ABSTRACT

Objective: The objective of this study was to compare the conversion degree of six different composite materials (Filtek Z 250, Filtek P60, Spectrum TPH, Pertac II, Clearfil AP-X, and Clearfil Photo Posterior) using three different light sources (blue light-emitting diode [LED], plasma arc curing [PAC], and conventional halogen lamp [QTH]).

Methods: Composites were placed in a 2 mm thick and 5 mm diameter Teflon molds and light cured from the top using three methods: LED for 40 s, PAC for 10 s, and QTH for 40 s. A Fourier Transform Infrared Spectroscopy (FTIR) was used to evaluate the degree of conversion (DC) (n=5). The results were analyzed with two-way analysis of variance and Tukey HSD test.

Results: DC was significantly influenced by two variables, light source and composite (P<.05). QTH revealed significantly higher DC values than LED (P<.05). However, there were no significant differences between DC values of QTH and PAC or between DC values of LED and PAC (P>.05). The highest DC was observed in the Z 250 composite specimens following photopolymerization with QTH (70%). The lowest DC was observed in Clearfil Photo Posterior composite specimens following photopolymerization with LED (43%).

Conclusions: The DC was found to be changing according to both light sources and composite materials used. Conventional light halogen (QTH) from light sources and Filtek Z 250 and Filtek P 60 among composite materials showed the most DC performance. (Eur J Dent 2013;7:102-109)

Key words: Degree of conversion; monomer; composites; light sources; FTIR

INTRODUCTION

Despite considerable improvements in dental composite materials, present-day composites still suffer from inadequate degree of conversion and problems with marginal adaptation. Inadequate polymerization results in inferior physico-mechanical properties, such as poor resistance to wear, poor color stability, secondary caries and adverse tissue reactions, increased rates of water...
sorption, solubility, and early restoration failure.\(^2\)\(^-\)\(^7\) Therefore, adequate polymerization is a crucial factor in obtaining optimal physical properties and a satisfying clinical performance of composites.\(^8\) To convert all of monomer to polymer during the polymerization reaction in a dental resin composite, the ideal condition expected. However, all di-methacrylate monomers exhibit considerable residual monomer in the final product, with a degree of conversion (DC) ranging from 55% to 75% under conventional irradiation conditions.\(^8\)\(^-\)\(^11\) The reason for this is believed to be limitations on the mobility of reactive groups imposed by the rapid formation of a cross-linked polymeric network.\(^12\) Low degree of monomer conversion might cause increased cytotoxicity,\(^13\) reduced hardness,\(^14\)\(^-\)\(^16\) increased wear, increased marginal breakdown,\(^17\) and possibly increased microleakage with resultant recurrent caries and pulpal irritation.\(^18\)

A number of methods have been used to determine the DC, with differential thermal analysis\(^19\),\(^20\) and gas chromatography\(^21\) the most commonly reported. Among these methods, Fourier transformation infrared spectroscopy (FTIR) has proven to be a powerful technique and widely used as a reliable method\(^15\),\(^20\),\(^22\),\(^23\) as it detects the C=C stretching vibrations directly before and after curing of materials. However, the procedure of measuring the DC of bulk composite by FTIR is time consuming because the polymerized specimens need to be pulverized.\(^20\)

The most widely used light sources for photo activating resin-based composite are light-emitting diodes (LED) and quartz tungsten halogen (QTH) lights. However, heat generation can cause deformations in the pulp, a major disadvantage of using the QTH as a light source.\(^24\) These types of light sources usually operate at light intensities between 400 and 800 mW/cm\(^2\) and polymerize a composite within 40 seconds at depths of up to 2 mm.

