surface asperities or third bodies during chewing and wear. The Vickers microhardness is important property of restorative materials used in posterior teeth, and an indicative of the strength against compressive loading and the wear (Brosh, 1997).

The purpose of this study was to analyze the Vickers microhardness number of the direct/indirect hybrid composite resin (Filtek P-60TM), submitted to conventional light curing and post-curing using two different additional polymerization methods, light and heat, using a laboratorial light curing unit and a conventional oven heat. The mechanical behavior (Vickers microhardness) of this resin was compare to an indirect composite resin (ArtglassTM).

2. Methods and Materials

Two commercially composites were used in this work, a direct/indirect hybrid composite resin (Filtek P60TM – 3M-ESPE, St. Paul, USA), and an indirect composite resin (ArtglassTM – Heraeus Kulzer, Germany). Vickers microhardness tests were performed in order to analyze the post-curing effect on the mechanical properties of the direct/indirect hybrid composite resin, and compare to the indirect composite resin.

The FiltekTM P60 restorative material is a visible-light activated, radiopaque, restorative composite. It is designed for use in posterior restorations. The filler in Filtek P60 restorative is zirconia/silica. The inorganic filler loading is 75.9% in weight or 61.0% by volume (without silane treatment) with a particle size range of 0.01 to 3.50µm. The composite contains Bis-GMA (Bisphenol A diglycidyl ether dimethacrylate), Bis-EMA (Bisphenol A polyethylene glycol diether dimethacrylate), and UDMA (urethane dimethacrylate) resins (3M Product Catalog), Table 1.

The composite laboratory ArtglassTM is a combination of the organic portions the conventional monomer Bis-GMA and a multi-functional monomer UTMA (urethane-tetramethacrylate). The inorganic portion of this composite is essentially a Barium glass and colloidal silica with average size particles of 0.70µm, corresponding 75.0% in weight (Oertli, 2002), Table 1.

Table 1 – Restorative Materials used in the study - Composition and Manufacturers

Composite	Composition	Particle size (µm)	Manufacturer
Filtek P60 TM	Organic Matrix: Bis-GMA, Bis-EMA and UDMA. Inorganic Filler: rounded zirconia/silica (75.9% in weight or 61.0% in volume)	0.01 – 3.50	3M-ESPE, St. Paul, MN, USA
Artglass TM	Organic Matrix: Bis-GMA and UTMA, Barium glass and colloidal silica (75.0% in weight)	~ 0.70	Heraeus Kulzer, Germany

2.1. The specimens preparation

The specimens were prepared in a PVC mold, a cylindrical opening of 5.0mm in diameter and 2.0mm in thickness (Figure 1). The composite was placed into the mold, which was positioned on top of a glass slide of 1mm thick. A second glass slide of 1mm thick was then placed over the mold and gentle pressure was applied to extrude excess material. Then, the specimens were polymerized from top and bottom surface through the glass slide using light curing.

2.1.1. Filtek P-60

Fifteen specimens were prepared with Filtek P-60TM composite resin. The top and bottom surfaces of the specimen were light polymerized using a conventional light curing equipment (Optilight PlusTM – Gnatus, Brazil) (Figure 2 a), for 40 seconds of each side of the specimen. In this equipment, the light guide available is the 11mm outer diameter, the inner diameter is 10mm, and the output intensity is 500mW/cm², as assessed using a curing radiometer (Gnatus, Brazil). These specimens were divided in three different groups with 5 specimens each. The first group (G-I) as polymerized only conventional photo polymerization. A complementary polymerization (post-curing) was accomplished, for the second group (G-II) using a laboratory light curing unit with 270W power (UniXSTM –Heraeus Kulzer, Germany), (Figure 2 b), for 180 seconds of each side of the specimen. And for the third group (G-III), the post-curing was accomplished using dry heat for 125±2°C in the conventional oven for drying and sterilizing applications (EL-1.1 Plus – Odontobras, Brazil) (Figure 3), for 600 seconds, see Table 2.

