EGYPTIAN DENTAL JOURNAL
www.eda-egypt.org
VOL. 70, 4047:4055, OCTOBER, 2024
PRINT ISSN 0070-9484 • ONLINE ISSN 2090-2360
CONSERVATIVE DENTISTRY AND ENDODONTICS
Submit Date : 2024-09-19 • Accept Date : 2024-10-01 • Available online: 05-10-2024 • DOI : 10.21608/EDJ.2024.320419.3206

EFFECT OF DIFFERENT CURING TIME ON DEGREE OF CONVERSION AND DEPTH OF CURING OF SUPRA-NANO FILLED RESIN COMPOSITE COMPARED TO A CONVENTIONAL ONE

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ABSTRACT

Objective: to evaluate the effect of curing time on degree of conversion and depth of curing of supra-nano filled resin composite compared to a conventional one.

Methods: Thirty-six cylindrical specimens measuring two millimeter diameter and six millimeter thickness were made for the degree of conversion (DC) assessment. For cuing depth evaluation, thirty cylindrical specimens (2 mm diameter x 6 mm height). Based on the resin composite that was employed, the specimens were split into two primary groups: the "ES" group (supra nano-filled composite). and the "FZ" group (composite filled with nano hybrid). Each group was subsequently split into three subgroups (T1, T2, and T3) based on the different curing times of 1, 3, and 10 seconds. A Fourier transformed the DC Infrared spectrometer Transform and the curing depth were determined using an electronic caliper. Data were tabulated and statistically analyzed.

Results: There was significantly increase in the DC with the increase of curing time regardless the type of resin composite. T3 showed the highest DC, followed by T2 while T1 showed the lowest DC. Depth of curing at T3 and T2 showed significantly higher values than T1 for both groups. At T1; "ES" group showed a significant higher curing depth than "FZ" group.

Conclusion: The increase of curing time is positively affecting both degree of conversion and depth of curing. Moreover, the short curing time was beneficial to obtain adequate depth of curing in supra-nano filled resin composite.

KEY WORDS: Degree of Conversion, Depth of Curing, Curing time, Supra Nano Filled composite, Nano hybrid composite.

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INTRODUCTION

Resin composites (RCs) have become the most popular used restorative material due to their optimal mechanical, aesthetic and physical properties that meet patients' demands [1]. In addition to their ability to bond to tooth tissues with corresponding adhesive systems, they provide conservative cavity preparation and preserve the remaining tooth structure. Thus, RCs have great attention in clinical practice and scientific research [2]. Over the years, progress in restorative dentistry has allowed for the development of composite resin compositions to improve their mechanical and optical properties [3].

RCs are composed of three main components: resin matrix, fillers, and an initiator [4]. During the process of polymerization, resin composite monomers are converted into polymers; the amount of monomer conversion is known as the degree of conversion [5]. Resin composite restorations that have a high degree of conversion (DC) produce restorations with optimal physical properties and proper clinical performance. Therefore, to obtain the optimal DC of resin composites, it is necessary to have enough irradiance energy during the polymerization process [6]. Many factors affecting the DC, as light characteristics, resin composition, curing time, distance from the curing device, light intensity, shade, and translucency [7].

A specific depth of polymerisation can be achieved in resin composites through light activation, contingent upon the duration of visible light penetration into the composite [1]. As the light from the curing unit travels through the composite layers, its intensity gradually decreases [1]. This may cause decrease in DC of resin composite monomer as the distance from the irradiated surface increases. The development of new light curing units with high-intensity is necessary to obtain sufficient curing depth and proper physical properties of RCs along the whole depth of composite increment [7]. Today, it is important to save the treatment time for both clinicians and patients. So, it is effective to increase curing depth by using third-generation LED curing units, which provide polymerization with high light intensity in a short time [8].