A plasma arc curing (PAC) light is also designed for high-speed curing of composite filling materials in direct resin restorations. A high-energy, high-pressure ionized gas in the presence of an electrical current is used to create a high temperature light source strong enough to increase the curing rate of composite resins. As the manufacturer ADT [San Carlos, CA, USA] has stated, highly filled and pigmented composite materials can be cured in 10 s, while more transparent materials can be cured within 5 s. This rapid-curing feature saves considerable chair-side time when compared with QTH. However, compared to QTH, the PAC devices are much more expensive. Further, concerns have been raised regarding radiation heating created by the high light intensity and negative side effects of the rapid polymerization shrinkage, which may compromise the marginal seal of restorations.\(^25\)\(^-\)\(^28\)

Light-emitting diodes (LED) feature very narrow spectral ranges and are highly efficient light sources.\(^27\) The high efficiency of LED allows the development of battery-powered cordless lights and eliminates the need for cooling fans.\(^27\) The narrow bandwidth of emitted radiation\(^29\) should be optimally suited for activating camphorquinone, but alternative photo-initiators absorbing at shorter wavelengths likely will be insufficiently activated. Heating of the irradiated objects by LED lights is expected to be minimal.\(^1\)\(^-\)\(^27\)

In studies related to DC, Tarle et al\(^1\) indicated that the QTH results revealed significantly higher degree of conversion value than those using PAC and LED. However, Yoon et al\(^8\) showed that when the same light energy was irradiated, DC by plasma arc and LED were not significantly different from QTH.

For light-cured resin composites, the DC of resin composites depends on the chemical structure of the monomer, filler size, shade and translucency, sufficient light intensity, curing time, distance from light source, thickness of the increment, and polymerization conditions, including atmosphere, temperature and photoinitiator concentration.\(^30\)\(^-\)\(^32\) Nevertheless, the relative contributions of these variables to the degree of conversion are still unknown; this information might be regarded as essential for the development of new strategies to increase DC. Furthermore, the effects of these variables on degree of conversion in composite resins still need to be determined. The objective of this study was to investigate the effect of some variables on the degree of conversion. Six different composite materials (Filtek Z 250, Filtek P60, Spectrum TPH, Pertact II, Clearfil AP-X, and Clearfil Photo Posterior) were illuminated with three different light sources [blue light-emitting diode (LED), plasma arc curing (PAC), conventional halogen lamp (QTH)], and the DCs obtained
from these curing procedures were compared using FTIR. The null hypothesis tested was that both light sources and composite resins would affect the degree of conversion.

**MATERIALS AND METHODS**

In this study, six commercially available light-cured resin composites were used. The list of composites, types, shades, and manufacturers are given in Table 1.

Three different light sources were used and evaluated with the above-mentioned composites (Table 2). The outputs of the light tips of the QTH (Hilux) and LED (Elipar Freelight) curing units were measured by a digital curing radiometer (Demetron, Danbury, CT, USA) (Table 2). The output of the PAC (Power PAC) system, which could not be measured by the curing radiometer, was 1200-1500 mW/cm² according to the manufacturer’s instructions.

Composites were placed in a space 5 mm in diameter by 2 mm high within a polytetrafluoroethylene mold. A transparent Mylar strip (0.07 mm; Du Pont Company, Wilmington, DE, USA) was placed on the top and bottom, and excess material was extruded by squeezing it between two microscope slides. The slides were then removed and the mold placed on a black background. Afterward, the tip of the radiation guide was applied to the Mylar strip on the top of the mold aperture. The samples were then irradiated according to the manufacturers’ instructions as follows: 40 s with QTH, 10 s with PAC, and 40 s with LED from the top of the mold. The light intensity of the curing unit was checked prior to the fabrication of each sample set using the external radiometer. Specimens were stored in lightproof boxes after the polymerization procedure to avoid further exposure to light. Five specimens were prepared for every combination of light source and composite luting material. The total number of specimens was 180.

A Fourier Transform Infrared Spectroscopy (FTIR) (1600 Series; PerkinElmer, Wellesley, MA, USA) was used to evaluate the conversion degree. Each specimen was pulverized into a fine powder with a mortar and pestle. Fifty micrograms of ground powder was mixed with 5 mg of potassium bromide powder (Carlo-Erba Reagents, Milan, Italy), and the absorbance peaks were recorded using the diffuse-reflection mode of FTIR. Spectra were also acquired from the same number of unpolymerized adhesives. For this, unpolymerized pastes were smeared onto thin potassium bromide discs. The amount of double vinyl bonds remaining in the specimen exposed to irradiation is shown by the intensity of the peak at 1637 cm⁻¹.

