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Edited by Hiroj Bagde
E-mail: hirojbagde8@gmail.com
Phone: +91 9766105900

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Effect of thermally assisted light polymerization technique (TALP) on resin based composite containing newer photo initiator systems - An *in vitro* study

Vankala Gautam¹, Srinidhi Vishnu Ballullaya¹, Dhana Prakash Gurram^{2*}, B. Priyanka², Ramesh Penumaka³ & Saveri Katragadda⁴

¹Department of Conservative Dentistry and Endodontics, St Joseph Dental College, Duggirala, Eluru, Andhra Pradesh, India;

²Department of Conservative Dentistry and Endodontics, Drs Sudha & Nageswara Rao Siddhartha Institute of Dental Sciences, Andhra Pradesh, India; ³Department of Prosthodontics & Crown and Bridge & implantology, Drs Sudha & Nageswara Rao

Siddhartha Institute of Dental Sciences, Andhra Pradesh, India; ⁴Department of Orthodontics and Dentofacial Orthopedics, Drs Sudha & Nageswara Rao Siddhartha Institute of Dental Sciences, Andhra Pradesh, India; *Corresponding author

Affiliation URL:

<https://stjosephdentaleluru.com/>

<https://sids.edu.in/>

Author contact:

Vankala Gautam - E-mail: vankala.gautam@gmail.com

Srinidhi Vishnu Ballullaya - E-mail: vbuidhi@gmail.com

Dhana Prakash Gurram - E-mail: drdhanaprakashendo@gmail.com

B Priyanka - E-mail: priyanka8487@gmail.com

Ramesh Penumaka - E-mail: ramesh51endo@gmail.com

Saveri Katragadda - E-mail: saveri.katragadda@gmail.com

Abstract:

Optimal polymerization of resin composites without harmful thermal effects on dentin and pulp remains a clinical challenge. Therefore, it is of interest to evaluate the effect of pre-heating to 60°C on microhardness, conversion, curing depth, photoinitiation and temperature changes in four composite systems. Microhardness, FTIR, UV-Vis spectrophotometry and customized thermal analysis were used for evaluation. Pre-heating significantly improved microhardness and depth of cure, while intradental temperature increased by only 0.77–1.43°C and conversion remained comparable in most groups. Thus, data shows the pre-heating resin composites to 60°C may enhance polymerization and mechanical performance without clinically significant thermal effects.

Keywords: Degree of conversion, depth of cure, intra dental temperature, intra pulpal temperature, UV-Vis spectrophotometer, vickers hardness number

Background:

The polymerization of light-cured dental composite resins commonly relies on a camphorquinone (CQ)-tertiary amine photoinitiator system [1]. Upon light irradiation, CQ becomes excited and interacts with a tertiary amine to form an exciplex that generates free radicals and initiates polymerization [2]. Because CQ alone has limited initiation efficiency, tertiary amines are incorporated to accelerate free-radical formation and the polymerization reaction [1, 2]. Efforts to improve photoinitiator systems have focused on enhancing initiation efficiency, matching initiator absorption with curing-light emission, reducing discoloration and generating more effective reactive centers [2, 3]. CQ is a Norrish type II photoinitiator that requires a reducing agent; whereas Norrish type I photoinitiators can directly produce free radicals without an additional co-initiator [4]. Alternative photoinitiators may improve color stability, reduce the potential adverse effects associated with CQ-amine systems and enhance monomer conversion. These alternative systems include acyl phosphines, acyl phosphine oxides and titanocenes, which may partially or completely replace CQ [5]. Among the newer photoinitiators, trimethylbenzoyl-diphenylphosphine oxide (TPO) has been extensively investigated because of its high polymerization rate, improved degree of conversion and reduced yellow discoloration [2, 3]. Radically amplified photopolymerization has also been proposed to reduce the amount of CQ by incorporating innovative co-activators into the photoinitiator system [6]. Pre-heating highly viscous posterior composites to approximately 48–60°C reduces viscosity and improves handling while preserving the properties of conventional restorative

composites [4]. Pre-heating has also been associated with improved monomer conversion, microhardness and polymerization efficiency because increased molecular mobility facilitates free-radical propagation [7]. However, evidence regarding thermally assisted polymerization of composite resins containing different photoinitiator systems remains limited [8]. Therefore, it is of interest to report the effect of pre-heating on the microhardness, degree of conversion, depth of cure, photoinitiation behavior and intradental and intrapulpal temperature changes of composite resins containing different photoinitiator systems.

