

Degree of conversion of two lingual retainer adhesives cured with different light sources

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SUMMARY The aim of this study was to evaluate the degree of conversion (DC) of two lingual retainer adhesives, Transbond Lingual Retainer (TLR) and Light Cure Retainer (LCR), cured with a fast halogen light, a plasma arc light and a light-emitting diode (LED) at various curing times. A conventional halogen light served as the control.

One hundred adhesive samples (five per group) were cured for 5, 10 or 15 seconds with an Optilux 501 (fast halogen light), for 3, 6 or 9 seconds with a Power Pac (plasma arc light), or for 10, 20 or 40 seconds with an Elipar Freelight (LED). Samples cured for 40 seconds with the conventional halogen lamp were used as the controls. Absorbance peaks were recorded using Fourier transform infrared (FT-IR) spectroscopy. DC values were calculated. Data were analysed using Kruskal–Wallis and Mann–Whitney *U*-tests.

For the TLR, the highest DC values were achieved in 6 and 9 seconds with the plasma arc light. Curing with the fast halogen light for 15 seconds and with the LED for 40 seconds produced statistically similar DC values, but these were lower than those with the plasma arc light. All of these light exposures yielded a statistically significantly higher DC than 40 seconds of conventional halogen light curing. The highest DC value for the LCR was achieved in 15 seconds with the fast halogen light, then the plasma arc light curing for 6 seconds. These two combinations produced a statistically significantly higher DC when compared with the 40 seconds of conventional halogen light curing. The lowest DC for the LCR was achieved with 10 seconds of LED curing. The overall DC of the LCR was significantly higher than that of the TLR.

The results suggest that a similar or higher DC than the control values could be achieved in 6–9 seconds by plasma arc curing, in 10–15 seconds by fast halogen curing or in 20 seconds by LED curing.

Introduction

Ideally, dental restorative resin would have all of its monomer converted to polymer during polymerization. However, all dimethacrylate monomers exhibit considerable residual unsaturation in the final product, with a degree of conversion (DC) ranging from 55 to 75 per cent under conventional irradiation (Ferracane and Greener, 1986; Eliades *et al.*, 1987; Ruyter and Oysaed, 1988). The final DC of a resin depends on the chemical structure of the dimethacrylate monomer and the polymerization conditions, i.e. atmosphere, temperature, light intensity and photoinitiator concentration (Selli and Bellobono, 1993).

The DC is one factor that affects clinical performance of resin composites (Pearson and Longman, 1989; Imazato *et al.*, 1995; Miyazaki *et al.*, 2003). It is important to evaluate the DC of polymeric adhesives, because with a low DC, a release of toxic substances is possible due to the development of a weak polymer network. This parameter is also considered a key factor in modulating the profile of the material, including a wide array of mechanical properties (Ferracane and Greener, 1986) and potential biological adverse reactions (Rathbun *et al.*, 1991). There are numerous studies investigating the bond strength of various combinations

of orthodontic adhesive resins and ceramic or stainless steel brackets. However, limited information is available regarding the DC of polymeric material commonly used in orthodontics (Eliades *et al.*, 1995a). The conversion rate is particularly important for lingual retainer adhesives, as they are exposed to the oral cavity and intended to serve in the mouth for a long period of time. Eliades *et al.* (2000) evaluated the DC of orthodontic adhesives with various polymerization initiation modes. Their results revealed DCs of 48–68 per cent, the highest being for a dual-cured product.

Most dental photoinitiator systems use camphoroquinone as the diketone absorber, with the absorption maximum in the blue region of the visible light spectrum at a wavelength of 470 nm (Althoff and Hartung, 2000). Currently, the most popular method of delivering blue light is halogen-based light curing units (Mills *et al.*, 1999). Halogen bulbs produce light when electric energy heats a small tungsten filament to high temperatures (Dunn and Taloumis, 2002). Despite their common use in dentistry, halogen bulbs have several disadvantages. The basic principle of light conversion by this technique is claimed to be inefficient as the light power output is less than 1 per cent of the consumed electrical power. Halogens also have a limited effective lifetime of