Table 1. Materials evaluated and their specifications.

| Composites Shade Composition  | Filler weight Manufacturer |
|-------------------------------|---------------------------|
| Filtek Z-250 (FZ250) B2 Zirconia/silica *BisGMA, **UDMA, ***Bis-EMA 77,4(wt) 3M ESPE, St. Paul, USA |
| Filtek P60 (FP60) B2 Zirconia/silica *BisGMA, **UDMA, ***Bis-EMA 83(wt) 3M ESPE, St. Paul, USA |
| Spectrum TPH [TPH] B1 Bariumaluminiumboronsilicate glass, highly dispersed silicon dioxide *BisGMA, **UDMA, ***Bis-EMA 77(wt) Dentsply, Konstanz, GERMANY |
| Pertac II [PII] B2 Ultra-fine milled quartz, highly dispersed silicon dioxide, yttrium fluoride *BisGMA 80(wt) ESPE, Seefeld, GERMANY |
| Clearfil AP-X (CAP-X) B2 Silanated Barium Glass, Silanated silica, Silanated Collaidal silica *BisGMA, ****TEGDMA 80 [wt] Kuraray, Osaka, JAPAN |
| Clearfil Photo Posterior [CPP] B2 Silanated Barium Glass, Silanated silica, Silanated Collaidal silica *BisGMA, ****TEGDMA, **UDMA 84,7[w] Kuraray, Osaka, JAPAN |

*BisGMA, bisphenol-A-glycidyl methacrylate. **UDMA, urethane dimethacrylate. ***BisEMA, ethoxylated bisphenol-A-glycidyl methacrylate. ****TEGDMA, triethylene glycol dimethacrylate.

Table 2. Light sources used in this study.

| Materials Brand | Power Density | Diameter of the tip (mm) | Manufacturer |
|-----------------|---------------|--------------------------|--------------|
| Hilux           | 500 mW/cm²    | 10                       | Express Dental Products, Toronto, Canada |
| Power PAC       | 1200-1500 mW/cm² | 6,5                      | ADT, San Carlos, CA, USA |
| Elipar Freelight| 400 mW/cm²    | 8                        | 3M Espe, St. Paul, MN, USA |
referring to the C=C stretching of the vinyl group and have been used in the study of polymerization of acrylates and methacrylates.\textsuperscript{33} The DC was directly related to the decrease of 1637 cm\textsuperscript{-1} absorption on the FTIR spectra as follows: DC = ((A0 - At)/ A0) x 100, where A0 is the absorption of the peak at 1637 cm\textsuperscript{-1} when time is equal to 0 and At is the absorption at time.\textsuperscript{11}

**Statistical Analysis**

Intercepts between DC of composite resin and light sources were analyzed with two-way ANOVA. Multiple comparisons were made by using Tukey HSD Post Hoc test. Statistical significance was considered as P<.05.

**RESULTS**

Table 3 and Figure 1 summarize the mean and standard deviations of the degree of conversion for six composite materials after illumination at 40 s with QTH and LED, and at 10 s with PAC.

Two-way ANOVA performed on the degree of conversion revealed that DC was influenced by light sources (P<.05), composite materials (P<.05). Degree of conversion was also affected by interaction between the light sources and composite resin (P<.05). Generally, Tukey HSD indicated that DC of composites illuminated by QTH was higher than LED, regardless of composites (P<.05). There was no significance between QTH and PAC or between LED and PAC (P>.05) (Figure 2).

Among the six materials, the highest DC was obtained from Filtek Z 250 and Filtek P60 regardless of the light sources (P<.05). The lowest DC was obtained from Clearfil Photo Posterior (P<.05).

Tukey HSD also revealed significant differences in mean DC values produced by different light sources and composite resins combinations (P<.05). The highest DC was obtained for Filtek Z 250 composite material cured by QTH. The lowest DC was for Clearfil Photo Posterior irradiated by LED.

