Degree of conversion and hardness of an orthodontic resin cured with a light-emitting diode and a quartz–tungsten–halogen light

Guilherme Pessôa Cerveira*, Telmo Bandeira Berthold*, André A. Souto**,
Ana Maria Spohr* and Ernani Menezes Marchioro*
*Preventative Department, School of Dentistry and **School of Chemistry, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

SUMMARY The purpose of this study was to assess the influence of two light units, a quartz–tungsten–halogen (QTH) and a light-emitting diode (LED), on the hardness and degree of conversion of an orthodontic composite resin. Sixty specimen disks were prepared from Transbond XT composite resin (3M Unitek) and light cured for 10, 20, and 30 seconds with a QTH (Curing Light XL 3000, 3M Unitek) or a LED (Ortholux, 3M Unitek) light-curing unit for 5, 10, and 15 seconds. Composite resin polymerization was evaluated by Fourier-transform infrared (FTIR) spectrophotometry and Knoop hardness number (KHN). The results were statistically analysed using analysis of variance and Tukey's multiple comparisons test ($\alpha = 0.05$).

The highest KHN was obtained with the QTH at 30 (25.19 KHN) and 20 (24.01) seconds, which did not differ statistically, and in the LED 15 second (21.86) group. The QTH 10 second group (20.53) did not differ statistically from the QTH 20 second or the LED 5 (19.96) and 15, or 10 second (18.95) groups. According to FTIR, there was no statistical difference for the degree of conversion among the groups, QTH 10 (43.42 per cent), QTH 20 (46.12 per cent), QTH 30 (45.30 per cent), LED 10 (47.02 per cent), or LED 15 (47.24 per cent) seconds. The lowest degree of conversion was obtained for the LED 5 second group (38.97 per cent), which did not differ statistically from the QTH 10 second group.

Light curing with the LED resulted in a reduction of 50 per cent in the time recommended for use of the QTH light with the composite resin, Transbond XT.

Introduction

There have been many advances in dental resin light activation processes, such as the introduction of light-emitting diode (LED) technology (Mills *et al.*, 1999). One advantage attributed to the LED is the coincidence of peak irradiance of LED light with camphorquinone, a photoinitiator agent commonly found in composite resin formulations used in dentistry (Nicholls, 2000; Hammesfahr *et al.*, 2002; Swift, 2002; Bennett and Watts, 2004; Uhl *et al.*, 2004; Wiggins *et al.*, 2004; Bala *et al.*, 2005). Other advantages resulting from the use of LED are the possibility of a reduced curing time (Bishara *et al.*, 2003; Wiggins *et al.*, 2004), a lamp duration time of approximately 10 000 hours (Mills *et al.*, 1999), no heat generation, and resistance to impacts (Mills *et al.*, 1999; Duke, 2001; Dunn and Taloumis, 2002). In addition to this, the LED appliance consumes minimal power and can be run on rechargeable batteries, allowing it to have a lightweight, ergonomic design (Wiggins *et al.*, 2004).

Hardness tests have been used to assess composite resin. These tests are based on the material capacity to resist the penetration of a tip. For materials with an elastic recovery characteristic, such as composite resins, the Knoop hardness test is mostly recommended (Anusavice, 1996; Asmussen and Peutzfeldt, 2003; Bouschlicher *et al.*, 2004; Knobloch

et al., 2004; Uhl *et al.*, 2004). With this test, the efficacy of polymerization can be indirectly inferred (Eliades *et al.*, 1995). However, to determine the degree of conversion, tests that measure the conversion of monomer into polymer are required, and spectrophotometry has been indicated (Gioka *et al.*, 2005).

According to Gioka *et al.* (2005), little attention has been paid to research related to polymerization efficiency of materials used in orthodontics. These studies are important since the degree of conversion of dental resins may be influenced by factors inherent in the material, such as the type and concentration of the photoinitiator agent or the quantity and type of organic matrix.

Therefore, the aim of this study was to assess the degree of conversion and the hardness of an orthodontic composite resin submitted to light curing by quartz–tungsten–halogen (QTH) and LED lights.

Materials and Methods

Two light sources were used: a QTH (Curing Light XL 3000, 3M Unitek, St Paul, Minnesota, USA) and an LED (Ortholux, 3M Unitek).

Sixty cylindrical stainless steel matrices were constructed with an external diameter of 10 mm, an internal diameter of

8 mm, and a thickness of 1.5 mm. The matrix was filled with Transbond XT (3M Unitek) composite resin on a glass plate, insulated with a polyester strip. Thirty samples were cured with a QTH light for 10, 20, and 30 seconds (10 samples for each time) and the other 30 samples with a LED light for 5, 10, and 15 seconds (10 samples for each time). Half of the samples were submitted to Knoop hardness testing and the other half to infrared spectrophotometry (FTIR).