approximately 100 hours due to the degradation of the bulb's components by the high heat generated (Mills *et al.*, 1999; Jandt *et al.*, 2000; Stahl *et al.*, 2000). Fast halogens with halogen bulbs of increased light intensity and turbo tips to focus the light emitted were introduced into the market after conventional halogens. Other light sources are lasers and plasma arc units. In contrast to lasers, plasma arc light sources do not emit distinct frequencies, but continuous frequency bands. However, these bands are much narrower than those of conventional lights. Consequently, radiation of undesired frequencies to be filtered is less. Due to the high intensity, the manufacturers state that 1–3 seconds of plasma irradiation cures many resin composites to a hardness comparable with that achieved after 40 seconds with conventional curing lights (Hofmann *et al.*, 2000).

Solid-state light-emitting diode (LED) technology for the polymerization of light-activated dental materials was proposed by Mills *et al.* (1999) to overcome the shortcomings of halogen visible light curing units. LEDs use junctions of doped semiconductors to generate light instead of the hot filaments used in halogen bulbs (Nakamura *et al.*, 1994). They have a lifetime of over 10 000 hours and undergo little degradation of output over this time (Haitz *et al.*, 1995). LEDs do not require filters to produce blue light, are resistant to shock and vibration, and require limited power (Mills *et al.*, 1999).

Curing lights with higher light intensities have great potential for use in orthodontics. Decreasing the total cure time for bonding may be beneficial for the clinician and the patient. Although it has been shown that higher curing light intensities may lead to superior physical and mechanical properties (Sakaguchi *et al.*, 1992), there are

no studies evaluating the effect of these light sources on the DC of various orthodontic adhesives.

The aim of the present study was to evaluate the DC of two lingual retainer adhesives cured with a fast halogen light, a plasma arc light and a LED at various curing times. A conventional halogen light served as the control. For the purposes of this investigation, the null hypothesis assumed that curing of two lingual retainer adhesives with different light sources at different exposures would not statistically significantly change the DC of the adhesives, and there would be no statistically significant difference between the overall DC values of two different lingual retainer adhesives.

Materials and methods

Two different orthodontic lingual retainer adhesives and four different lights were used in the present study (Table 1).

One hundred lingual retainer adhesive samples, 5 mm in diameter and 2 mm in height, were cured between microscope slides, using a Teflon mould, to evaluate the DC. The curing times of the various adhesive–light source combinations are presented in Table 2. Selection of the curing times for each light source was based on a previous study where the optimum curing time for each adhesive–light source combination was determined through measurement of surface microhardness (Usumez *et al.*, 2003). Samples cured for 40 seconds with a conventional halogen light were used as the controls. Five samples were prepared for every combination of curing mode and adhesive resin.

Following curing, the specimens were pulverized into fine powder with a mortar and pestle. Fifty micrograms of the ground powder was mixed with 5 mg of potassium

Table 1 The adhesives and light curing units used in the study.

Adhesives							
Brand	Company	Basic ingredient*				Lot no.	
Transbond Lingual Retainer (TLR)	3M Unitek, Monrovia, California, USA	Bisphenol A diglycidyl ether dimethacrylate 5–10 per cent				CG/1AK	
Light Cure Retainer (LCR)	Reliance, Itasca, Illinois, USA	Bisphenol diglycidylmethacrylate 5–10 per cent				106060	
Light curing units							
Model	Company	Type	Tip (mm)	Energy consumption (W)*	Power density (mW/cm ²)*	Serial no.	
XL3000	3M, St. Paul, Minnesota, USA	Halogen	13	75	580	120277	
Optilux 501	Kerr, Danbury, Connecticut, USA	Fast halogen	8	80	850	53102755	
Power PAC	ADT, Corpus Christi, Texas, USA	Plasma arc	6.5	Not provided	1200–1500	8247	
Elipar Freelight	3M ESPE, Seefeld, Germany	Light-emitting diode	8	0.75†	400	939800008375	

*As provided by the manufacturer.

†In stand-by mode.

Table 2 The exposure times employed in the study.