**DISCUSSION**

The degree of conversion of six commercially available hybrid composites (TPH, Filtek Z 250, Pro60, Pertact, Clearfil Photo Posterior, and Clearfil AP-X) after being irradiated with three different light sources (blue light-emitting diode [LED], plasma arc curing [PAC], and conventional halogen lamp [QTH]) was measured using FTIR.

![Figure 1. DC of composite resins after irradiated with QTH, PAC and LED](image1)

![Figure 2. DC of light sources regardless of composite resins.](image2)

![Figure 3. DC of composite resins regardless of light sources.](image3)

| Composites | QTH | LED | PAC |
|------------|-----|-----|-----|
| FZ250      | 68.40 ± 3.9 a A | 69.60 ± 6.1 a A | 67.20 ± 7.2 a A |
| P660       | 67.20 ± 7.2 a A | 68.40 ± 6.1 a A | 67.40 ± 6.1 ab A |
| TPH        | 59.80 ± 3.1 a A | 54.60 ± 5.3 bcd B |
| PII        | 43.80 ± 4.3 b B | 54.60 ± 5.3 bcd B |
| CAP-X      | 43.80 ± 7.4 b C | 55.40 ± 5.0 bcd B |
| CPP        | 48.00 ± 5.5 d B | 55.40 ± 5.0 bcd B |

Means followed by the same small letter in the column and capital letter in the row indicate no statistical difference (P>.05).

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The results revealed the following: Filtek Z 250 and Filtek P60 showed a higher degree of conversion compared to other composite materials regardless of the light sources. Clearfil Photo Posterior showed the lowest DC in comparison to the other materials; however, there were no significant differences among the DCs of TPH, Pertact II, and Clearfil AP-X. The results also indicated that QTH showed higher DC than LED regardless of materials. There were no significant differences between QTH and PAC or between LED and PAC. The highest DC was for the Filtek Z 250 composite material irradiated with QTH. The lowest results registered were for the Clearfil Photo Posterior illumination with LED. The hypothesis for the emergence—that both light sources and composite resins would be effective on degree of conversion—was accepted.

In the present study, the colors of the materials were similar (B1, B2) for all composites tested. In addition, factors influencing the transmission of light—such as thickness of the restorative material and distance of light tip to the restoration surface—were standardized. The only changes were in the composite materials and light sources. Hence, in the present study, any differences in DC can be attributed to differences in the composition of materials and the illumination characteristics of the light units.

Nomoto and Hirasawa34 stated that the depth of cure is dependent upon the light permeability of the filler as well as the monomer composition and type and concentration of initiator, inhibitor, and accelerator in the resin materials. The composite resins used in this study include some different inorganic filler types, sizes, loading, and monomer composition (Table 1). The higher DC value that was obtained for Filtek Z 250 and Filtek P60 might be partly explained by the filler type because they contain filler different from the other composites (Table 1). Another reason for the higher degree of conversion seen in fully cured Filtek Z250 and Filtek P60 composite could be explained by the presence of UDMA and Bis-EMA(6) in the organic structure of these materials. In Filtek Z250 and Filtek P60 restorative, the manufacturer had replaced a majority of TEGDMA with a blend of UDMA (urethane dimethacrylate) and Bis-EMA(6)1 (Bisphenol A polyethylene glycol diether dimethacrylate). Both of these resins are of higher molecular weight and, therefore, have fewer double bonds per unit of weight. Emami and Söderholm39 also suggested that a higher conversion level of Filtek Z 250 might have been related to partial substitution of the relatively stiff and hydrogen-bonded Bis-GMA molecules with the longer, more flexible Bis-EMA(6) molecules.

The ratio of filler relative to resin is also important: The higher the proportion of filler, the more difficult it is for the composite to cure.8 The percentage of filler in the resin composites in these investigations ranged between 70% and 84.7% by weight. Although it is claimed that the type, size, and percentage of filler in resin composite might influence polymerization and DC, the results of this study indicated that the DC might not be dependent on the percentage of filler: Clearfil AP-X (80%), and Clearfil Photo Posterior (84.7%)—approximately the same ratio of filler with other composites—revealed lower DC than did Pertac II (80%), Spectrum TPH (77%), Filtek Z 250 (77.4%), and Filtek P60 (83%). Price and colleagues’ (2000) studies reported that the relationship between percentage of filler and light transmission was not linear. They found that Z 100, which was the most heavily filled at 84.5%, transmitted more light than did Pertac II (80%), Alert (83.5%), Filtek P 60 (83%), or SureFil (81.5%).

