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Research Article

Influence of Tooth Thickness on Depth of Cure and Degree of Conversion of a Photo-Activated Resin Composite Irradiated Through the Tooth -

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ABSTRACT

Objective: To investigate the influence of trans-dental photo-curing on the Depth of Cure (DoC) and Degree of Conversion (DC) of a resin composite irradiated by a light curing unit operating in different curing modes.

Methods: Six main groups were formed in accordance with different tooth thicknesses which were further divided into six subgroups (n=5) according to light modes. DoC was evaluated by means of an ISO4049 standard scraping technique. Fourier Transformation Infrared (FTIR) spectroscopy was used to determine DC.

Results: DoC and DC decreases with increasing intervening tooth thickness. At 3mm tooth thickness, except for Turbo mode, there is no obvious curing performance. Turbo and High light modes could achieve the resin composite 2mm DoC even when tooth structure was 1.0mm thick. However for the rest of the light modes, the 2mm composite layer could adequately be cured when the tooth thickness was 0.5mm only. FTIR analysis mean DC values for the through-tooth-structure indirect curing were lower than the 45%-75% direct curing values. Among the groups, 0.5mm group had the highest DoC and DC mean values, 2.5 mm and 3mm groups had the lowest mean values. The composite resin showed lower DoC and DC mean values when irradiated indirectly compared to control direct curing ($p < 0.005$). 0.5 mm and Turbo light groups recorded the highest DoC and DC values than other groups, differences between tooth thickness groups were statistically significant with $p < 0.005$.

Conclusions: (1) A nano-composite resin cured through different tooth thickness will have lower DoC and DC compared to direct curing. (2) In clinical operation, composite should be directly cured, indirect curing must be avoided at all possibilities.

Keywords: Indirect Curing; Depth of Cure; Degree of Conversion; Photosensitive Composite Resin; FTIR

INTRODUCTION

Dental caries is a bacterial infectious disease of the hard dental tissues, and is one of the most common human diseases, especially affecting many children. Bacteriological research and experimental animal model studies have confirmed that the Streptococcus mutans to be the most important cariogenic bacteria. Currently treatment for caries aims at filling technology, the main restorative materials are metal alloys, ceramics and composite resin materials.

Following research development of the dental materials, light-cured composite resins, due to their good mechanical strength and aesthetic performance, are widely used in the clinics. In order to get good results for composite resin restoration of tooth structure defects, one should consider two aspects-enamel and dentin. Due to the development of modern tooth-colored materials, concept of restoration has undergone changes, during repair of posterior teeth with resins, occlusal surface of the cavity margin does not require the cavity margin bevel preparation, the reason for the cavity walls of the posterior teeth to have straight occlusal cavity margins, is to avoid beveling of the enamel prisms which would widen the width of the cavity margins, which in turn can accelerate restoration wear, moreover the filling located on the cavity edge bevel is relatively weak and easily fracture. Preparation of the proximal part includes bucco-lingual wall shape, the bucco-lingual width, retaining grooves' position and gingival wall, in different retention situations all have necessary requirements, complying with the tooth structure conservation. According modern restoration concepts, during cavity preparation more of the tooth structure may be retained. Clinical cavity filling procedures meet complex cavity shapes. In order to obtain composite restorations with minimal level of shrinkage and the greatest degree of polymerization, and at the same time ensuring marginal seal and bonding strength, currently, placement of composite resin in layers and successive curing technique is a widely encountered application during filling of the cavities. The filling thickness of the material should be based on the properties of the materials used and the curing device performance. It's advocated that each filling layer thickness should be approximately 2 mm, while selecting the soft-start mode to further reduce polymerization shrinkage. Soft start mode increases the time to reach the gel point, such that there is sufficient time to take advantage of the resin's own flow to compensate for the polymerization volume shrinkage generated by the reaction, however when the light intensity decreases, inevitably may lead to insufficient polymerization. While ensuring preservation of as much as possible tooth structure, direct irradiation curing of composite resin may not be feasible, thus affecting the degree of polymerization of the composite resins. The depth of cure of the composite resin is one of the important factors influencing the filling placement. Incomplete curing can lead to composite resin restoration early degradation, wear, and affect the functional durability, eventually leading to restoration failure; the depth of cure is also affected by material particle size, light intensity, curing time and variety of other factors. Light-cured composite resins have remarkably been on constant improvement since they were introduced into restorative dentistry and in recent years they have been used in abundance as an alternative to dental amalgam for the restoration of cavities in posterior teeth stress bearing areas which have to withstand high masticatory forces, as well as reliable aesthetic materials for anterior teeth restorations [1,2]. Achieving optimum degree of polymerization is still a challenge to dentists all over the world. Several factors play crucial roles in meeting the goal for safe, biocompatible and functional durable composites. Some authors have pointed out that achieving adequate depth of cure and maximum degree of monomers conversion is critical to the success of resin composites [3].