Materials and Methods:

The research protocol of the present study was approved by the institutional review committee. Sample size was calculated using G*Power Version 3.1.9.2 considering 95% Confidence level with 80% power. The dental composite resins were tested for dentine and intrapulpal temperature on preheating, the type of photoinitiators by UV-Vis spectroscopy, microhardness, depth of cure and degree of conversion.

Composite resins and photo initiators:

Four commercially available dental composite resins with different photo initiator system Camphorquinone (CQ), Ivocerin and Camphorquinone (IC & CQ), Camphorquinone and trimethyl-benzoyl diphenylphosphine oxide (CQ & TPO) and Radical amplified polymerization (RAP) were taken in the study. The composition and photoinitiators of dental composite resins are listed in Table 1. The composite resins were dispensed into composite dispensing cavifills and colour coded to represent

each composite resin type. The operator was trained in the dispensing of composite resin and usage of light curing unit.

Customized composite preheater model:

The apparatus comprises of a composite syringe holder, digital temperature controller with J-type thermocouple, 200 W bulbs to generate heat, a thermostat for heat control and a micro fan for uniform dissipation of heat.

Dentine and intrapulpal temperature profile:

Teeth required for this study was sourced from the department of oral surgery from patients undergoing extraction for the periodontal reasons. Informed consent was taken from the patient and institutional ethical clearance was taken from the patient. After extraction, the teeth were cleaned, calculus removed and stored in 10% formalin until use. Four teeth specimens were sectioned 2mm below the CEJ. The pulp chamber was cleared off pulp tissue and pulp stones and ultrasonicated for five minutes. Class I cavity was prepared of dimension 3x3x3 mm. The model consists of a water bath with thermostatically controlled temperature at 37°C. Each tooth was fixed to a plexiglass sheet with cyanoacrylate glue. Polyethylene tube of 4 mm diameter was penetrated into the plexiglass sheet to perfuse the pulp chamber with sodium chloride (0.09 g/l). A hole was drilled in a plexiglass sheet to enable insertion of a k-type thermocouple for measuring intrapulpal temperature. A second hole was drilled from the proximal side to allow the k-type thermocouple to be inserted adjacent to the buccal wall of the cavity to measure intradentinal temperature. The thermocouple was then attached to 8 channel data logger for data acquisition. The preheated composite was inserted into the prepared class I tooth preparation and the temperature profiles were recorded at five intervals as explained by Balestrino *et al.* [7] (a) at the time of composite placement and contouring (b) during light-curing (c) 20 seconds after light curing. After 2 minutes waiting period, the samples received a second post-cure (to measure rise of temperature solely by curing light). Each composite resin was tested for temperature profiles quadruplicate.

Absorption spectra by UV-Vis spectroscopy:

200 mg of composite resin was mixed in 1000µl of HPLC grade methanol. The mixture was ultrasonicated for 10 minutes, followed by centrifugation at a speed of 13000 rpm for 200 seconds to allow filler sedimentation. The supernatant was collected and immediately placed in a glass-slide cuvette. Absorption spectra of the photoinitiators were measured using a UV-Vis spectrophotometer (Agilent Technologies Ltd., U.S.A) over the range of 350-520 nm. The investigation was performed in triplicate.