Another important parameter is the amount of light energy a light-activated resin composite receives.35 It has been suggested that the total energy delivered by a light-curing units remarkably influences the degree of C=C conversion and hardness in resin composites.36-39 The density of energy received by a resin composite can be calculated by multiplying the density of power from the curing light by the duration of the light exposure. Among the light sources used in this study, the QTH light source emits 20000 mj/cm² for 40 s (500 mw/cm² x 40 s), the PAC light source emits 12000-15000 mj/cm² for 10 s (1200-1500 mw/cm² x 10 s), and the LED light source emits 16000 mj/cm² for 40 s (400 mw/cm² x 40 s). Hence, the QTH light source produces a higher energy in total than PAC and LED light sources and, thus, also provoked a higher degree of conversion. However, the differences between degrees of conversion of the PAC and QTH were not statistically significant. The results indicated that other influential factors—such as type of filler, monomer, initiator, and inhibitor—might be contributing to the results. Similarly, Knezevic et al40 reported that the DC of composites (Tetric...
The polymerization of light-cured resin depends not only on the quantity of light but also on its quality such as wavelength. The intensity of 300 mw/cm² or greater in the wavelength range of 450 nm to 500 nm is needed for complete polymerization of composite up to 2 mm in depth. However, the most effective band lies within the 460 nm-480 nm range for the composite using camphorquinone as initiator, with an optimum at 468 nm. Outside this range, however, the wavelength dependence is much stronger, and the conversion rate drops rapidly. A typical halogen lamp produces greater output intensity at the longer wavelength in 300 nm-800 nm. The emission of halogen lamp designed for dental use is filtered to pass radiation of wavelengths between 400 nm and 500 nm, corresponding to the carbonyl absorbance of camphorquinone. Any wavelengths below about 430 nm and above 500 nm are not utilized in the electron promotion of the ketone groups, as camphorquinone ignores these wavelengths. The unwanted wavelengths do produce additional heat, affecting the kinetics of the reaction and, in so doing, might influence the reaction. This is why it is necessary to use the curing units for 40 s to get the same degree of efficiency. No filters are required in LED units because the spectral output of LED falls conveniently within the absorption spectrum of the camphorquinone photo initiator (470 nm). The PAC units are characterized by a high output of light energy at rather narrow wavelengths of around 470 nm. The spectral output from some PAC curing lights might not match the photo-initiator in some resin composites, which may then fail to polymerize. Assuming that all materials employ camphorquinone as photo initiator, the difference may be associated with the use of different amines, forming complexes with camphorquinone of different absorption characteristics. As we could not obtain the photo-initiator contents of the composites, we consider that the amine accelerators used in the investigated resin composites might well be of different chemical compositions. Another reason might be the existence of different photo-initiators chemistry used within these composites. The level of conversion change in the composite resins has revealed that each composite contains different photo-initiators and has variability in maximum conversion degree at a different wavelength or light energy level. Therefore, the manufacturers of resin...
composites should provide information on the type of photo initiator used or else all resin composites should carry a label stating the energy density and spectral bandwidth required to polymerize the resin composite. It would then be possible to select the appropriate curing light and calculate how long it takes to irradiate the composite.  

**CONCLUSIONS**

Within the limitations this study, the following conclusions can be drawn:

1. The degree of conversion was found to be dependent upon both light source and composite materials.
2. The results showed that the DC array of composite materials did not change according to light sources; the characteristics of composites were more important than the light source type in change of DC.
3. The study also indicated that the DC of QTH, having a higher total energy, shows higher DC values than PAC and LED, having a lower light energy. Therefore, the amount of total energy the composite receives is also a secondary significant determinant for DC.

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