



The effect of different photoactivation sources on a nanocomposite resin: LED, halogen and argon laser application

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ABSTRACT. This paper evaluates the effects of different light sources on the monomer conversion (DC%) and Vickers microhardness number (VHN) of a nanocomposite resin. Three different depths were measured in this study (1, 2 and 3 mm), and photoactivation methods including a halogen lamp, a LED and an argon ion laser were used. In contrast to other studies, the conditions for each photoactivation source such as the exposure time and the same power density were considered and kept the same. It was also shown that increasing the depth lowered the VHN. At each of the depths, it was observed that the three light sources behaved similarly. The correlation between the VHN and DC% is shown. However, only a weak correlation was observed, which implies that a higher DC% value does not necessarily indicate a higher VHN.

Keywords: photopolymerization, mechanical properties, infrared spectroscopy, composite resin.

O efeito de diferentes fontes de fotoativação em uma resina de nanopartículas: aplicação de LED, halógeno e laser de argônio

RESUMO. Este artigo avalia os efeitos das diferentes fontes de luz no grau de conversão monomérica (DC%) e microdureza Vickers (VHN) de uma nanoresina composta. Três profundidades diferentes foram medidas neste estudo (1, 2 e 3 mm), enquanto que foram utilizadas três diferentes fontes de luz: uma lâmpada halógena, uma LED e um laser de argônio. Em contraste com outros estudos, foram mantidas as mesmas condições para cada fonte de fotoativação, o mesmo tempo de exposição, assim como a mesma densidade de potência de cada fonte de luz. Com o aumento da profundidade há diminuição dos valores de VHN, e para as diferentes fontes de luz foram observados valores similares de DC% para cada profundidade. Analisando a correlação entre os valores VHN e DC% o que se observa é apenas uma fraca correlação entre ambos os valores, o que implica que altos valores de DC% não é necessariamente um indicativo de altos valores de VHN.

Palavras-chaves: polimerização, propriedades mecânicas, espectroscopia de infravermelho, resinas compostas.

Introduction

The demand for aesthetic dental restoration has increased over the years, which has, consequently, increased the use of resin composites. However, to obtain better results with these resin composites, several studies have emphasized the need to assess the optimal conditions to improve the capabilities of these resins for clinical restoration (CALIXTO et al., 2008; DENIS et al., 2012; RASTELLI et al., 2008; SAADE et al., 2009). In this regard, one of the factors that affects the efficiency of the resins is the degree of polymerization; determining the cause of insufficient curing of these resins is important because of the innumerable problems in the clinical performance associated with insufficient curing (GALVÃO et al., 2010; RODRIGUES et al., 2009; VALENTINO et al., 2011).

In dentistry, the number of different light sources used during dental resin polymerization has increased. Quartz-tungsten halogen lamps were the most common light source used in dental clinics for several years; however, because of their low efficiency with regards to heat generation (KRAMER et al., 2008), several alternatives are currently available. An alternative light source is the argon laser, which characteristically produces a high energy beam of light with an exactly controlled wavelength that can adjusted for each composite material (RASTELLI et al., 2008). Another alternative is a light-emitting diode (LED) source, which has become an easy-to-use and low-cost technology based on recent advances (KRAMER et al., 2008; VALENTINO et al., 2011). It is important to take the clinical requirements into account when working with resin-based

photopolymerization processes because of the different light cure values for different resin-based composites. This necessity emphasizes the importance of a critically evaluating the different light-curing protocols.

Both indirect and direct methods have been used to assess the DC% of composite resins. Indirect methods measure mechanical properties such as hardness and flexural tests (GONCALVES et al., 2010; TORNÓ et al., 2008). The most frequently employed methods for evaluating denture teeth and composite resin hardnesses are the Knoop and Vickers tests; however, other methods have also been used (SHAHDADA et al., 2007). Direct methods measure the DC% through either spectroscopic (FENG et al., 2009; SILVA et al., 2008) or thermal analysis (FENG et al., 2009). It is well known that direct methods more accurately measure the DC% than indirect methods.

Because of continuing advances in the development of nanocomposite resins (CHEN, 2010), the purpose of this study was to assess the effects that different light sources have on the Vickers microhardness number (VHN) and DC% properties of a nanofilled resin. The light sources studied were a LED, an argon ion laser and a halogen lamp, and variations in the depth of cure were considered here. In contrast to other studies, the conditions used for each photoactivation source, such as the exposure time and the power density, were considered and kept the same. The relationship between VHN and DC% was also evaluated in this study.

