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Research Article

Volume 2 Issue 1 *Journal of Dental Treatment and Oral Care*

Evaluation of Depth of Cure and Degree of Conversion of a Photo-Active Composite Resin Irradiated Through Tooth Substance

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Citation: Ferdinand Mabula Machibya and Samson Alexander Tarimo (2018) Evaluation of Depth of Cure and Degree of Conversion of a Photo-Active Composite Resin Irradiated Through Tooth Substance. J Dent Treat Oral Care 2(1): 102

Received: January 26, 2018; **Published:** February 28, 2018

Abstract

Aims: The study investigated the influence of trans-dental photo-curing on the depth of cure (DoC) and degree of conversion (DC) of resin composite irradiated by different curing modes.

Methods: A curing lamp having six different curing modes was used for indirect photo-activation of a nano-composite through six different thicknesses of tooth tissues ranging from 0.5 to 3 mm. The DoC was evaluated by means of an ISO 4049 standard technique and Fourier transformation infrared (FTIR) spectroscopy was used to determine the DC.

Results: The composite resin DoC and DC mean values decreased with increasing intervening tooth thickness for each of the six light curing modes. Lower DoC and DC mean values were recorded when indirect irradiation was used compared to control direct curing (P<0.01). The DoC and DC mean values for turbo and high light modes were significantly higher than normal, soft-start, pulse and pulse-soft light modes (P<0.01).

Conclusions: Curing composite resin through tooth structures lowers DoC and DC. Turbo and High light curing modes through tooth structures achieve significantly higher DoC and DC mean values than other radiation modes.

Keywords: *Indirect Curing; Depth of Cure; Degree of Conversion; Irradiation Modes; Photosensitive Composite Resin; Ftir*

Abbreviations: *DOC: Depth of cure; DC: Degree of conversion; FTIR: Fourier transformation infrared; QTH: Quartz-Tungsten Halogen Bulbs; PAC: Plasma-Arc Lamps; LED: Light Emitting Diodes; CQ: Camphor Quinone*

Introduction

Dental caries is prevalent in communities of all socio-economic status affecting most children and adults worldwide [1,2]. The current treatment of caries largely rests on filling technology, with alloys, ceramics and composite resin being the main restorative materials. Due to their improved mechanical strength and aesthetic appearance, light-cured composite resins have gained worldwide popularity in dental clinics [3].

Factors affecting composite restoration success

According to Sabbagh the success of a composite restoration depends on three main factors which are quality of composite and bonding system as well as use of a light-curing system capable of delivering enough energy to properly complete the polymerization process [4]. Consequently, any technical error committed during one of those three steps, can result in clinical failure.

The required energy (joules) for complete cure is a function of power (mW/cm2) and duration of curing light that may vary by the type and shade of composite material. The delivered amount of energy is also dependent on distance of the light tip from the resin material, movement of light tip during curing, condition of the light curing unit, indirect or direct curing as well as thickness of the resin being cured [5-7]. Even the most powerful curing light will not cure a composite if it is not properly placed. Just because the top layer of the composite is hard, that does not mean that the composite is cured at the bottom [5]. Depth of cure (DoC) along with DC governs and influence the quantity and quality of polymerization and are also affected by material particle size, light intensity, curing time and variety of other factors [8-10].

Cavity preparation procedures can encounter complex clinical cavity shapes and more tooth structure may be retained. The remaining tooth structures may sometimes prevent direct irradiation, risking incomplete cure with indirect irradiation [6,7]. It is advocated that each filling layer thickness should be approximately 2 mm, while selecting the soft-start mode to reduce polymerization shrinkage [11]. There are at least four main types of curing lights, quartz-tungsten halogen bulbs (QTH), plasma-arc lamps (PAC), argon-ion lasers and light emitting diodes (LED).

LEDs have been hailed in delivering greater efficacy on polymerization of light cured composites. LEDs give out light with a wavelength between 410 and 490 nm, which is the light spectrum most effective for light curing. The 468 nm wavelength is the absorption peak of Camphor Quinone (CQ), a photo-initiator present in most modern composite resins [12]. Different types of irradiation modes have been introduced to the dental profession to improve resin polymerization by decreasing internal stresses in order to achieve better marginal adaptation in bonded composite resin restorations [13]. Furthermore, studies have shown that, LED light output power is related to different curing modes [12]. There are at least six different irradiation modes, which are standard (normal), soft-start, pulse, pulse-soft, high-light and turbo modes. Polymerization shrinkage during composite resin filling process is a major drawback; and soft-start mode has been suggested to reduce contraction stress [11]. Whereas, turbo and high intensity modes are based on a concept that total energy delivered at once decrease exposure time and are claimed to have a greater depth of cure compared to conventional lights [14].