	XL3000 (conventional halogen)	Optilux 501 (fast halogen)	Power PAC (plasma arc)	Elipar Freelight (light-emitting diode)
Curing times (seconds)	40	5 10 15	3 6 9	10 20 40

Table 3 Degree of conversion (DC) values in the order of highest to lowest.

Transbond Lingual Retainer					Light Cure Retainer				
	Cure time (seconds)	%DC	SD	Test*		Cure time (seconds)	%DC	SD	Test*
Power PAC	9	66.9	1.9	D	Optilux	15	75.3	1.4	E
Power PAC	6	66.6	1.8	D	Power PAC	6	69.6	0.6	D
Elipar Freelight	40	58.6	2.4	C	Power PAC	9	66.8	1.0	DC
Optilux	15	57.0	0.2	C	Elipar Freelight	40	65.5	5.1	DC
Optilux	10	49.9	0.6	B	Optilux	5	65.2	1.0	DC
Optilux	5	49.0	3.0	B	Optilux	10	65.1	1.3	DC
Power PAC	3	42.3	1.2	A	Elipar Freelight	20	64.6	0.6	DC
Elipar Freelight	20	40.5	1.9	A	XL3000	40	62.7	4.0	CB
XL3000	40	40.3	5.1	A	Elipar Freelight	10	57.9	1.8	B
Elipar Freelight	10	36.2	7.6	A	Power PAC	3	52.6	2.9	A

SD, standard deviation.

Groups with different letters are statistically different from each other.

Power PAC, plasma arc light; Elipar Freelight, light-emitting diode; Optilux, fast halogen light; XL3000, conventional halogen light.

bromide powder (Carlo-Erba Reagenti, Rodano, Michigan, USA), and the absorbance peaks were recorded using the diffuse-reflection mode of Fourier transform infrared (FT-IR) spectroscopy (Perkin-Elmer, 1600 Series, Norwalk, Connecticut, USA). Five samples of uncured adhesive resin were prepared by smearing it onto thin potassium bromide discs to determine the control spectra for each resin.

The amount of double vinyl bonds remaining in the sample exposed to irradiation is shown by the intensity of the peak at 1637 cm^{-1} , referring to the C=C stretching of the vinyl group which has been used in the study of polymerization of acrylates and methacrylates (Decker, 1992). The DC was directly related to the decrease in 1637 cm^{-1} absorption on the FT-IR spectra. The DC was calculated according to the following equation:

$$\%DC = ((A_0 - A_t)/A_0) \times 100$$

where A_0 is the absorption of the peak at 1637 cm^{-1} when time is equal to zero and A_t is the absorption at time t (Sideridou *et al.*, 2002).

Statistical analysis

The values recorded for each adhesive were subjected to statistical analysis separately using a non-parametric Kruskal-Wallis test followed by a Mann-Whitney

U -test for pairwise comparisons. An independent samples t -test was used to statistically evaluate the differences between the combined overall DC values of the two adhesives (SPSS, version 10.0.1, Chicago, Illinois, USA).

Results

Representative FT-IR spectra of the composites before and after setting are shown in Figure 1. Tables 3 and 4

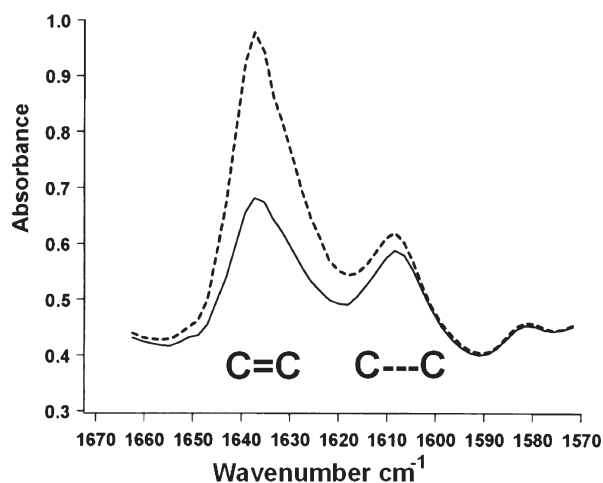


Figure 1 Representative Fourier transform infrared (FT-IR) spectra of an adhesive before (---) and after (—) irradiation.