Material and methods

The Filtek™ Supreme (Nanofilled, 3M-ESPE) commercial composite resin was used in this study. The three different light sources used were a halogen lamp (Optilux 501/ Demetron-Kerr/ serial number: 5815687), a LED (Lec MMÓptics/ prototype) and an argon ion laser (INNOVA-100/Coherent/ serial number: 3240). The composite resin samples were exposed to light for 40 seconds with a power density of 600 mW cm^{-2} . The power density values were measured using a radiometer (Fieldmaster / Coherent) and calculated from the ratio of the power (in milliwatts) to the area of the light tip (in square centimeters). A USB 2000 spectrometer was used to evaluate the emission spectrum of each light source, and the spectrum of camphorquinone was obtained using a UV-Vis Cary (Varian) spectrometer and can be seen in Figure 1.

After photo-activation, the specimens prepared for FTIR spectroscopy analysis were finely

pulverized with a hard tissue-grinding machine (Marconi, model MA590).

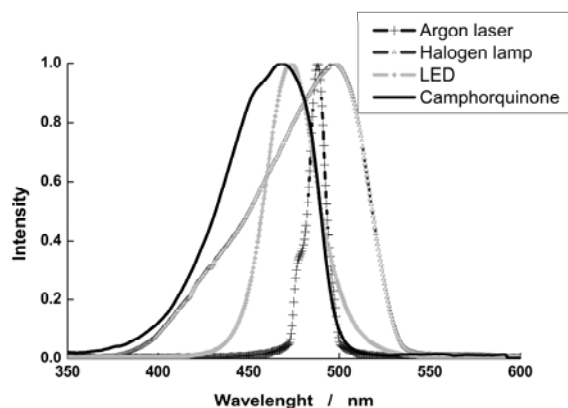


Figure 1. The wavelengths of camphorquinone and the different light sources.

The pulverized composite was maintained in a dark room until the moment of FTIR analysis. After 24h, the ground powder was mixed with KBr powder salt. The mixture was pressed to form a 15 mm diameter pellet. The pellet was then placed into an attachment in the optical compartment of a Fourier transform infrared spectrophotometer. The infrared spectra were collected using a Bomem FT-IR spectrometer (model MB-102) equipped with a TGS detector using a KBr pellet sample. For each spectrum, 64 single beam scans were averaged with a resolution of 4 cm. The DC% was calculated by using the peak intensity of the aliphatic C–C stretching vibrations at $1,637 \text{ cm}^{-1}$ (as the analytical frequency) and the aromatic C = C stretching vibrations at $1,607 \text{ cm}^{-1}$ (as the reference frequency). The infrared spectrum was measured initially on the uncured material. Then, after polymerization, the spectrum was remeasured. The monomer conversion was assessed by subtracting the percentage of remaining aliphatic C = C from 100%, i.e., the DC% can be calculated according to the following formula: $\text{DC\%} = [1 - (\text{cured}) / (\text{uncured})] \times 100$.

Three different depths were measured in this study 1, 2 and 3 mm and each of the test conditions was repeated three times. The samples were inserted into a disk-shaped Teflon matrix mold (1 mm thick \times 5 mm in diameter) that was transversely sectioned at 1-mm intervals. This mold was developed for this study to evaluate the different resin polymerization depths without further sectioning of the samples. The sample surface was placed in contact with the light-curing tip during photoactivation. The measurements were made on the top surface for the first and second sections whereas the base surface

was used for the third millimeter. In this way, the surface closest to the light source can be assessed along with both the intermediate and more distant surfaces. After the photopolymerization measurements, the samples were stored in distilled water at 37°C for 24 hours. Prior to the VHN test, the specimens were polished flat using silicon carbide paper and a Sof-Lex disc (3M ESPE, St. Paul, MN, USA) and then blotted dry.

The VHN test was performed in a VMHT MOT (Germany) using a Vickers diamond indenter with a load of 50 gf for 30 seconds. For each sample, the surfaces were impressed in each quadrant, and the mean values of the VHN values were calculated.

A statistical analysis of each parameter was performed using a two-way ANOVA for the different curing conditions. Tukey's post hoc test was employed as a multiple comparison test at $\alpha = 0.05$.

Results and discussion

The mean values of the different light sources are presented in Figure 2 and 3 for each specific depth studied. Both Tukey's test and a variance analysis ($p < 0.05$) were used to investigate the effects of the light sources, the depth and the interactions between the results. Table 1 shows the statistical analysis performed for the VHN test and the DC% results.

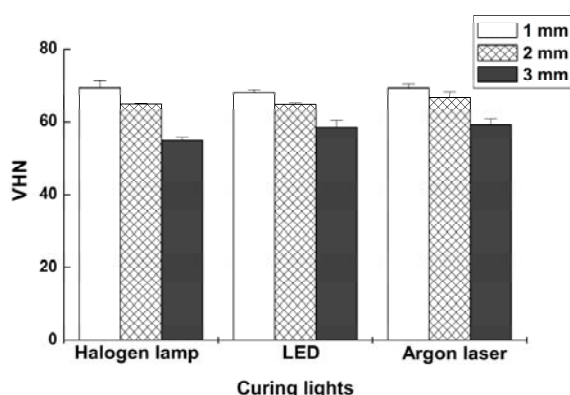


Figure 2. The influence of depth on the VHN values for each light source.