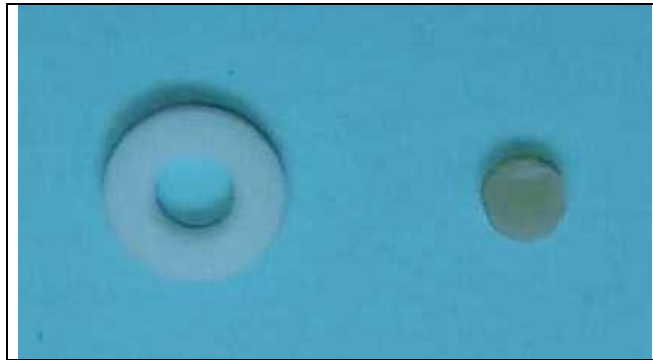


Figure 1 – PVC mold, with a cylindrical opening of 5.0mm in diameter and 2.0mm in thickness, and the composite specimen

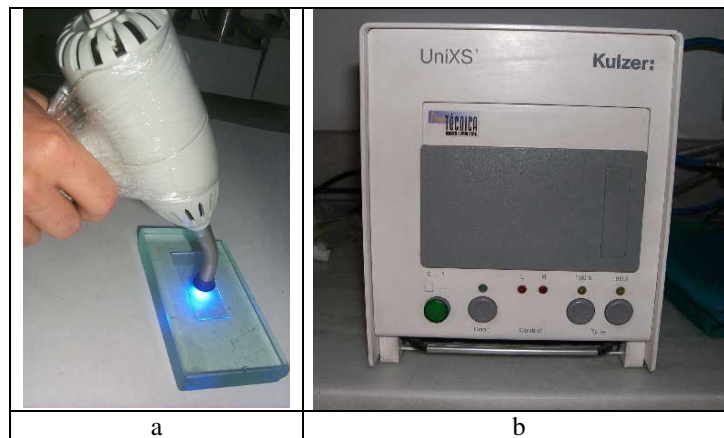


Figure 2 – Photo polymerization, (a) conventional photo polymerization equipment (Optilight) with a light guide of 10mm inner diameters and an output intensity of 500mW/cm², (b) laboratory light curing unit UniXSTM with 270W power



Figure 3 – The conventional oven for drying and sterilizing applications (EL-1.1 Plus), using in dry heat

2.1.2. Artglass

Five specimens were prepared with Artglass™ resin, the fourth group (G-IV), the top and bottom surfaces were then light polymerized, by curing using a laboratory light curing unit with 270W power (UniXS™ – Heraeus Kulzer) (Figure 2 b), for 180 seconds of each side, in accordance with the manufacturer recommendation, see Table 2.

Table 2 – The curing method using, pre-curing and post-curing, for composite resin Filtek P60™ (G-I, G-II, G-III) and Artglass™ (G-IV)

Composite	Pre-curing	Post-curing
G I	light curing 500mW/cm ² for 80 seconds	–
G II	light curing 500mW/cm ² for 80 seconds	laboratory light curing unit 270W for 360 seconds
G III	light curing 500mW/cm ² for 80 seconds	125±2°C in the oven, for 600 seconds
G IV	laboratory light curing unit 270W for 360 seconds	–

After polymerization, all specimens was placed in a light proof container with de-ionized water at 37±1°C for 24 hours. This procedure is used in order to complete post irradiation hardening and to simulate the effect of the aqueous oral environment (Tanoue, 2000 and 2003). After this procedure all specimens was stored in a dark container at room temperature until the microhardness test.

2.1.3. Vickers hardness test

The top surfaces of each specimen were ground with a series of silicon carbide (SiC) paper (600, 1200 and 2000-grit) and polished with felt and diamond paste (3µm and 1/4µm) to produce smooth and uniform surfaces. The Vickers hardness number was determined using a microhardness tester (Durimet – Ernst Leitz, GMBH, D-6330, Wetzlar, Hannover, Germany). The Vickers hardness number (HV) was calculated after application of a 50 gram-force loading for 15 seconds of dwell time (ASTM E-384, 1999). The Vickers microhardness average was determined using five indentations.