Assessment of Light-cured Composite Resin Polymerization Degree

Several methods can be employed to measure polymerization degree of a cured composite resin. These techniques include the use of ISO 4049 standard technique which is a simple method measuring DoC. This method measures the height of the remaining hardened material after scrapping away the unset soft material. DoC is then calculated as 50% of the remaining hardened specimen height [15]. Other methods for assessing the extent of composite cure utilize optical microscope, stepped surface hardness and Fourier transform infrared spectroscopy (FTIR) which measures the degree of conversion (DC) of composite resin double bonds [15-18].

To date, there are only few researches devoted to study the through-tooth structure indirect light curing of composite materials to clarify the impact of intervening tooth structure on the final properties of the composite restorations irradiation [6,7]. The current study aimed at evaluating the influence of tooth thickness and irradiation modes on degree of cure of a Nano-composite using ISO 4049 standard technique for DoC and FTIR analysis for DC.

Materials and Methods

Ethical clearance to conduct the study was obtained from the ethical committee of the Jilin University, China. Recently extracted caries-free human molar teeth were collected. The teeth were temporarily stored for a period of seven days in 8% formalin solution for disinfection and fixation. Dental calculus and attached periodontal soft tissues on the surfaces of the teeth were removed with a hand scaling instrument, cleaned with running water in a rubber cup and slurry of pumice and then kept in 1% thymol solution at room temperature for storage.

The teeth were sliced into small squared pieces about 6 x 6 mm, at different thicknesses using a 0.1mm thick diamond disc (Jiangyin disc, Jiangsu, China).

The thicknesses of the slices were approximately 0.5 mm, 1 mm, 1.5 mm, 2 mm, 2.5 mm and 3 mm. The thickness for each slice was measured using a digital caliper (Shan 132A Series Digital Caliper, Guilin China) in three different places to get an average thickness. The dental slices were allocated into six groups according to their thicknesses and were stored in distilled water at room temperature while waiting for further steps. In preparation for composite light curing, the prepared dental slices were removed from the storage, treated for 15 seconds with 37% phosphoric acid etching agent (SCi-PHARM Gel Etch, USA), the etchant was washed by water spray and dried by air blow. A thin layer of single bond adhesive system (DenFilTM Flow adhesive, USA) was applied on the side of the tooth slice facing the composite mold. In order to obtain a thin layer of adhesive on the tooth, air blow was used to disperse any excess. The bonding agent was then indirectly cured for 20 seconds by LED light unit (Dr's Light, Good Doctors Co, Ltd. South Korea) by transmitting the light through the tooth.

The curing light operated in six different modes; normal, high, pulse, soft-start, pulse-soft and turbo modes. The tip of the light source was placed close enough to touch the tooth specimen being irradiated. Lateral direct light was prevented from curing the composite by using a shutter fabricated from light-proof x-ray film paper folds, so that only light filtered through the dental slices was allowed to cure the composite.

At the beginning of the experiment a curing light meter (Cure Rite, Model No: 646726 (Dentsply Caulk, Milford USA) was used to measure the power output of the curing light. A detailed experimental protocol is provided in a previous publication [19].

ISO 4049 Standard -Evaluation of depth of cure (DoC)

Composite resin indirectly cured through tooth slices was used for experimental groups, while direct curing was applied in control group. For the indirect curing groups; composite resin was carefully packed into the plastic cylindrical molds having 4 mm internal diameter and 7 mm height. A 10 mm tall mold was selected for the direct curing (control) group. Adequate condensing was done to avoid air voids during composite placement. The tooth slices were treated as explained above and the light was used to cure the composite via tooth slices. The standard ISO 4049 was used to determine the DoC of composite specimens in each group immediately after photo-activation. The DoC mean values for experimental groups were compared against each other and against DoC mean values of the control group [15].