The light-curing times of 10, 20, and 30 seconds for the QTH light were based on the predetermined availability of the system, while the light-curing times of 5, 10, and 15 seconds for the LED light represented half the time of that of the QTH light. The light intensity of the appliances was monitored with a conventional digital radiometer (model 8000; EFOS, Lake Bluff, Illinois, USA) for the QTH light and with an analogue radiometer (Demetron, Danbury, Connecticut, USA) for the LED. Gauging was undertaken of the measurements of each group, with their respective items of equipment and sources. The mean intensity values were 638 mW/cm^2 for the QTH and 450 mW/cm^2 for the LED.

Immediately after light activation, the Knoop hardness number (KHN) was measured with a HMV hardness tester (Shimadzu, Tokyo, Japan). Three indentations were made on the opposite side of the light incidence, at different areas on the composite resin surface, under a 200 g load for 15 seconds. The final KHN of each specimen was the arithmetic mean of three measurements.

Fourier-transform infrared (FTIR) spectroscopy was performed with the attenuated total reflectance (ATR) accessory and a plate of zinc selenite crystal at 45 degrees. All measurements were obtained under the following conditions: resolution of 4 cm^{-1} and four internal scans per reading. For each cured resin matrix, the same non-cured matrix served as the control. The non-cured matrix measurement was recorded with the mass of resin inserted in the metal matrix; this being coupled to a metal plate, which served as a distance guide for the light source extremity (1 mm), placed on the crystal plate of the ATR accessory, and isolated with a pure mineral oil (Nujol, Shering Laboratory-Plough, Kenilworth, New Jersey, USA). After the reading, the resin mass was light cured in accordance with the type of light source and at specific times.

Before the first reading and between each new set of measurements of the non-cured resin (monomer) and cured resin (polymer), a baseline spectrum was obtained with all the artefacts that would be used when the resin mass filled the matrix. The purpose of this first measurement was to determine the spectra of the artefacts used in the measurements, which would be deducted by the equipment in the subsequent monomer and polymer measurements of each specimen.

The light was applied on the side opposite the infrared reading beam scan. Between each set of monomer/polymer spectra, the crystal plate of the ATR accessory was cleaned with absorbent paper and acetone and then dried with a serigraphic blower, so that there would be no residues to prejudice the new set of monomer/polymer spectrum measurements. The spectra of the monomers and their respective polymers were compared to determine the conversion rate of the double bonds into simple carbon bonds. The peaks were measured at the frequencies of 1608 per cm (corresponding to the aromatic ring bonds) and 1636 per cm (corresponding to the bonds between carbons of the methacrylate groups; Rueggeberg *et al.*, 1990). The following formula was used to calculate the conversion rate of the double carbon bonds into simple bonds (Eliades *et al.*, 1995):

$$\%\text{Conversion} = 100 \times 1 - \frac{\text{Polymer}(C=C) \times \text{monomer}(C-C)}{\text{Monomer}(C=C) \times \text{polymer}(C-C)}$$

According to the Kolmogorov–Smirnov test, the distribution was normal for all groups. The results were statistically analysed using analysis of variance and Tukey's multiple comparisons test ($\alpha = 0.05$).

Results

The highest KHN was obtained in the QTH 30 (25.19) and 20 (24.01) second groups, which did not differ statistically, and with the LED 15 second group (21.86). The QTH 10 second group (20.53) did not differ statistically from the QTH 20, the LED 5 (19.96) and 15, or the LED 10 (18.95) second groups (Table 1).

There was no statistical difference for the degree of conversion among the QTH 10 (43.42 per cent), 20 (46.12 per cent), 30 (45.30 per cent), or LED 10 (47.02 per cent) and 15 (47.24 per cent) second groups. The lowest degree of conversion was found in the LED 5 second group (38.97 per cent), which did not differ statistically from the QTH 10 second group (Table 2).

Table 1 Knoop microhardness number (KHN) in the experimental groups using a quartz–tungsten–halogen (QTH) light or a light-emitting diode (LED) for different exposure times.

Groups	n	Mean (KHN)	Standard deviation
QTH			
10 s	5	20.53[AC]	0.81
20 s	5	24.01[AB]	1.04
30 s	5	25.19[B]	1.38
LED			
5 s	5	19.96[C]	3.64
10 s	5	18.95[C]	2.43
15 s	5	21.86[ABC]	1.64

Different letters indicate statistically different mean values ($P<0.05$).

Table 2 Polymerization degree (%) in the experimental groups using a quartz–tungsten–halogen (QTH) light or light-emitting diode (LED) with different exposure times.