One of the remarkable factors that affects the DC and depth of curing of RCs is filler percent and content. The final composite properties are enhanced by the filler type, which has proved to be successful in many clinical situations [9]. A convention adopted from the early days of RCs evolution and along the development of resin composite fillers is that RCs are often named according to filler type; such as hybrid, micro-hybrid, nanofill, microfill and nanohybrid RCs[2]. The application of nanotechnology in the manufacturing of composite fillers allows incorporation of more filler content due to their small size, which results in better mechanical properties [8]. Therefore, this study was conducted to evaluate the effect of curing time on degree of conversion and depth of curing of supra-nano filled resin composite compared to a conventional one.

MATERIALS AND METHODS

Two resin composite restorations of shade A2 were used in the current study; supra Nano-filled composite (Estelite ®Alpha, Tokuyama) and Nano-hybrid filled (Filtek Z250 Universal Restorative, 3M) as be seen in table (1).

Sample size calculation

Regarding the degree of conversion; In order to apply a statistical test of the null hypothesis which states that there should be no difference in the degree of conversion across the various tested groups — a power analysis was created with sufficient power. The minimal total required sample size (n) was discovered to be 36 samples by adopting an alpha (α) level of (0.05), a beta (β) level of (0.2) (i.e., power=80%), and an effect size (f) of (0.691) estimated based on the results of a prior study [10].

TABLE (1) The applied materials in the current study

Resin Composite Description	Composition		Manufacturer and lot No.
	Fillers	Matrix	
Estelite ®Alpha Supra Nano spherical filled	0.2μm submicron Silica-zirconia spherical filler represent 82 % by weight plus composite filler	Bis-GMA (Bis-phenol A di-glycidyl-methacrylate), and TEGDMA (Tri-ethylene glycol di-methacrylate)	Tokuyama, Tokyo, Japan. E726M6 https://www.tokuyama.dental.com
Filtek™ Z250 XT Nano-hybrid	Surface-modified zirconia / silica with a median particle size 3 μm, and 20-nanometer surface-modified silica particles represent 82% by weight.	Bis-GMA (Bis-phenol A diglycidyl-methacrylate), TEGDMA (Tri-ethylene glycol di-methacrylate), PEGDMA (poly ethylene glycol dimethacrylate), UDMA (Urethane di-methacrylate), and BIS-EMA (Ethoxylated bisphenol A poly-ethylene glycol dietherdmeth-acrylate)	3M ESPE, Saint Paul, MN, USA. 1470 https://www.3mespe.com

For the depth of curing; In order to apply a statistical test of the null hypothesis that there would be no difference between the various tested groups with respect to the depth of curing — a power analysis was created with sufficient power. The minimal total required sample size (n) was found to be (18) samples by adopting an alpha (α) level of (0.05), a beta (β) level of (0.2) (i.e., power=80%), and an effect size (f) of 1.50) computed based on the findings of a prior study [10]. For Windows *, R statistical analysis software version 4.4.1 was used to calculate the sample size.

Specimens' preparation and grouping

The study was approved by Scientific Research Ethical Committee, Faculty of Medicine, Fayoum University and coded as (R 591 - 120 – 14 / 7 /2024). Regarding the degree of conversion assessment, Thirty-six cylindrical shaped specimens measuring two millimeter diameter and six millimeter thickness were made using a specially designed Teflon split mold. According to type of resin composite used specimens were split into two major groups (n=18); "ES" group (Estelite Alpha, supra nano filled composite) and "FZ" group (Filtek Z250, nano-hybrid filled composite). Another subdivision was done for each group into three subgroups (n= 6) according to the curing time: (T1; 1 second, T2; 3 second and T3; 10 second).

In order to evaluate the curing depth, thirty cylindrical shaped specimens of two millimeter diameter and six millimeter height were prepared. Based on the resin composite that was employed, the specimens were sectioned into two primary groups (n = 15); "ES" and "FZ". Each group was further split into three subgroups (n= 5) according to the curing time: (T1; 1 second, T2; 3 second and T3; 10 second).