Microhardness test:

Four different dental composites resins at room temperature and after pre-heating were inserted into a Polyethylene cylinder of 4 mm height and 4 mm diameter in one increment resulting in sample size of 120 composite specimens (n=30 of each dental

composite resin). A stainless steel matrix was inserted to bisect the composite resin and to provide a flat surface for Vickers microhardness measurement. The surface of the composite resin was covered with a layer of glycerin and was cured with Bluephase N LED light cure unit at an intensity of 1200w/cm² for 40 seconds. After curing, the composite resin samples were stored in a light-proof bottle containing distilled water for 24 hours at 37°C. The top and bottom surface microhardness was measured before removing it from the polyethylene cylinder. After removing the matrix, the samples were individually fixed in the holder such that the surface to be tested was placed perpendicular to the indenter tip. Measurements were made at 0.5 mm, 1.5 mm, 2.5 mm and 3.5 mm. The means of the three measurements were taken for assessing microhardness. All microhardness measurements were taken using a calibrated Vickers indenter using a load of 300 g and a dwell time of 10s.

Depth of cure:

The depth of cure (based on VHN measurement) is the level where the microhardness measurement equals the surface measurement multiplied by an arbitrary ratio of 0.8 or, in other words, 80% of the surface microhardness (HV 80%) [8].

Degree of conversion:

The degree of conversion (DC) was studied using Fourier transform-infrared spectroscopy (FTIR) (Agilent Technologies Ltd., U.S.A) in ATR mode. Composite was inserted into polyethylene tubing of 4 mm depth and 4 mm diameter placed on a glass slide. The composite resin was cured with a Blue phase N LED light-curing unit with an intensity of 1200w/cm² for 40 seconds. The final curing of the composite was done after applying a layer of glycerin to avoid an oxygen inhibited layer of surface composite. The glycerin was washed after curing. The cured samples were stored for 24 hours in distilled water at 37°C before testing. Two spectroscopic measurements were taken from the top and bottom of the composite surface. Three spectroscopic measurements were taken for four different composites in the uncured state, which was used as baseline values for calculating the degree of conversion of the cured samples. Infrared spectra were collected between 1637 and 1610 cm⁻¹ at a rate of one-per-second at 2 cm⁻¹ resolution. The DC (%) was calculated from the ratio between the peak intensities of the aliphatic C=C bond (1638 cm⁻¹) to the aromatic C=C bond (1608 cm⁻¹), obtained from the cured and uncured specimens by the following equation:

$$\text{DC (\%)} = \frac{\text{Absorbance cured } 1637 \text{ cm}^{-1} / \text{Absorbance cured } 1610 \text{ cm}^{-1}}{\text{Absorbance uncured } 1637 \text{ cm}^{-1} / \text{Absorbance uncured } 1610 \text{ cm}^{-1}}$$

Statistical analysis:

The statistical analysis was carried out with SPSS Version 20.0 (IBM, NY, USA). All the parameters associated with microhardness, depth of the cure and degree of conversion were subjected to descriptive analysis followed by two-way ANOVA and Tukey post hoc tests for multiple comparisons between 4 groups. All the tests were analyzed at statistical significance level set as p<0.05.

Results:

Use of preheated composite resin increased the intradentinal temperature by factor of 0.20°C - 0.50°C. On light curing there was a substantial increase of 2.0°C $\pm$ 0.4°C in the dentinal temperature. The exothermic heat of polymerization was 0.77°C to 1.43°C. UV-Vis spectroscopy measurements revealed CQ absorption band from 400 to 500 nm with peak at 480 nm. IC & CQ, CQ & TPO, RAP had absorbance peak at 420 nm, 410 nm and 390 nm respectively. Vickers microhardness values are depicted in (Table 4, 5). The highest hardness value was noted with Filtek Z350 XT both at room temperature and after pre-

heating at 60°C followed by Vit-l-escence, Tetric EvoCeram and Estelite sigma quick. For preheated composite resins 80% of the surface hardness was achieved at a depth of 3.5 mm for Vit-l-escence and Estelite sigma quick whereas FiltekZ350 and Tetric EvoCeram composite resins had hardness at 3.5 mm comparable to that of surface. At room temperature all composite resins had depth of cure of about 2-3 mm. The degree of conversion was in the range of 53.6°C $\pm$ 2.17°C to 71.73°C $\pm$ 2.1°C at room temperature whereas after pre-heating at 60°C there was higher degree of conversion for all composite resin tested which ranged from 71.07°C $\pm$ 3.1°C to 75.4°C $\pm$ 2.8°C (Table 2, 3).