This experiment focused on the indirect exposure through the tooth structure of composite resin, at the same time taking into account the effects of different light-curing modes on the polymerization of composite resin. Using dental tissue slices prepared from extracted human teeth, to establish in-vitro indirect composite resin curing models, through ISO4049 standard scraping test method uncured composite resin was scraped away, then assessing the height of the remaining cured material in order to assess the depth of cure of the composite resin. FTIR spectroscopy was used for assessment of the cured resin degree of conversion, to further explore the different light modes, different thickness of the tooth structure irradiation on the depth of cure and the degree of conversion of composite resin. The tooth structure conservation principle during clinical procedures for dental composite resin restorations and choices for different exposure modes provided a theoretical basis and technical support [4-7].

RESULTS

ISO4049 Standard Scraping technique--DoC

DoC mean values for all groups according to tooth slice thickness were statistically significant different among each other ($p < 0.005$), with 0.5 mm thick tooth slice groups having the greatest DoC mean values while 2.5 and 3 mm thick tooth slice groups having the least DoC mean values (Figure 1). DoC showed a decreasing tendency with increasing tooth thickness. Except for the Turbo light curing mode, at about 3 mm tooth thickness there was no any appreciable DoC. For all curing light modes, 0.5 mm was noted as maximum tooth thickness where 2 mm composite resin incremental layer can adequately be cured as assessed by the ISO4049 standard scraping technique. *T*urbo and High light curing modes could however cure the standard 2 mm composite layer even when light filtered through 1 mm dentine thickness. Some of the groups under the light mode grouping were statistically significant different from each other as outlined in the (Table 1). Soft Start and Pulse light curing modes showed no significant different for all tooth thicknesses, except under 1.5 mm thickness.

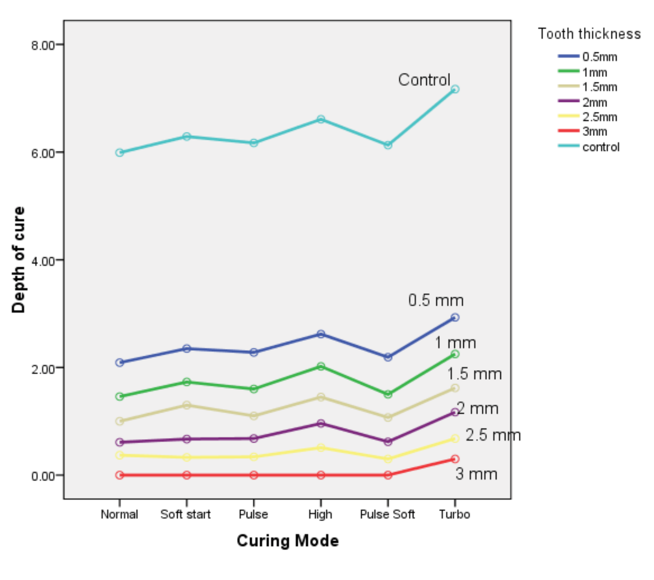

Figure 1: Graph showing how trans-dental light curing DoC mean values vary among different tooth thicknesses and light modes.

Generally Turbo light mode recorded the highest DoC mean values than any of the other light curing modes while Pulse Soft and Normal modes had the lowest DoC mean values. Turbo light showed statistically significant difference at $p < 0.05$ as compared to the other groups particularly with Normal, Pulse, Pulse Soft and the Soft Start light curing modes. High light mode also showed significant difference at $p < 0.05$ with the Pulse Soft mode.