Specimen's prepared by using a thin glass slide covered by a mylar strip (Stripmat, Polydentia, Mezzovico, Switzerland). The resin composite material in each corresponding group were packed into the mold as three increments 2mm each according to the manufacturer. Each increment was light cured form the top surface using LED light curing unit with a light intensity 1000mW/cm^2 ~ 2500mW/cm^2 (Woodpecker I LED - light curing unit, Woodpecker Medical Instrument, Co., Ltd., Information Industrial Park, Guilin National High-Tech Zone, China).

* R Core Team (2024). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/.

The final increment of composite resin was covered with a second mylar strip, and surplus material was removed by gently compressing another glass slide. The light guide tip was positioned so that it made contact with the glass slide on the specimen's upper surface in order to carry out photo-activation. Every specimen was exposed to radiation for the designated amount of time one, three, or ten seconds depending on the group of specimens.

Degree of conversion assessment

Prior to testing, the specimens were kept dry and at room temperature for a full day in separate, dark containers following photoactivation. Utilising Fourier transform infrared spectroscopy [FTIR], the degree of conversion was assessed. Using a mortar and pestle, every polymerised specimen (n=6) in each matching group was ground into a fine powder. A thin disc was created by pressing 50 micrograms of the powder with 5 milligrams of potassium bromide powder. This disc was then put in a specimen container and brought to the spectrophotometer. Using the diffuse-reflection mode of an FTIR equipped with a diamond ATR crystal system, the absorbance peaks were recorded with a 4 cm-1 resolution in the 4000-400 cm-1 spectrum band. Using an FTIR spectrometer, an attenuated total reflection spectrum was obtained (Bruker Optik GmbH, Germany; Vertex 80). Each corresponding group's unpolymerized specimens (n=6) were spread onto thin potassium bromide discs and put in a spectrophotometer cell holder. A spectrum was then acquired using the identical parameters as previously stated for the polymerised specimens [11].

By measuring the variations in peak height ratio between the uncured material and the absorbance intensities of an internal standard peak of aromatic C=C at 1608 cm-1 and the aliphatic C=C peak at 1638 cm-1 after polymerisation, the degree of conversion was ascertained. The following formula was used to get the DC % for each specimen:

$$DC\% = \left\{1 - \frac{(1638\ cm^{-1}/1608\ cm^{-1})\ cured}{(1638\ cm^{-1}/1608\ cm^{-1})\ uncured}\right\} x100$$

Depth of cure measurement

The curing depth was tested following ISO 6874/2015 specification[12]. Immediately after curing of each specimen, it was removed from the split mold. The uncured composite materials were scrapped off from the bottom surface till set material was reached using sharp lanced. The remaining cured materials height was estimated at the central portion of the resulting cylinder. A digital caliper (Digital caliper, Bacolis, Egypt) were used to calculate the curing depth values. This value is 50% of the obtained height. The test was repeated 5 times for each of the tested materials at each tested curing times. The data was recorded, tabulated and statistically analyzed.

Statistical analysis

Numerical data were presented as mean and standard deviation (SD) values. They were tested for normality and variance homogeneity by viewing distribution and using Shapiro-Wilk's and Levene's tests, respectively. The depth of cure data was non-parametric and was analyzed using Aligned Rank Transform (ART) analysis. The assumptions were not violated for the degree of conversion data, and it was analyzed using a two-way ANOVA test. The comparisons of simple effects were made using the error term of the main model with p-value adjustment using the False Discovery Rate (FDR) method. All tests were conducted with a significance level of $p<0.05$. The statistical analysis was carried out using R statistical analysis software version 4.4.1 for Windows.*

RESULTS

The ANOVA test results presented in Table (2) showed that only curing time significantly affected the DC ($p<0.001$). Table (3) and Figure (1) represent the comparisons of simple effects.

* R Core Team (2024). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/.

There was significantly increased in the DC with the increase of curing time (p<0.001) regardless the type of resin composite. There was a significant difference between the three curing time evaluated; where the T3 showed the highest DC, followed by T2 while T1 showed the lowest DC. There was no statistically significant difference between the two "ES" and "FZ" groups. FTIR plots showed the degree of conversion for "ES" and "FZ" groups at different tested curing time presented in Figure (2).