Table 4 Overall degree of conversion (DC) values of two different lingual retainer adhesives used in the study.

	%DC	SD	Independent samples <i>t</i> -test
Transbond Lingual Retainer	55.5	10.3	***
Light Cure Retainer	67.6	5.7	

****P* < 0.001.
SD, standard deviation.

show the DC values obtained and the results of their multiple comparisons for the tested lingual retainer adhesives.

Transbond Lingual Retainer (TLR)

The results demonstrated that the highest DC values for the TLR were achieved with the plasma arc curing light for 6 and 9 seconds, followed by 40 seconds with the LED and 15 seconds with the fast halogen light. DC values produced by 5 or 10 seconds of curing with the fast halogen light were statistically similar. All of these light exposures yielded statistically significantly higher DC values when compared with 40 seconds of conventional halogen light curing, which was used as the control. On the other hand, curing for 3 seconds with the plasma arc light, and 10 or 20 seconds with the LED produced statistically similar DC values compared with the control samples (Table 3 and Figure 2a).

Light Cure Retainer (LCR)

The highest DC value for the LCR was achieved by 15 seconds of curing with the fast halogen light, followed by 6 seconds of plasma arc curing. Both of these results were statistically significantly higher compared with the control samples. This was followed by 9 seconds of plasma arc curing. DC values statistically comparable with the control samples were achievable in 5 seconds with the fast halogen light, in 6 seconds with the plasma arc light and in 10 seconds with the LED. The lowest DC value, which was significantly lower than the control, was produced by 3 seconds of curing with the plasma arc light (Figure 2b).

TLR versus LCR

Overall DC value data for each adhesive were acquired by pooling the DC value data produced by the highest exposure of each light source, i.e. 40 seconds with the conventional halogen light, 15 seconds with the fast halogen light, 9 seconds with the plasma arc light and 40 seconds with the LED. The overall DC data were subjected to statistical evaluation using an independent

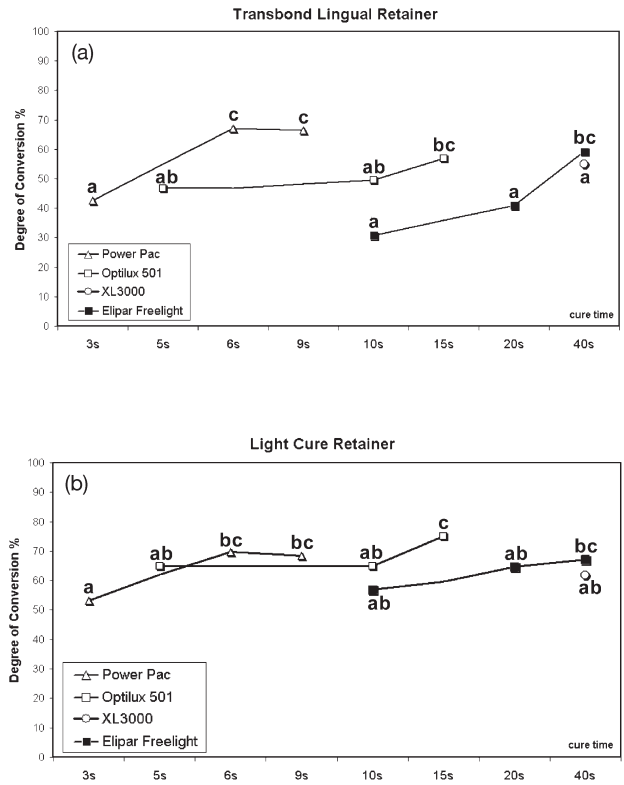


Figure 2 Degree of conversion of (a) Transbond Lingual Retainer and (b) Light Cure Retainer with various light sources and exposure times.

samples *t*-test. The results revealed that the overall DC values of the LCR (67.6 ± 5.7 per cent) were significantly higher (*P* < 0.001) than those of the TLR (55.5 ± 10.3 per cent).