FTIR spectroscopy technique--DC

Regardless of the type of light-curing mode, DC mean values for all tooth slice groups were statistically significant different from each other ($p < 0.005$). 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.

DC could only be assessed to the maximum 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 presented lowest values. Statistical significant differences for the groups according to light curing modes are summarized in (Table 2) and (Figure 2). Illustrates how trans-dental light curing DC mean values vary among different tooth thicknesses and light modes.

DISCUSSION

Factors affecting degree of polymerization of light-cured resin composites are so many. These factors include composite materials' own internal factors; clinical situation related factors, such as procedural techniques, curing equipment performance related factors and so on. Scholars have conducted extensive researches in this aspect of degree of polymerization.

In the process of measuring resin mold DoC with extra-oral scraping methods, DoC is easily affected by the type of material, resin shade, exposure time, light source performance and many other

Table 1. DoC (`x ± S) mean values for ISO 4049 standard scraping technique.

Light Mode	Tooth thickness mm					
	0.50mm	1.00mm	1.5mm	2.0mm	2.5mm	3.0mm	Control
Normal	2.09 ± 0.020^{a}	1.46 ± 0.015^{a}	1.00 ± 0.025^{k}	0.61 ± 0.013^{x}	0.37 ± 0.008^{u}	Null	5.99± 0.101^{a}
Soft start	2.35 ± 0.017^{b}	1.73 ± 0.018^{b}	1.30 ± 0.016^{l}	0.67 ± 0.022^{r}	0.33 ± 0.017^{s}	Null	6.29± 0.050^{b}
Pulse	2.28 ± 0.140^{b}	1.60 ± 0.026^{b}	1.10 ± 0.015^{m}	0.68 ± 0.011^{r}	0.34 ± 0.015^{s}	Null	6.17± 0.064^{b}
Pulse soft	2.19 ± 0.019^{c}	1.50 ± 0.020^{a}	1.07 ± 0.065^{n}	0.62 ± 0.012^{y}	0.30 ± 0.011^{s}	Null	6.13 ± 0.050^{b}
High	2.62 ± 0.027^{d}	2.02 ± 0.017^{c}	1.45 ± 0.018^{o}	0.96 ± 0.008^{z}	0.51 ± 0.01^{v}	Null	6.61 ± 0.070^{d}
Turbo	2.93 ± 0.041^{e}	2.25 ± 0.061^{d}	1.62 ± 0.560^{p}	1.17 ± 0.03^{a}	0.68 ± 0.03 ^{r}	0.32±0.03	7.17 ± 0.162^{c}

Means with different superscript letters, on a column are statistically significant different at $p < 0.05$.

Table 2. DC (% ± S) mean values for the FTIR Spectroscopy technique.

Light mode	Tooth thickness			
	0.5mm	1mm	1.5mm	Control
Normal	13.86 ± 0.751^{a}	7.98 ± 0.304^{c}	4.23 ± 0.14^{g}	44.33 ± 0 .979^{i}
High	15.39 ± 0.343^{b}	10.75 ± 0.223^{d}	5.45 ± 0.514^{h}	53.98 ± 0.111^{j}
Turbo	16.44 ± 0.586^{b}	12.34 ± 0.272^{e}	5.45 ± 0.309^{h}	54.70 ± 0.360^{j}
Soft start	14.72 ± 0.208^{a}	8.29 ± 0.353^{c}	4.55 ± 0.186^{g}	47.61 ± 0.535^{i}
Pulse	14.74 ± 0.255^{a}	8.12 ± 0.626^{c}	4.44 ± 0.200^{g}	47.15 ± 0.555^{i}
Pulse soft	13.73 ± 0.270^{a}	8.32 ± 0.32^{f}	4.37 ± 0.201^{g}	45.47 ± 0.618^{i}

Means with different superscript letters, on a column are statistically significant different at $p < 0.05$.