TABLE (2) Two-way ANOVA for degree of conversion:

Source	*Sum of squares (II)*	*df*	*Mean square*	*f-value*	*p-value*
Material	0.11	1	0.11	**0.02**	**0.893**
Curing time	1694.95	2	847.47	**142.93**	**<0.001***
Material * curing time	1.45	2	0.73	**0.12**	**0.885**

df degree of freedom, * significant (p<0.05).

TABLE (3) Simple effects comparisons for degree of conversion:

Curing time	*Degree of conversion (%) (Mean±SD)*		*p-value*
	FZ	*ES*	
T1	68.97±2.09^{C}	68.62±3.16^{C}	**0.824**
T2	79.93±2.49^{B}	80.64±1.60^{B}	**0.649**
T3	87.00±3.01^{A}	87.00±1.85^{A}	**0.999**
p-value	**<0.001***	**<0.001***	

Values with different superscripts within the same vertical column are significantly different, * significant (p<0.05).

The ART results for the depth of cure presented in Table (4) and showed a significant interaction between both tested variables (p=0.044). The comparisons of simple effects presented in Table (5) and Figure (3). There was a significant difference between "ES" and "FZ" groups at T1 where; "ES" group had a significantly higher depth of cure than "FZ" (p=0.009). However, no significant difference exhibited at T2 and T3 for "ES" and "FZ" groups. T3 and T2 demonstrated a significant higher curing depth values than T1 (p<0.001).

TABLE (4) ART for depth of cure:

Source	*Sum of squares (II)*	*df*	*Mean square*	*f-value*	*p-value*
Material	11.52	11.52	1	**0.08**	**0.778**
Curing time	4380.14	2190.07	2	**48.75**	**<0.001***
Material * curing time	909.14	454.57	2	**3.40**	**0.044***

df degree of freedom, * significant (p<0.05).

TABLE (5) Simple effects comparisons for depth of cure:

Curing time	*Depth of cure (mm) (Mean±SD)*		*p-value*
	FZ	*ES*	
T1	0.27±0.05^{B}	0.42±0.07^{B}	**0.009***
T2	1.84±0.39^{A}	1.68±0.28^{A}	**0.522**
T3	1.90±0.41^{A}	2.30±0.47^{A}	**0.522**
p-value	**<0.001***	**<0.001***	

Values with different superscripts within the same vertical column are significantly different, * significant (p<0.05).

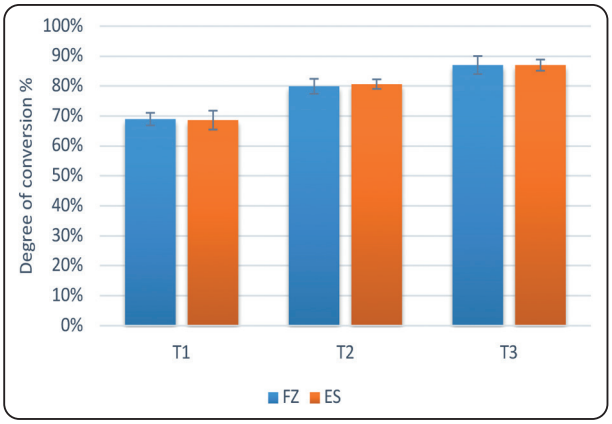

Fig. (1) Bar chart illustrating mean and standard deviation SD values of degree of conversion (%).