From the results in Figure 3, it can be seen that composite resins with thicknesses of 1 and 2 mm have very similar VHN values for the different light sources. The divergence between the LED and argon ion laser is less than 2% when comparing the resins with either a 1- or 2-mm thickness. Nevertheless, this difference increases for the results from the 3-mm-thick sample, indicating that the depth has an effect on VHN values. At a depth of 3 mm, the LED- and argon ion laser-treated samples

possess VHN values almost 10% greater than the samples exposed to the halogen lamp.

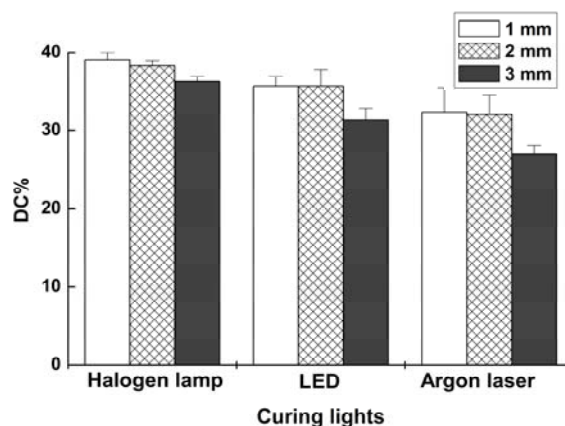


Figure 3. The influence of depth on the DC% values for each light source.

Table 1. The two-way analysis of variance of DC% and VHN that considers the different light sources and depths.

| Groups | DC% | | VHN | |
|--------------|------|---------|-------|---------|
| | F | p-value | F | p-value |
| Light source | 37.2 | 0 | 4.6 | 0.02 |
| Depth | 13.2 | 0 | 162.2 | 0 |
| Interaction | 0.4 | 0.74 | 2.9 | 0.04 |

Rode et al. (2009) investigated a nanofilled resin composite and observed that LEDs and argon ion lasers produced different microhardness values, which contradicts with the results obtained in this study. In addition, some studies have suggested that a thickness of up to 1 mm would be sufficient to obtain an adequate hardness and DC% (BALA et al., 2005; PERIS et al., 2005; RODE et al., 2009), whereas other investigations have found no significant differences between the hardness and DC% for thicknesses up to 3 mm (OBICI et al., 2005). In general, higher VHN microhardness values are indicative of an increase in the elastic modulus of the resin composite. Our VHN values ranged from 52.5 to 73.3, which is higher than the range observed by Galvão et al. (2010) and an intermediate result when compared with the results of Sharkey et al. (2001) and Rode et al. (2009) (these authors observed values from 25.4 to 186.4). Although Craig and Powers (2002) considered VHN values below 50 to be ideal for avoiding a small resistance to fractures, there has not, so far, been a consensus on the ideal value for the VHN, which may be due to the high correlation between the resin composition and the inorganic filler content (CORCIOLANI et al., 2008; SOARES et al., 2007).

Several important observations can be made based on the statistical analysis presented in Table 1.

It was also shown that increasing the depth lowers the VHN value. From the VHN analysis, it is possible to observe that the results obtained using the 3-mm-thick samples are significantly different from the other two. In contrast, the three light sources behaved similarly at each of the depths. Statistically significant differences are shown for some interactions such as the interaction when VHN result is compared from 3-mm-thick samples exposed to the halogen lamp and argon ion laser light sources. In contrast, no significant difference was observed between the halogen lamp and LED results.

Statistically significant variations in the DC% values are also shown in Table 1 for both the thickness and light sources. To detect where these differences occur a multiple comparison test, the Tukey's test, was applied to the light-curing results. According to the Tukey test, the argon ion laser possesses the lowest significant difference, followed by the LED and the halogen lamp. Nevertheless, the data in Table 1 do not indicate that there was any interaction between the light sources and the sample thickness.