FTIR Spectroscopy Evaluation of Degree of conversion (DC)

The composite resin prepared for FTIR spectroscopy analysis was meticulously packed into cylindrical molds (4 mm inner diameter and 2 mm height), and then photo-activated using the same curing protocols as for the ISO 4049 standard technique. Contrary to the ISO 4049 standard technique which used 0.5 mm to 3 mm tooth thickness, FTIR only used 0.5 mm, 1 mm and 1.5 mm thicknesses. The decision based on the observation that DC could only be recorded to the maximum of 1.5 mm tooth thickness with a 2 mm composite layer.

Immediately after photo-curing, at room temperature and under light protection, each cured specimen was reduced into fine powder by using a 0.1 mm thick with 20 mm diameter diamond disc (Jiangyin disc, Jiangsu, China) mounted on a slow speed hand-piece motor. The fine powder was collected and subjected to the FTIR spectrophotometer (Nicolet 5700, Thermo Electron Corporation, Madison, USA) for analysis. For FTIR spectrophotometer assessment, 10 mg of the composite powder was mixed with 100 mg of potassium bromide (KBr) powder salt. The mixture was placed into a pelleting device and pressed in a hydraulic press with a load of 8 tons to obtain a pellet.

This pellet was then placed in a holder attachment within the spectrophotometer for analysis. The uncured composite was analyzed using a metal siliceous window. The measurements were recorded in absorbance mode with the FTIR Spectrophotometer coupled to a computer. Monomer conversion was calculated using changes in the ratios of aliphatic (C=C) to aromatic (C–C) absorption peaks in the uncured (monomer) and cured (polymer) states obtained from the infrared spectra. All experiments were carried out in triplicates. The FTIR analysis of C=C peaks was done using Origin Lab Pro 8.6.0 software.

Statistical analysis

Statistical Package for the Social Sciences (SPSS) 18.0, (Chicago, IL, USA), was used to analyze original data collected. A paired sample t-test was run to compare the mean depths of cure and degrees of conversion for the experimental (indirect curing) and control (direct curing) groups. For the experimental groups one-way ANOVA tests were also done to compare DoC and DC between the groups according to irradiation mode and tooth thickness. Multiple comparisons of DoC and DC by Tukey's test (α=5%) followed the analysis.

Results

Our dependent variables DoC and DC were normally distributed as assessed by the Shapiro-Wilk tests. Statistically, the experimental (indirect curing group) showed significant difference when compared to control (direct curing) group ($P<0.05$).

ISO 4049 Standard evaluation of Depth of cure (DoC)

The DoC mean values for all groups according to tooth slice thickness were statistically significantly different among each other ($p<0.05$), with 0.5 mm tooth slice group having the greatest DoC mean values while 2.5 and 3 mm groups had the least DoC mean values. DoC showed a decreasing tendency with increase in tooth thickness. At 3 mm tooth thickness there was no any appreciable DoC except for the turbo light curing mode. Turbo and high curing modes could cure the standard 2 mm composite layer even when light filtered through 1 mm dentine thickness. However, the rest curing modes adequately cured the 2 mm composite resin layer only at 0.5 mm tooth thickness as assessed by the ISO 4049 standard. Some of the curing mode groups were statistically significant different from each other as outlined in the table 1. Soft-start and pulse light curing modes showed no significant different, except for thicknesses under 1.5 mm.

Generally turbo light mode recorded the highest DoC mean values than any of the other curing modes: While, pulse-soft and normal modes had the lowest DoC mean values. Turbo light showed higher DoC means compared to normal, pulse, pulse-soft and the soft-start light curing modes (P<0.05), Figure 1. High light mode also showed significantly higher DoC than pulse-soft mode (P<0.05).

Light Mode	0.5 mm $\bar{x}$(S)	1.0 mm $\bar{x}$(S)	1.5 mm $\bar{x}$(S)	2.0 mm $\bar{x}$(S)	2.5 mm $\bar{x}$(S)	Control $\bar{x}$(S)
Normal	2.09(.020)^{a}	1.46(.015)^{a}	1.00(.025)^{k}	0.61(.013)^{x}	0.37(.008)^{u}	5.99(.101)^{a}
Soft-start	2.35(.017)^{b}	1.73(.018)^{b}	1.30(.016)^{l}	0.67(.022)^{r}	0.33(.017)^{s}	6.29(.050)^{b}
Pulse	2.28(.140)^{b}	1.60(.026)^{b}	1.10(.015)^{m}	0.68(.011)^{r}	0.34(.015)^{s}	6.17(.064)^{b}
Pulse-soft	2.19(.019)^{c}	1.50(.020)^{a}	1.07(.065)^{n}	0.62(.012)^{y}	0.30(.011)^{s}	6.13(.050)^{b}
High	2.62(.027)^{d}	2.02(.017)^{c}	1.45(.018)^{o}	0.96(.008)^{z}	0.51(.01)^{v}	6.61(.070)^{d}
Turbo	2.93(.041)^{e}	2.25(.061)^{d}	1.62(.560)^{p}	1.17(.03)^{a}	0.68(.03)^{r}	7.17(.162)^{c}