Table 1: Characterization of the materials used

S.no	Brand and type of composite	Composition	Photoinitiator system	Suggested wavelength	Manufacturer
1.	FILTEK Z 350 Nanocomposite	Bis-GMA, Bis-EMA, UDMA,TEGDA; camphoroquinone; silica(non-clustered) 20nm+zirconia(non clustered) (4-11nm) +silica/zirconia nanoclusters (0.6-1.4micron), 63.3 vol%	Camphorquinone (CQ)	460-470 nm	3M, Saint Paul, Minnesota, United States
2	TETRIC EVO CERAM Nano hybrid	Bis-GMA, UDMA Fillers: Barium glass, ytterbium trifluoride, oxides and prepolymers Catalysts and Stabilizers:0.3% Pigments, Camphorquinone (CQ), trimethylbenzoyldiphenylphosphineoxide (TPO) photoinitiators<0.1	Ivocerin and CQ	411-418nm	Ivoclar Vivadent Amherst, New York, USA
3	VIT L ESCENCE Microhybrid	Bis GMA, TEGDMA; Fillers: barium boron aluminium particles of 0.4 -0.7nm; 61 vol% ;78 % weight	CQ &TPO	390-392nm	Ultradent products West Ultradent Drive South Jordan, UT.
4	ESTELLITE SIGMA QUICK Supra Nano composite	Bis-GMA, TEGDMA Filler: SiO ₂ -ZrO ₂ , SiO ₂ -TiO ₂ 71% volume ;82% weight	RAP (radical amplified polymerization)	390-397nm	Tokuyama, Ontario, Canada

Table 2: Mean comparisons of microhardness of the top surface between 4 groups at room and preheated 60°C temperature with two way ANOVA

Position	Material	Material	Material	Material	
	TEC	FIL	VIT - I	ESTELLITE	p value
Room temp	54.3 $\pm$ 4.70	70.39 $\pm$ 2.40	62.76 $\pm$ 2.71	46.22 $\pm$ 1.19	p < 0.001*
Preheated at 60°C	59.24 $\pm$ 2.09	72.96 $\pm$ 2.59	68.42 $\pm$ 1.93	53.49 $\pm$ 1.18	p < 0.001*

*p value < 0.05 denotes statistical significance.

Table 3: Mean comparisons of microhardness between 4 groups of the bottom surface at room and preheated 60°C temperature with two way ANOVA

Position	Material	Material	Material	Material	
	TEC	FIL	VIT - I	ESTELLITE	p value
Room temp	42.17 $\pm$ 1.97	53.36 $\pm$ 1.82	43.63 $\pm$ 2.91	37.9 $\pm$ 1.41	P < 0.001*
Preheated at 60°C	56.27 $\pm$ 3.01	64.94 $\pm$ 1.70	63.38 $\pm$ 1.71	49.21 $\pm$ 1.48	P < 0.001*

*p value < 0.05 denotes statistical significance.

Table 4: Mean comparison of degree of conversion between 4 groups using two ways ANOVA of top surface at room and preheated temperature.

Position	Material	Material	Material	Material	
	IC&CQ	CQ	CQ&TPO	RAP	p value
Room temp	62.47 $\pm$ 2.68	53.6 $\pm$ 2.17	71.73 $\pm$ 2.1	63.4 $\pm$ 2.5	P < 0.001*
Preheated at 60°C	71.07 $\pm$ 3.1	72.3 $\pm$ 6.2	75.4 $\pm$ 2.8	73.7 $\pm$ 2.7	P < 0.010*

*p value < 0.05 denotes statistical significance.

Table 5: Mean Comparison of Degree of Conversion between 4 groups using two ways ANOVA of Bottom surface at room and preheated temperature.