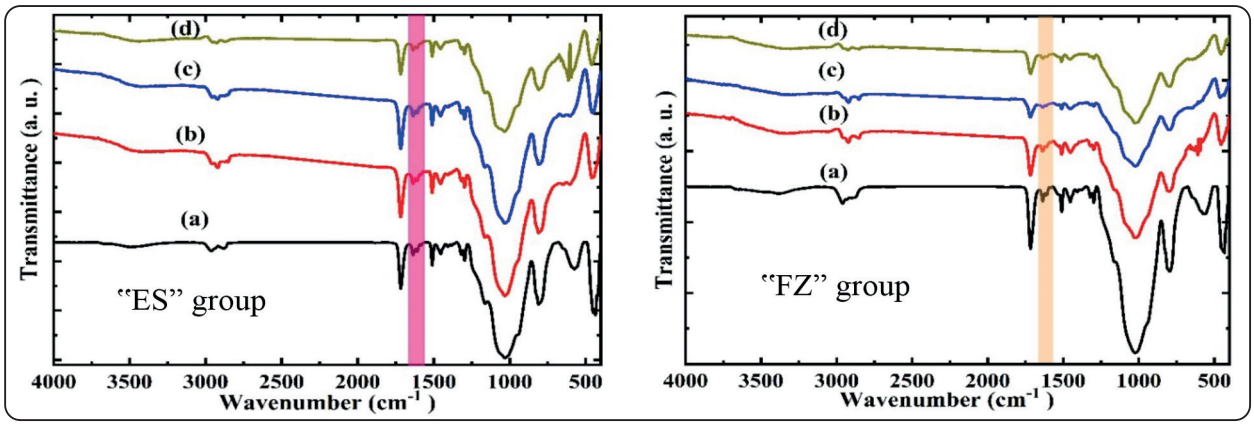

Fig. (2) FTIR plots showed the DC for "ES" and "FZ" groups where; (a) uncured, (b) cured for one second, (c) cured for three second, (d) cured for ten second

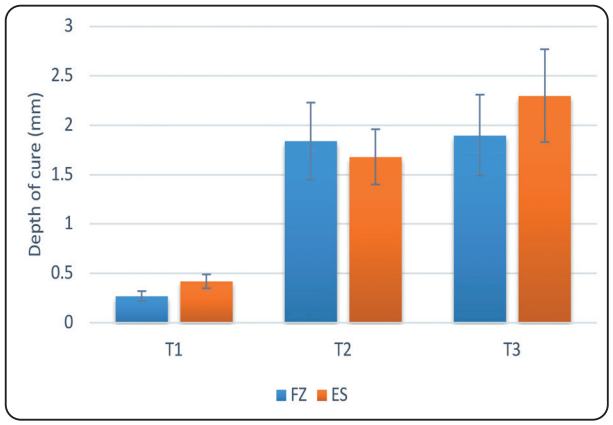

Fig. (3) Bar chart illustrating mean and standard deviation measurements of depth of cure (mm).

DISCUSSION

The current study was carried out to evaluate the effect of curing time on degree of conversion and depth of curing of supra-nano filled resin composite compared to a conventional one.

The primary goal of studying resin composite is to attain proper restoration with an excellent clinical performance [2]. The improvement of resin composite fillers allows better bonding to the resin matrix, and subsequently enhances the properties of RCs [13]. The resin matrix surrounds fillers in a three-dimensional network structure after curing; typically, it has been treated with a coupling agent to strengthen the binding and permit stress transmission between the fillers and the matrix [9]. It is important to achieve adequate polymerization to reduce the residual monomer that washed out from the polymerized resin which may affect the clinical success of restorations [14]. During the polymerization process, all resin monomers should be transformed into polymers to obtain a high a DC [15]. In other words, the high DC influences the RCs physical and mechanical properties [16].

The DC in the present study was measured using FTIR, which is a trusted method for DC evaluation of resin composites. This technique detects the C=C bonds stretching vibrations, which are involved in polymerization process before and after curing of the RCs. This method calculates the degree of conversion by comparing the heights of the peaks that correspond to aromatic and aliphatic double bonds [10].

Results of degree of conversion revealed that there was significantly increased in the DC with the increase of curing times regardless composite type. The three evaluated curing times showed a significant difference between them; where the T3 (ten seconds) showed the highest DC, followed by T2 (three seconds) while T1 (one second) showed the lowest DC. This can be explained by the influence of the curing time and light intensity on the DC. Moreover, the type of curing device and the

distance between the tip of the light source and the restoration affecting the DC [5, 15].