Means with different superscript letters, on a column are statistically significant different at P<0.05
Table 1: DoC mean and standard deviation values for ISO 4049 standard technique

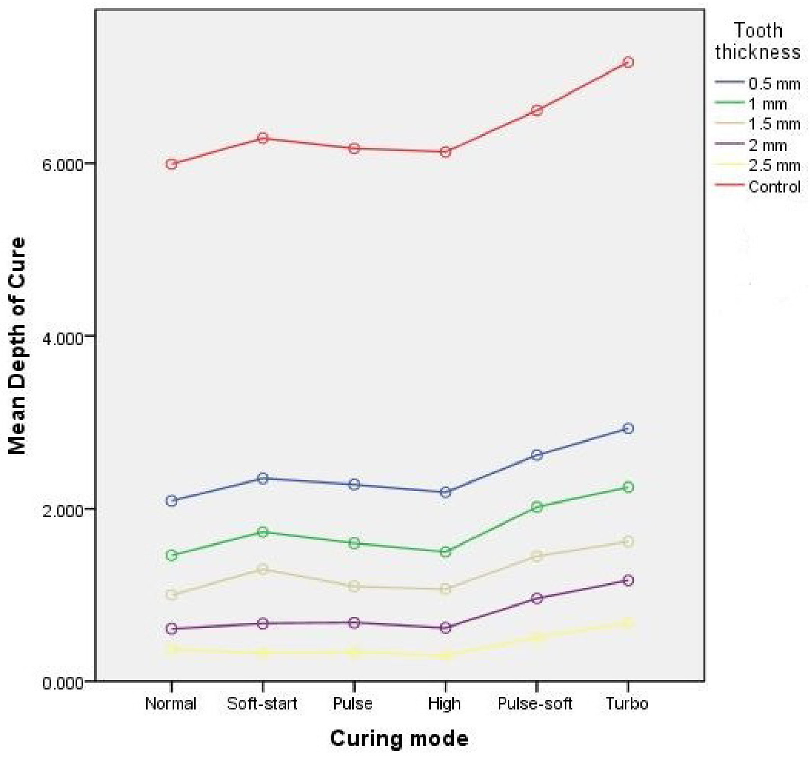

Figure 1: A linear plot showing the DoC mean values according to light curing modes through different tooth thickness

The Degree of Conversion (DC) by FTIR analysis

Regardless of the type of light-curing mode, DC mean values for all tooth slice groups were statistically significant different from each other (P<0.05). It was also noted that DC mean values decreased with increasing tooth thickness, with 0.5 mm groups having the greatest DC values while thicker dentin groups showed the least values. The DC could only be assessed to the maximum of 1.5 mm tooth thickness at which a 2 mm layer of composite was assessed. Generally turbo light mode recorded the highest mean DC values than any other light mode while normal light mode presented the lowest values. Figure 2 Illustrates how trans-dental light curing DC mean values vary among different tooth thicknesses and light modes.