Groups	n	Mean (%)	Standard deviation
QTH			
10 s	5	43.42^{AB}	2.84
20 s	5	46.12^A	3.72
30 s	5	45.30^A	4.41
LED			
5 s	5	38.97^B	2.08
10 s	5	47.02^A	1.84
15 s	5	47.24^A	2.44

Different letters indicate statistically different mean values ($P < 0.05$).

Discussion

The composite resin used in this study was Transbond XT, which is a light-cured material specifically for bonding in orthodontics and widely used in debonding strength studies; however, the literature has little information about its cure efficiency (Eliades *et al.*, 1995; Dunn and Taloumis, 2002; Cacciafesta *et al.*, 2005).

In the present study, a LED was compared with a QTH light. The latter is the most widely disseminated and has disadvantages such as heat generation, low useful life of the lamp, and degradation of the filters. However, they are accessible appliances and easy to maintain, in addition to being efficient for curing composite resins (Miyazaki *et al.*, 1998; St Georges and Miguez, 2001; Hammesfahr *et al.*, 2002; Pereira *et al.*, 2003).

The Knoop hardness test demonstrated that the LED can attain hardness similar to that of the QTH light, as the LED 15 second group did not differ statistically from the QTH groups. The results of the present study are contrary to the findings of Kurachi *et al.* (2001), Leonard *et al.* (2002), and Rahiotis *et al.* (2004), who reported that the QTH light presented higher hardness compared with the LED. The LED units used in those studies emitted low intensity (the highest being 280 mW/cm^2), which could explain this difference. Furthermore, there was a waiting time of 24 hours between light activation and hardness measurement in the studies of Leonard *et al.* (2002) and Rahiotis *et al.* (2004). This could influence the results since, with the passage of time, hardness significantly increases (Dannheimer *et al.*, 1996).

However, there is agreement when the results of research conducted with similar equipment are compared. Uhl *et al.* (2004) used the LED prototype with an intensity of 901 mW/cm^2 and found that the LED provided greater composite resin hardness 24 hours after light curing.

The present study used FTIR to verify the degree of conversion of monomer into polymer, being a method of surface analysis (Gioka *et al.*, 2005). The measurements were made before and immediately after light curing, in addition to using the double carbon aromatic bonds as the internal parameter. The reason for using this method was to make the results more accurate since the same resin mass was used as the parameter, before and after light curing.

The degree of conversion was similar for the QTH and LED lights. This finding corroborates the study of Wiggins *et al.* (2004), who also used second generation LED and QTH lights. However, Knezevic *et al.* (2001) and Bala *et al.* (2005) found higher percentages of conversion using transmittance with potassium bromide pellets. This FTIR technique allows the recording of a larger area of the cured resin since the resin is ground and the reading beam penetrates the specimen. Furthermore, in the study of Knezevic *et al.* (2001), the specimens were stored for 24 hours.

Rahiotis *et al.* (2004) used the same spectrophotometry method as in the present study and reported conversion of 55 per cent for the QTH light and 43 per cent for the LED after storage for 24 hours. The higher conversion rate of QTH light in that study in relation to the present findings can be explained by the higher power of the source used (840 against 638 mW/cm^2) and by the storage time. When comparing the performance in relation to the LED, similarity is found, in spite of the specimens having been stored for 24 hours, which would be expected to result in a higher conversion rate. This similarity is possibly explained by the low power of the appliance used in that study and the higher power of the appliance used in the present research.

In vitro investigations do not reproduce the clinical situation. The thickness of the specimens (1.5 mm) is more than the average thickness used when bonding teeth. In addition, brackets bonded to the teeth can interfere with polymerization of the composite resin. It would therefore be interesting to compare, *in vitro*, the bond strength of brackets to enamel using QTH and LED lights with different curing times to verify if the polymerization obtained is sufficient to bond brackets to enamel and withstand the applied forces. However, analysing the results obtained, it is feasible to reduce the exposure time of orthodontic resin Transbond XT with a LED by 50 per cent, compared with a QTH light.

Conclusions

1. The Knoop hardness of Transbond XT light cured with a LED for 15 seconds was similar to light curing with a QTH for 10, 20, or 30 seconds.
2. The degree of conversion of Transbond XT was similar when light cured with LED or QTH lights.
3. Light curing with a LED allowed a reduction of 50 per cent in the time recommended for the use of a QTH light with Transbond XT.

Address for correspondence

Ana Maria Spohr

Laboratory of Dental Materials, School of Dentistry (Block 6)

Pontifical Catholic University of Rio Grande do Sul (PUCRS)

Avenue Ipiranga 6681, CEP: 90616-900 Porto Alegre—RS

Brazil

E-mail: anaspohr@terra.com.br

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