Vickers hardness test were performed, in order to analyze the post-curing effect on the mechanical properties of the direct/indirect hybrid composite resin (Filtek P60™), and compare to indirect composite resin (Artglass™). All microhardness data were then subjected to Variance Analysis (ANOVA) at a significant level of 0.05 to determine the differences among Vickers microhardness average value, and the Duncan's test to investigate inter-group differences of the Vickers microhardness average value.

3. Results

Table 3 presents the average results of the Vickers microhardness test (50 gram-force for 15 seconds), and the standard error (SE) of the five specimens, Filtek P60™ resin using conventional polymerization (G-I), post-curing using light and heat method (G-II and G-III), and Artglass™ resin using conventional polymerization (G-IV).

Table 3 - The means of Vickers microhardness number (HV) (50 gram-force for 15 seconds) and standard error (SE), as a function of the curing method for direct/indirect hybrid composite resin (Filtek P60™) (G-I, G-II, G-III) and indirect composite resin (Artglass™) (G-IV)

Group	HV (50gf)	SE
G-I	98.07	1.12
G-II	109.03	1.48
G-III	106.46	0.80
G-IV	59.45	0.45

The Filtek P60™ resin when polymerized using a conventional light curing method produced HV=98.07 with standard error SE=1.12, the post-curing resulted in higher Vickers hardness values, using a laboratory light curing unit for 360 seconds produced HV=109.0 and SE=1.48, and in the oven heat at 125±2°C for 600 seconds produced HV=106.46 and SE=0.80.

The Artglass™ resin when polymerized using a laboratory light curing unit for 360 seconds produced HV=59.45 and SE=0.45.

The ANOVA point out a significant difference among the four groups. The Duncan test was used for subsequent multiple comparisons. Significance level was always set at 95%. Table 4 shows the differences among the groups. It can be verified that groups G-II and G-III are equal, but are different from G-I and G-IV. The Vickers hardness values of the groups G-II and G-III are bigger than G-I, and much bigger than G-IV.

Table 4 –Statistic analysis. The subgroups (2 and 3) identified by different colors (G-I and G-IV) are significantly different ($p \leq 0.05$). The subgroups (1) identified by the same color (G-II and G-III) are not significantly different ($p > 0.05$)

Group	Subgroup		
	1	2	3
G-II			
G-III			
G-I			
G-IV			

4. Discussion

In this paper, the post-curing polymerization performance of a prosthetic composite material (Filtek P60TM) using a laboratory light curing unit and a conventional oven heat was evaluated by Vickers microhardness test.

Table 3 shows than the means Vickers microhardness value (HV) of Filtek P60TM resin using an additional laboratory light curing polymerization or heat post-curing methods resulted in higher Vickers hardness value when compare to the conventional polymerization method, and still higher Vickers hardness than ArtglassTM to the conventional polymerization method.

Vickers hardness for Filtek P60TM increased with post-curing treatments, in a good agreement with the open literature (Rueggeberg, 1988, Khan, 1993, Park, 1996, Park, 1996, Yamaga, 1995).

In Table 4 shows that the Filtek P60TM resin, with an additional laboratory light curing polymerization or heat post-curing methods, the hardness results had not shown significant difference among the groups.

The results indicated that the Vickers microhardness value using post curing light (laboratory light curing unit) or heat (conventional oven) had similar effect.

5. Conclusions

In accordance with the methodology used in this study, it is possible to conclude:

- The direct/indirect composite resins (Filtek P60TM) presented higher mechanical properties (Vickers microhardness), before post-curing treatments.
- The post-curing method produced a similar increase in the Vickers microhardness values for both methods, showing that increase in hardness is independent of the post-curing treatments (laboratory light curing or heat in oven).
- Filtek P60TM using a conventional light curing or the post-curing resulted in higher Vickers microhardness than the ArtglassTM using a laboratory light curing unit for 360 seconds.
- The employment of post-curing using heat methods on direct/indirect composite Filtek P60TM resulted in similar mechanical properties obtained using a laboratory light curing unit, this gives dental professionals an important clue, because the use of some equipment normally present in their offices can provide indirect composite restorations with good mechanical properties. and resulting in great reduction in treatment costs. The laboratory light curing unit is much more expensive than conventional oven for drying and sterilizing applications.

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7. References

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