In this study, effects of different curing times (one, three and ten seconds) degrees of conversion and depth of curing of supra-nano filled resin composite were evaluated compared to a conventional one using LED light curing unit with a light intensity 1000 mW/cm2 ~2500m W/cm2. This type of light curing units allows adequate polymerization at short exposure times starting from 1 second at high irradiance. During the specimen's preparation, a thin glass slide (1 mm thickness) and a mylar strip were used in order to establish constant proportion from light source tip and the resin. Using such thin glass slide wouldn't affect light intensity, thus the polymerization degree [17].

This outcome was commensurate with another study [18] which recorded that three second curing time show higher DC when compared to one second. Moreover, this results agreed with another study demonstrated that Led curing units produce high DC [17]. However, it disagreed with a previous study [3] which report that less DC of both "ES" and "FZ" resin composite. This difference may be attributed to the different curing times.

Furthermore, there was no statistically significant difference between "ES" and "FZ" groups. This may be due to similar matrix composition of the two groups of Bis-GMA and TEGDMA. When Bis-GMA is diluted with low-viscosity monomer as TEGDMA, it results in increase in the DC [1]. This finding agreed with other studies [1, 8] which demonstrated high DC with the nanofilled resin composite by the increase of exposure time. However, this result was contradicted by a previous study [19] that reported that the nano filled resin composite demonstrated unacceptable degree of conversion. This difference may be attributed to the difference in curing unit and curing time applied, in addition to the DC evaluation methods as this study used Vickers test to determine the DC.

Despite the light activated RCs used in various restorative application, there are still controversies about the depth of curing of the light activated resin[7]. The irradiation time and the light curing unit is considered crucial factors to obtain acceptable depth of cure for resin composites. Up till now 20 seconds curing time is usually recommended for layered composite restorations of 2mm thickness[18]. Today, there are a lot of ultrafast curing devices available within the market, which promote high light intensity in shorter time[5]. Therefore, the use of high-intensity curing unit which allows less exposure time is necessary for uncooperative patients [18].

The properties of the polymerised resin composite have been assessed using a variety of methods that take the depth of curing into consideration. These methods include scraping away the unset material and measuring the top and bottom hardness and degree of double bond conversion in the polymer. The depth of cure metric is defined as the remaining specimen height. as stated by the ISO standard for dental resins [10]. Consequently, the depth of RCs curing was assessed using the scraping away approach.

Results of depth of curing revealed a significant difference between "ES" and "FZ" groups at T1 where; "ES" group revealed a significant higher curing depth than "FZ". This may be attributed to filler type and content of "ES" group. As Estelite®Alpha is supra nano spherical filled resin composite which contain 82 % silica-zirconia filler by weight. Also, the presence of nano spheroidal zirconia particles results in high DC and hardness value of the resin [3].

However, "ES" and "FZ" groups showed no significant difference at T2 and T3. Where T3 and T2 showed a significantly higher depth of cure values than T1. This may be explained by the fact that the depth of cure increase as the polymerization time increased. Also this might be correlated to

increase of DC by increase in curing time [1]. In addition to, the depth of cure of RCs is dependent on many factors as exposure time, the light device type, distance from light source, light intensity, filler type and quantity, resin composition, and transmitting property [5].

This finding was compatible with ***Bayrak etal., 2022*** result which demonstrated improved depth of curing with an increase in the light-curing intensity and time where; 10 second curing time was efficient to produce accepted depth of curing [20]. However, this result was contradicted by a previous study [21] that reported lower depth of cure of the conventional RCs. This difference may be attributed to curing time, composite type, curing unit and the study design as they compared it with bulk fill resin composites.

CONCLUSIONS

Considering the limitations of this study, it could be concluded that the increase of curing time is positively affecting both degree of conversion and depth of curing. Moreover, the short curing time was beneficial to obtain adequate depth of curing in supra-nano filled resin composite.

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