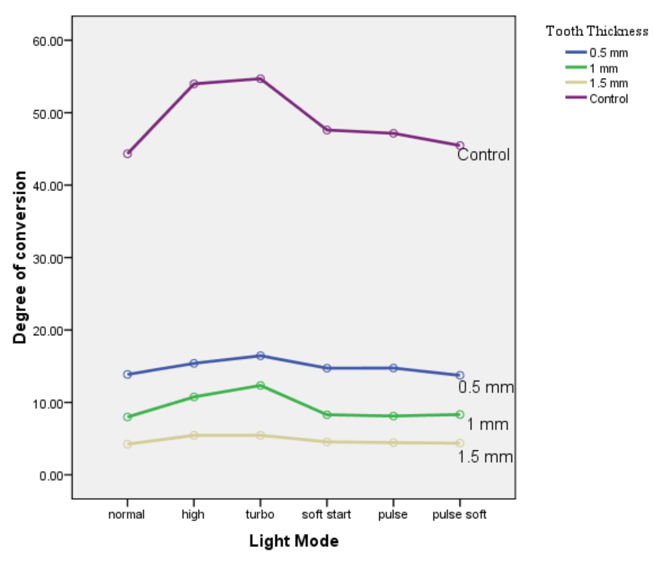

Figure 2: Graph showing how trans-dental light curing DC mean values vary among different tooth thicknesses and light modes.

factors. Due to the use of different types of equipments, materials and test conditions, the comparability of the studies' results is poor. And the results are also likely to be affected by the properties of the mold material, having a larger difference between the values of the type of mold material as measured by the same depth of cure of the resin. FTIR spectroscopy is a common method used to determine degree of conversion. The technique being fast, sensitivity and cost effective can be used for solids, liquids and gases. Infrared is an electromagnetic radiation in the form of heat which can cause both the vibration and rotation of molecules. The molecular functional groups absorb electromagnetic radiation in the infrared region resulting into specific absorption bands along a spectral region [8-22]. DC of methyl-acrylate bonds is the percentage to which carbon double bonds is converted to single carbon bonds to form cross-linked polymer chains [22-25]. DoC and DC mean values differ for all groups according to 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 values may be due to different light irradiation curing intensities changes generated by different curing modes.

Turbo light mode recorded the highest DC values for all the groups while Pulse soft and Normal light modes showed the lowest values. Turbo light showed statistically significant difference at ($p < 0.05$) among the groups especially when compared to the Normal, Pulse, Pulse soft and the Soft start modes. High light also showed significant difference at ($p < 0.05$) with the Pulse soft mode. Generally there was no significant difference between Pulse soft, Soft start, Normal and Pulse modes, while Turbo mode showed significant difference among the groups. According to Sakaguchi RL and Berge HX; 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 depth of cure and degree of conversion values [26]. Energy density (J/cm^2 or mWs/cm^2) is the product of the power intensity (mW/cm^2) and irradiation time (s). Turbo light mode has its intensity rapidly rising to $1600 mW/cm^2$. In 20 seconds the energy density is calculated to be approximately $32 J/cm^2$, 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 result findings. Similarly for the Normal and Pulse Soft modes which have the lowest energy densities $16 J/cm^2$ and $18 J/cm^2$ respectively showed the lowest depth of cure and degree of conversion values. In a study done by Belvedere PC, composite was placed in bulky and cured through the tooth from the buccal and lingual aspects, composite hardness was measured, beginning from the occlusal surface and proceeding toward the pulp. The hardness of the indirect cured bulk-filled restorations was significantly less than the incrementally direct cured restorations.

This experiment evaluated only one kind of resin material, with the same particles size, same resin material shade was evaluated, certainly these were limitations. Also the tooth tissue sections used in the experiment were cut by a 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.

From the experiment it is suggested to consider increasing the energy density of the proposed method in order to improve the degree of polymerization, more studies should be conducted to expand a broad composite resin curing research scope. I believe in the near future, through further research, will provide to clinicians more help and guidance on dental composite resin filling process. According to the current results, intervening tooth thickness can affect Filtek™ Z350 nano-filled resin composite depth of cure and degree of conversion. Therefore, the null hypothesis of this study is rejected.