The Filtek Supreme resin composite uses camphorquinone as the photoinitiator. As shown in Figure 1, camphorquinone possesses a broad band between 370 and 510 nm with a maximum at 468 nm. The halogen lamp also exhibits a broad band in the region from 340-540 nm with a peak at 497 nm. In contrast, the argon ion laser has a narrow band ranging between 470 and 500 nm with a peak at 488 nm, and the band for the LED ranges from 430 to 520 nm with a peak at 473 nm. In general, the light source with the emission wavelength closest to the camphorquinone should exhibit the highest capacity to activate the photoinitiator and, consequently, show the highest DC% value. Although the peak emission closest to the photoactivation peak of camphorquinone is from the LED light source, the highest DC% value was observed for the halogen lamp. It is possible to attribute this result to the larger overlap between the halogen lamp and camphorquinone curves (OBICI et al., 2006). Another factor that may explain the excellent performance of the halogen lamp is that this photoactivation source produces a temperature increase that causes complementary polymerization in the resin composite (GUIRALDO et al., 2008; HANSEN; ASMUSSEN, 1993). Both the differences between the light sources and the high value from the halogen lamp are in good agreement with other studies (GUIRALDO et al., 2008; KURACHI et al., 2001). In contrast, the low efficiency of the argon ion laser may be a

consequence of the nearly 20-nm redshift of its emission peak from the camphorquinone peak. As other studies have demonstrated, the high polymerization efficiencies of LED and argon ion laser light sources are primarily caused by the difference in the employed intensities (BALA et al., 2005; VARGAS et al., 1998).

The restriction imposed by polymer vitrification (STANSBURY; DICKENS, 2001) limits the conversion of the double bonds to values below 100% (ANDRZEJEWSKA, 2001; CHUNG; GREENER, 1990), and the value that would be sufficient for clinical environments has been of some concern. Stansbury and Dickens (2001) and Chung and Greener (1990) have shown that DC% values that range from 55 to 75% and from 43.8 to 73.8%, respectively, are considered safe levels for resin composites. However, there is no consensus on what level would be safe for an adequate clinical restoration (RASTELLI et al., 2008; SOARES et al., 2007). As shown in Figure 3, the DC% values of the 1-mm-thick resin are 39, 37 and 32% using the halogen lamp, LED and argon ion laser, respectively, and the differences between the 2- and 3-mm thickness are also very small. It should be noted that our results are lower than the suggested safe levels (ANDRZEJEWSKA, 2001; CHUNG; GREENER, 1990), which may be because of the small energy density of the light sources employed in this work.

There are many possible explanations for the similar results for DC%. (i) In this study, the same power density and the same photoactivation time were used for all of the light sources. Many researches agree that the DC% value increases linearly both with the energy density (HALVORSON et al., 2002; INUE et al., 2005; PEUTZFELDT et al., 2000), and the photoactivation time (RASTELLI et al., 2008). Nevertheless, using identical conditions for all three light sources may prevent the results from showing the large discrepancies between the different photoactivation sources seen in several other studies (OBICI et al., 2005, 2006). (ii) Another possible explanation may be related to the nanocomposite resin composition. The Filtek Supreme resin used in this investigation is a combination of monodispersed, nonagglomerated and aggregated silica particles of 20 and 75 nm in size. However, the average size of the agglomerated particles is between 0.6 and 1.4 μm . Therefore, it may be that the smaller size is unable to scatter light, and the light does not penetrate significantly into the nanofilled resin (MENDES et al., 2005; TURSSI et al., 2005). (iii) Another potential reason the similar results may be that the high surface area of the nanofilled agglomerates may be close to the light-curing wavelength (RODRIGUES JUNIOR

et al., 2008), which would also explain the small decrease in the DC% value from the halogen lamp to the argon ion laser.

The correlation between the VHN and DC% values was also performed. The correlation between these two properties was measured by calculating the Pearson correlation coefficient (r) at the 5% significance level. The Pearson coefficient and Tukey's test were 0.32 and 0.402, respectively. In general, a strong correlation is detected when $0.7 < r \leq 1$, while a weak correlation is observed for $0.4 < r < 0.7$. Nevertheless, no correlation was detected between these two variables, which implies that a higher DC% does not necessarily indicate a higher VHN value. In fact, the very weak correlation between these two properties may be explained by the high monomer conversion not leading to a high density of cross-linked bonds. Another important aspect is that the mechanical properties are highly associated with the polymeric network density, which is not directly related to the monomer conversion. Therefore, this strong correlation coefficient between DC% and VHN microhardness cannot be assumed to be true for all experimental conditions.

Conclusion

In this study, similar results for the DC% were observed. One of the primary reasons for this was the similar DC% value obtained for the different light sources, which may be attributed to (a) the application of identical power densities by all the light sources, (b) the composition of the nanocomposite resin, (c) the surface area of the nanofilled agglomerates and (d) the identical light-curing time employed for each light source. It was also observed that the argon ion laser results possessed the lowest significant difference, followed by those for the LED and then the halogen lamp. In addition, a strong correlation between the VHN and DC% values was not observed, in contrast to previous findings in the literature. Based on our results, a careful analysis should be performed before assuming that a surface mechanical property describes the behavior of the bulk material.

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