Position	Material	Material	Material	Material	
	IC&CQ	CQ	CQ&TPO	RAP	p value
Room temp	52.53 $\pm$ 3.2	35.9 $\pm$ 2.7	48.2 $\pm$ 1.6	53.2 $\pm$ 2.3	p < 0.356
Preheated at 60°C	55.7 $\pm$ 3.08	42.8 $\pm$ 2.2	64.4 $\pm$ 3.3	59.2 $\pm$ 1.6	p < 0.368

Discussion:

The first hypothesis was rejected because pre-heating improved the microhardness, depth of cure and degree of conversion of the tested composite resins. The second hypothesis was also rejected because composites containing alternative photoinitiator systems showed differences in conversion compared with the conventional camphorquinone system. Few studies have simultaneously evaluated the influence of pre-heating on composites containing different photoinitiators. Therefore, four commercially available composites representing CQ, Ivocerin-CQ, TPO and radically amplified photopolymerization systems were investigated. A pre-heating temperature of 60°C was selected because this temperature promotes monomer mobility and is commonly used in commercial composite-warming devices [3, 4, 9 and 10]. Pre-heating has also been reported to improve handling and selected mechanical properties by reducing composite viscosity [11]. Jakupović *et al.* demonstrated an approximately 2.5-fold reduction in viscosity following pre-heating and further showed that material temperature declines rapidly after removal from the heating device [3]. In the present study, the observed thermal behavior was consistent with previous evidence that the actual temperature of the composite delivered into the cavity is lower than the temperature selected on the pre-heating unit [4]. Pre-heating improved the depth of cure of all tested composites. Albuquerque *et al.* reported a greater depth of cure with a CQ-amine system than with a TPO-based photoinitiator system [12]. In contrast, the present findings showed no marked influence of photoinitiator type on curing depth at either room temperature or after pre-heating. This difference may reflect variations in resin composition, filler characteristics, photoinitiator concentration and the spectral output of the curing unit. All composites demonstrated higher microhardness after preheating, with significant differences between specimens polymerized at room temperature and those polymerized after warming. The upper surfaces showed greater microhardness than the lower surfaces, reflecting the progressive attenuation of curing light through the material. A specimen thickness of 4 mm was selected because newer photoinitiators are increasingly incorporated into materials intended to reduce yellow discoloration and permit greater curing depths. Filtek Z350 XT demonstrated the highest microhardness, which may be related to its nanofiller and nanocluster structure and the optical and mechanical behavior of its zirconia-silica fillers. Ayub *et al.* similarly reported superior microhardness for a nanofilled composite after pre-heating, whereas Vit-l-escence showed lower hardness values [13]. The present results therefore suggest that filler composition and structure may exert a greater influence on surface hardness than photoinitiator type alone. The degree of conversion of resin composites at room temperature has generally been reported to range from approximately 50% to 70% [14]. Pre-heating provides additional thermal energy, reduces viscosity and increases molecular mobility, thereby allowing greater propagation of free radicals before vitrification limits further polymerization [15, 16]. This mechanism may explain the higher degree of conversion

observed after pre-heating in all tested materials. The increase in conversion following pre-heating agrees with the observations of Mundim *et al.* [17]. Although a relationship between degree of conversion and microhardness has been reported, this association was not evident in the present study. Filtek Z350 XT showed the greatest hardness, whereas Vit-l-escence demonstrated the highest degree of conversion, indicating that these properties are influenced by different compositional factors. A similar divergence between hardness and conversion has previously been described [1]. The higher conversion of Vit-l-escence may be associated with differences in monomer content and resin-matrix composition. Ribeiro *et al.* also found greater conversion for Vit-l-escence than for Filtek Z350 irrespective of the curing unit used [18]. The greater conversion of the microhybrid Vit-l-escence compared with the nanofilled Filtek Z350 XT is also consistent with previous findings [19, 20]. Differences in the organic matrix, filler volume, particle type and light transmission may explain the variation among materials. Overall, the findings indicate that pre-heating improves polymerization-related properties, while the magnitude of improvement remains dependent on the specific composition and photoinitiator system of each composite.

Conclusion:

We show the pre-heating resin composites improved microhardness, depth of cure and degree of conversion without producing harmful thermal changes. Alternative photoinitiator systems may further enhance polymerization, particularly when activated with compatible polywave curing units. Thus, clinical pre-heating should follow manufacturer recommendations because not all composite materials are suitable for warming.

Conflict of interest:

No conflicts of interest to declare.

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