METHODS AND MATERIALS

FTIR spectroscopy technique--DC

Recently extracted caries-free human molar teeth were collected. Research ethical issues were adhered in the collection of the teeth. The teeth were temporarily stored for a period of seven days in a solution of 8% formalin 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.

A diamond disc 0.1 mm thickness, diameter 20 mm (Jiangyin disc, Jiangsu, China) was used under water cooling to slice out the teeth into small squared pieces about 6 x 6 mm length, at different thicknesses. The thickness of the slices was 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 thickness and while waiting for further steps were stored in distilled water at room temperature. The dental slices were maintained in water all the time to avoid drying out which could have affected their optical properties (Table 3).

The prepared dental slices were removed from the water, treated for 15 seconds with 37% phosphoric acid etching agent (SCi-PHARM Gel Etch, USA), the etchant was washed away by water spray and air dried. A thin layer of single bond adhesive system (DenFil™ Flow adhesive, USA) was applied on the side of the tooth slice facing the composite mold by using a non-linting brush and in accordance with the manufacturer's instructions. In order to obtain a thin layer of adhesive on the tooth as per requirement of a standard composite filling procedure, air spray was used to blow away any excess. The bonding agent was then indirectly cured for 20 seconds by the LED light unit (Dr's Light, Good Doctors Co, Ltd. South Korea) by transmitting the light through the tooth (Figure 3).

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. Table 4, shows the light curing modes, exposure time and the light intensities for each curing technique.

Table 3. Material name, composition and manufacturer.

Product name	Composition		Manufacturer
Filtek™ Z350 nanofilled Universal Restorative A2 shade Lot: N327325	Resin matrix: Bis-GMA Bis-EMA UDMA with small amounts of TEGDMA	Filler loading: Non-agglomerated 20 nm nanosilica fillers Loosely bound agglomerated nanocluster formed of agglomerates of primary zirconia/silica particles with average size of 5-20 nm (cluster size: 0.6 to 1.4 microns)	3M ESPE Dental Products, St. Paul, MN, USA

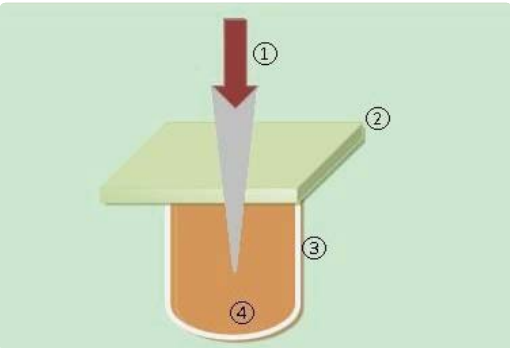

Figure 3: Indirect trans-dental curing; composite mold, dental slice setup 1. Curing light source, 2. Dental slice, 3. cylindrical mold, 4. Resin composite sample.

Table 4. Irradiation modes and light intensities.

Mode of Irradiation	Light intensity
Normal Mode	800 mW/cm²
Turbo mode	Rise rapidly to 1600 mW/cm2
Soft start mode	0 to 5s 600 mW/cm², and for 15s continued at 1200 mW/cm²
High mode	1200mW/cm²
Pulse mode	1080mW/cm², interval 0.05s, total time 20s
Pulse soft mode	0 to 5s increased from 150 to 1085 mW/cm², with the pulse mode, intensity steadily rising, with pulse interval 0.05s. After 15s 1080 mW/cm², maintained up to the 20th second
Exposure time for each light mode was 20 seconds.s	

At the beginning of the experiment a curing light meter (Cure Rite, Model No: 646726 (Dentsply Caulk, Milford USA) was used for the measurements of power output of the curing light.

ISO4049 Standard Scraping Technique--DoC

Composite resin was either indirectly cured by light transmitted through the tooth slices or cured directly; the later was used as a control. For the indirect curing groups; composite resin was carefully packed into the plastic cylindrical molds having 4 mm internal diameter and 7 mm height. 10 mm height was selected for the control direct curing groups. Enough care was taken to avoid air voids during composite placement, by adequately condensing the composite. The tooth slices were treated as explained above, light was allowed to pass through the tooth substance and cure the composite. The standard scraping technique ISO4049 was used to determine the DoC of composite specimens in each group immediately after photo-activation. After the irradiation, the molds were opened; the DoC of composite resin measured by removing the soft uncured material with a spatula. Height of the remaining material was measured in three places to get an average value in the nearest 0.01 mm using a digital caliper. (Shan 132A Series Digital Caliper, Guilin China.) DoC was tabulated as 50 percent of the remaining height after removing the soft uncured composite. The mean values for DoC for the experimental groups were compared against each other and against DoC mean values for the control groups. Means were also compared against a standard 2 mm depth which is the proposed composite placement thickness for incremental layering.