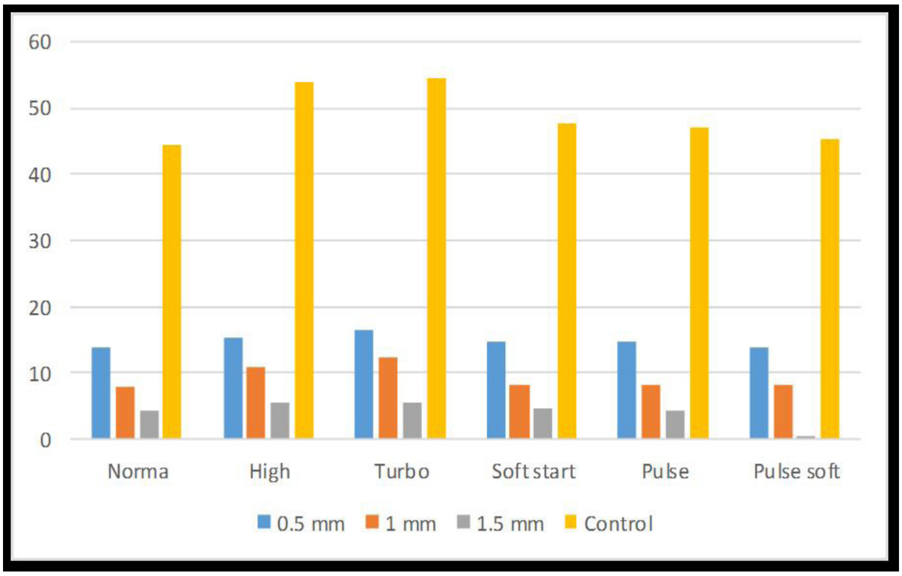

Figure 2: A bar chart showing how DC mean values vary with different tooth thicknesses and light modes

Discussion

For optimal physical, mechanical and biochemical properties of composite restoration, proper DoC and DC must be achieved. However, complete ideal polymerization of light-cured composite resins may not be a practical reality in some clinical situations. Some composite materials properties and clinical situation related factors, such as procedural techniques and curing equipment performance hamper the practical efforts to achieve the best level of composite polymerization [18-20]. In some complex cavities, parts of enamel and dentine are preserved during caries excavation to maximize the retention for composite restoration. In such conditions, curing composite through tooth structure may be the only option [17]. However, indirect exposure through tooth structure may reduce conversion degree and depth of cure [18]. In order to evaluate the effects of indirect through-tooth irradiation on composite DoC and DC, the experiment used freshly extracted human teeth slices of different thickness.

Influence of trans-dental curing on composite resin DoC

The study recorded lower DoC mean values for all indirect irradiation compared to the control (direct) irradiation groups regardless of the intervening tooth thickness (Table 1, Figure 1). Dietschi., et al. [20] reported marked photo-polymerization differences between indirect and direct exposure. From Table 1 it can be learned that curing through 0.5 mm tooth structure could achieve 2 mm or more DoC. Conversely, when the tooth structure thickness was about 3 mm, the DoC was extremely low such that it became insignificant to assess. However, some authors researching on the final properties of dental resin composites cured directly have reported that ISO 4049 standard tendend to overestimate DoC [21,22]. The phenomenon was observed in the current experiment when a more reliable FTIR spectroscopy analysis showed substantial under polymerization for the indirect curing methods: Thus, none of the through-tooth curing met the optimum polymerization percentages which are in the range of 43- 75% [22]. Curing degree of visible light-cured resins depends on the intensity and quantity of light reaching the resin. The recorded lower DoC in experimental groups may be related to the attenuation of light, as the light beam transmits through the tooth structure to reach the composite resin. Arikawa., et al. [7] who evaluated the light-attenuating effects of human enamel on the final properties of composite resins using artificial filters simulating dental tissues concluded that the light attenuating effects of dental tissues reduced polymerization efficiency, resulting in poorer mechanical properties of light cured composite resins. This finding is consistent with the trend observed in our experiment, that the intensity of light depends on the thickness of the tooth tissue it traverses and may undergo different degree of scattering or absorption, resulting into diminished polymerization degree hence, reducing the DoC and DC.

Influence of trans-dental curing on the composite resin DC

The experiment employed different thickness of tooth structure for indirect irradiation of light-cured composite resin. Seen in Figure 2 are mean DC values for the experimental and control groups. The average DC values for all experimental groups were lower than the control group. For the experimental groups, 0.5 mm group recorded the highest DC mean values, with every increase in tooth thickness, the DC decreased. The Degree of conversion (DC) of methyl-acrylate bonds determined by FTIR spectroscopy, is the percentage of carbon double bonds converted to single carbon bonds to form cross-linked polymer chains [20]. Conversion degree is an important parameter in the performance of composite restoration. As expected, the thicker the tooth slices the less light intensity reaching the resin composite photo initiator, as a result, 0.5 mm groups had the highest DC mean values while 1.5 mm recorded the lowest values. There were statistically significant differences in DC mean values between 0.5 mm, 1 mm and 1.5 mm groups ($P<0.05$). The 2 mm 2.5 mm and 3 mm groups were not used for DC assessment with FTIR, since negligible DC was recordable in these groups.