Discussion

The results of this study revealed that there were statistically significant differences between the DC values of lingual retainer adhesives cured with different light sources and exposure time combinations. The overall DC values of different adhesives were also statistically different. Thus, the null hypothesis was rejected.

Among several methods to determine the DC of composites, FT-IR has been proven to be a powerful technique and has been widely used as a reliable method (Eliades *et al.*, 1987; Imazato *et al.*, 1995), as it detects the C=C stretching vibrations directly before and after curing of materials. However, the method chosen to determine the DC with FT-IR has some limitations. The DC values reported in this study are averages from the whole specimen and ignore the depth of cure, i.e. possible DC differences between the top and the bottom of the adhesive cured. Another limitation of the present investigation might be considered to be the relatively small number of samples per test group

($n = 5$), although studies with smaller sample sizes are found in the literature (Lane *et al.*, 1998; Peutzfeldt *et al.*, 2000; Silikas *et al.*, 2000; Caughman *et al.*, 2001; Imazato *et al.*, 2001; Sideridou *et al.*, 2002).

The DC of resins is a major factor that influences their physical properties. In general, the higher the conversion of double bonds the greater the mechanical strength. The unreacted double bonds may either be present in free monomer or as pendant groups on the network. The unreacted monomer may leach from the polymerized material and irritate the soft tissues. For example, tetraethyleneglycol dimethacrylate (TEGDMA), a common type of monomer used in adhesive resins, is reported to affect bacterial growth around a restoration (Hansel *et al.*, 1998). Furthermore, monomer trapped in the restoration may reduce the clinical serviceability of composite through oxidation and hydrolytic degradation, which may be manifest in forms such as discoloration of the fillings and accelerated wear (Tanaka *et al.*, 1991). These unfavourable changes may lead to detachment of the bonded lingual retainer from the tooth surface, exposure of the retainer wire due to excessive wear of the adhesive, and caries formation around the adhesive. Additionally, discoloured adhesive may be visible from the labial surface on smiling. Therefore, these disadvantages are important in the clinical application of lingual retainer adhesives.

The DC is also very important due to adverse biological reactions with a low DC (Rathbun *et al.*, 1991). This has recently gained interest in the light of new evidence that some resins may release bis-phenol A, a bis-GMA monomer precursor, which exhibits oestrogenicity (Olea *et al.*, 1996). The amount of residual monomer leaching from chemical and visible light-cured orthodontic adhesives and the relationship of this release with DC were evaluated by Eliades *et al.* (1995b). Those authors reported a statistically significant linear correlation between the DC of directly irradiated specimens and the bis-GMA concentrations eluted by these groups of specimens. No or very little bis-GMA leach was present when the DC reached 55–60 per cent. The overall DC values of 56–68 per cent determined in the present study using the overall DC data (Table 4) may be sufficiently high to prevent monomer leach from the lingual retainer adhesives tested. However, this is an indirect assumption and should be evaluated with caution.

The results of the present study suggest that statistically higher DC values for the TLR can be obtained with shorter exposures with newer light sources than with 40 seconds of conventional halogen curing. Higher DC values for the LCR were also obtained with the new light sources. However, only with 15 seconds of fast halogen curing or 6 seconds of plasma arc curing was the DC statistically higher than the control.

The mean power densities of the light curing units used in this investigation are presented in Table 1.