FTIR Spectroscopy technique--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 ISO4049 standard scraping technique. The LED light curing unit (Dr's Light, Good Doctors Co, Ltd. South Korea) operated in the six curing modes in time 20 s per each mode. Photo-activation was performed according to the following curing light modes; Normal, Turbo, Soft Start, High, Pulse and Pulse Soft. The light curing tip was positioned close enough to touch the tooth slice surface and light was transmitted through the tooth slice to cure the composite. 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.

Immediately after photo curing, at room temperature and under light protection, each cured specimen was reduced into fine powder by using a diamond disc 0.1 mm thickness, diameter 20 mm (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, Verona Madison USA) for analysis.

10 mg of the composite powder was mixed with 100 mg of potassium bromide (KBr) powder salt. This mixture was placed into a pelleting device and then 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 operating under the following conditions: 300-4000 cm^{-1}

wavelength; 4 cm^{-1} resolution; 32 scans. Monomer conversion was calculated using changes in the ratios of aliphatic to aromatic C=C absorption peaks in the uncured (monomer) and cured (polymer) states obtained from the infrared spectra. The following formula was used to deduce the percentage of remaining monomers (%C=C). Where AbsAl and AbsAr are the height or the area of absorption bands for aliphatic C=C (peak at 1638 cm^{-1}) and aromatic C-C (peak at 1608 cm^{-1}) respectively. DC was obtained by subtracting the residual percentage of aliphatic C=C from 100% (DC%= 100-(%C=C).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 from studying composite resin Filtek™ Z350 concerning the effect of tooth thickness and curing light modes on depth of cure (DoC) by ISO4049 scraping standard technique and DC by FTIR spectroscopy analysis. 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 DCs between the groups according to irradiation mode and tooth thickness (Table 4). Multiple comparisons of DoC and DC by Tukey's test (at alpha = 5%) followed the analysis.

CONCLUSIONS

The objective of the study was to investigate the influence of tooth substance on the depth of cure and degree of conversion of nano-composite resin when incident curing light energy is attenuated by absorption and scattering as it travels across the tooth thickness. The results of this study showed that when indirectly photo-curing composites by passing LED light though the tooth structure, the thickness size of intervening tooth as well as photo-activation methods influence depth of cure and degree of conversion of the dental nano-composite. Clearly, more studies should be done to evaluate other parameters of different brands of resin composite as well as various types and modes of curing lights.

ISO4049 scraping technique results showed that, the incremental 2 mm thick layer buildup is adequately cured at maximum 0.5 mm dentin thickness for all light modes. Turbo and high light intensity modes could cure the 2 mm composite layer even at 1 mm tooth thickness. However FTIR spectroscopy analysis showed that whenever composite was indirectly irradiated, regardless of both intervening tooth thickness and light mode used; degree of conversion was lower by a least one third of the direct curing value. ISO 4049 technique is not a reliable method in evaluating degree of polymerization; it tends to inflate depth of cure results.

Indirect curing should not be an option except only in situations where direct curing is impossible. The study findings emphasize that significant problem may arise when composite is indirectly cured resulting into lower cure depths and reduced degrees of conversion. Dentists should try to avoid indirect curing at all possibilities. In situations where some of the incident light will have to pass through tooth thickness before reaching composite, curing a composite layer in multiples at different angles, longer curing times or post curing at higher intensity can compensate for this problem.

In the process of attempting to save as much as possible tooth structure under the tooth structure conservation cavity preparation principle should the need necessitate indirect curing of composite resin, filling thickness should be less than 2 mm, and at the same time taking into account an increase in energy density methods so as to improve the degree of polymerization.

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