Dental composite resin degree of double bond conversion ranges between 43% -75% [22]. According to our findings with FTIR spectroscopy analysis, there is substantial under polymerization for the indirect curing methods; thus none of the through-tooth curing reached the optimum polymerization percentages. Experimental groups with lower DC may have resulted from light attenuation as the light beams transmit through the tooth structure to reach the composite resin. The reduced DC may be due to scattering or absorption of light during transmittance through tooth tissue as previously speculated [7]. Dietschi,. et al. [20], recorded the least critical situation when 0.5 mm of composite Z100 was cured through 1 mm enamel. Although the experiments involved through-tooth tissue curing, only the quantitative analyses of hardness-based measurements on the degree of polymerization were employed, no qualitative analyses were done.

Influence of different irradiation modes on DoC and DC

The DoC and DC mean values significantly varied with different curing modes. Turbo and high light curing modes showed the highest average DoC, while normal and pulse-soft light curing modes recorded the minimum values. This difference in mean DoC and DC values may be due to different light curing intensities generated by different curing modes. The results of this study show that the DoC and DC are affected by the thickness of intervening tooth as well as light curing modes. Light energy density is positively correlated with DoC and DC, while dental tissue thickness showed negative correlation with DoC and DC.

In the settings of the study, with different curing modes it was found that DoC was more than 2 mm when irradiating through 0.5 mm of tooth structure. Turbo and high light curing modes produced more than 2 mm DoC even when the tooth thickness was 1 mm: In contrary, all other light modes when transmit through more than 0.5 mm tooth thickness, the resultant DoC was less than 2 mm. Moreover, at every tooth thickness tested in the study, it was found that the highest mean values were observed with the turbo and high intensity light modes. The finding is in agreement with Rueggeberg., et al. [23], who obtained higher DoCs by using lights of higher intensity. However, the authors cautioned that the high intensity may result in greater polymerization shrinkage and greater marginal leakage. Turbo light showed statistically significant difference at ($p<0.05$) among the groups especially when compared with normal, pulse, pulse-soft and the Soft-start modes. Moreover, High-light also showed significant difference with pulse-soft mode at ($p<0.05$). Generally there was no significant difference between Pulse soft, Soft start, Normal and Pulse modes.

According to Sakaguchi and Berge polymerization process seems to be more dependent on the total energy available for photo-activation, so any method that provides a higher amount of energy to the resin composite material would have a higher DoC and DC values [24]. Energy density is the product of the power intensity (mW/cm2) and irradiation time (s). Turbo light mode has its intensity rapidly rising to 1600 mW/cm2, being the highest energy density of all the light modes employed in our study: It was expected to show the highest mean values as noted in our findings. On the contrary, the normal and pulse-soft modes which have the lowest energy densities 16J/cm2 and 18J/cm2 respectively showed the lowest DoC and DC mean values. Dental tissues are reported to reduce dramatically the intensity of the light transmitted across it, rendering inadequately composite polymerization. Based on similar observation, Belvedere suggested 2-3 fold energy density increase when curing composite through tooth structure [25]. The current experiment support the suggestion of increasing energy density when indirect light curing is employed to compensate for the observed lower polymerizations when curing light gets attenuated by tooth structure. Based on this study's findings the authors suggest that composite resin curing process should avoid indirect irradiation. Should the need arise to indirectly irradiate composite resin, the composite filling thickness ought to be less than 2 mm, and the operator should consider ways to increase the energy density in order to improve the degree of polymerization.

The tooth tissue sections used in the experiment were cut by hand held instruments, thus rendering a certain degree of difficulty to obtain uniform thickness; each group used approximated value which may have introduced some errors. Temperature effect was not taken into account in our study due difficulty in controlling temperatures led us into irradiating the tooth pieces and composite at significantly lower temperatures. Curing reaction being positively affected by higher temperatures would probably have recorded higher values in temperatures similar to those in the human oral cavity.

Conclusions

Nano-composite resins cured through different tooth thickness have lower DoC and DC compared to direct curing. The DoC and DC decrease with increasing intervening tooth thickness. The DoC and DC mean values for turbo and high light modes were statistically higher compared to normal, soft start, pulse and pulse soft light modes. ISO 4049 standard technique can record adequate DoC of 2 mm composite layer at maximum 0.5 mm dentin thickness for all light modes. Whereas, turbo and high light intensity modes can cure the 2 mm composite layer even at 1 mm tooth thickness.

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