The plasma arc and fast halogen curing units had higher mean power density values compared with the conventional halogen light. Therefore, the higher monomer conversion with shorter exposures with these lights in the present study may be attributed to the higher light energy delivered to the resin material. This result is in agreement with Silikas *et al.* (2000). On the other hand, the LED unit used in this investigation had the lowest power density, but was able to achieve similar or higher monomer conversion in shorter cure times compared with the control light. Fujibayashi *et al.* (1998) demonstrated that the quality of light polymerization is not exclusively due to the light intensity; the narrow absorption peak of the initiator system must also be taken into account. This makes the emitted spectrum an important determinant of a curing light's performance. The absorption curve of camphorquinone extends between 360 and 520 nm, with its maximum at 465 nm. It has been shown that within this range, the optimal emission band width of the light source lies between 450 and 490 nm (Nomoto, 1997). With conventional curing devices, a major portion of the photons are emitted outside the optimal spectrum range for light curing. These photons cannot, or only with reduced probability, be absorbed by camphorquinone. In contrast, 95 per cent of the emission spectrum of blue LEDs is situated between 440 and 500 nm. The emission maximum of the blue LEDs used in this study was approximately 465 nm, which is almost identical to the absorption peak of camphorquinone. These factors may explain the similar or higher DC values obtained with the LED with shorter exposure times. At clinical levels of irradiance, Mills *et al.* (1999) reported a greater depth of cure when the composites were polymerized with a LED lamp in comparison with a halogen lamp, despite the former having a measured output of approximately 70 per cent of the latter (276 versus 388 mW/cm² when measured between 410 and 500 nm). Knezevic *et al.* (2001) demonstrated only marginally higher DC values with even 66 times stronger light output halogen curing units compared with an LED of minimal intensity of 12 mW/cm². These previous findings also support the importance of considering the emission spectra of curing lamps relative to the absorption spectrum of camphorquinone when assessing the quality of light polymerization.

When the overall DC values of the two different lingual retainer adhesives used in the present study were evaluated, the highest DC values were 66.9 per cent (36.2–66.9 per cent) and 75.3 per cent (52.6–75.3 per cent) for the TLR and the LCR, respectively. These values are close to those reported by Eliades *et al.* (2000) for different light-, chemical- or dual-cured products. Statistical evaluation of the pooled overall DC data of the two different adhesives demonstrated that the overall DC values for the LCR (67.6 per cent) were significantly higher than for the TLR (55.5 per cent). This implies

that the former converts its monomers to polymers more effectively. This significant difference between the LCR and the TLR might be a consequence of differences in the chemical structure of the dimethacrylate monomer and photoinitiator concentration (Selli and Bellobono, 1993). However, it is also interesting to note that the control results for the LCR were already higher than for the TLR (62.7 versus 40.3 per cent). When the differences between the control and overall DC data produced by the test lights are considered, it can be seen that the TLR produces a further 15 per cent conversion compared with that of the LCR at 5 per cent. Therefore, it would have been difficult for the test light sources to have improved their DC rates. This brings into question whether the DC values of the TLR could be increased significantly with longer exposures to light sources other than those employed in this study. However, this seems unlikely. It has been shown previously that monomers, including bis-GMA and TEDGMA, reach 85–90 per cent of their final DC in 40 seconds with a XL 3000 halogen light source, which was also used in this study (Sideridou *et al.*, 2002).

According to the results of the current investigation, the plasma arc light, the fast halogen light and the LED produced similar or better DC values with shorter exposure times compared with conventional halogens. In particular, the xenon plasma arc lights demonstrated higher DC values with markedly reduced curing times (6–9 seconds). However, from a practical point of view, the plasma arc curing units are bulky and usually incorporate a noisy cooling fan, the light is transmitted through a moderately flexible optic cord, which may cause a hazard during clinical use, they produce too much heat and are more expensive. Fast halogens are generally more expensive than conventional curing units, but are similar in ease of clinical use. LEDs have certain advantages over both halogen and plasma arc curing lights. They are cordless, smaller, lighter, do not require a cooling fan, and have an estimated life of over 10 000 hours (Haitz *et al.*, 1995). Moreover, LED technology is still developing, and high-intensity LED curing lights are available, which might be even more efficient. According to Dunn and Taloumis (2002), halogen-based light curing units may be replaced by LEDs as semiconductor technology improves. Consequently, all light sources have their own advantages and disadvantages and the individual clinician should make a selection according to their clinical needs and expectations.

Conclusions

This study investigated the effects of various light exposures from a fast halogen light, a plasma arc light and an LED on the DC of two commercially available lingual retainer adhesives and used 40 seconds of

halogen-based light exposure as the control. Within the limits of this study, the results suggest that a similar or higher DC than the control values could be achieved in 6–9 seconds with plasma arc curing, in 10–15 seconds with fast halogen curing or in 20 seconds with a LED.

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