



Effect of Different Storage Temperatures on Degree of Polymerization of Tetric Evoceram Bulk Fill Composite Resin (An in vitro study)

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Abstract

Aims: This study aims to evaluate the effects of different storage temperatures -18°C, 5°C, 23°C and 40°C on degree of conversion of bulk fill composite. **Materials and Methods:** The tetric evoceram was randomly allocated into four equal treatment groups (n=10/group) according to the storage temperatures: A: 23°C (control), B: 5°C, C: -18°C and D: preheated to 40°C. After the material removal from storage. The forty-disc shape specimens were prepared with 8mm diameter and 4mm thickness all specimens cure with monowave LED light (LEDition light cure). After photoactivation, the specimens were stored in artificial saliva for 24 hours, at 37°C for complete polymerization. The degree of conversion of the top and bottom surface of composite resin specimens were determined after 24 hours of curing using FTIR-ATR. **Results:** There was a significant difference in different storage temperatures on degree of conversion. As the temperature of resin composite increase, the top and bottom degree of conversion of the specimens also increased. **Conclusions:** The degree of conversion of bulk-fill composite resin increase as the increase of pre-cure temperatures.

الخلاصة

الأهداف: تهدف الدراسة الى تقييم تأثير درجة حرارة الخزن على درجة التحول (البلمرة) لمادة الراتنج المركب ذات التعبئة بالكتلة الواحدة. **المواد وطرائق العمل:** تم تقسيم مادة الراتنج المركب ذات التعبئة بالكتلة الواحدة (TECBF) عشوائيا حسب درجة حرارة الخزن الى أربع مجاميع متساوية (عدد= 10 لكل مجموعة)، المجموعة الأولى (أ) تحفظ بدرجة حرارة الغرفة 23 ± 5°C، المجموعة الثانية (ب) تحفظ بدرجة حرارة 5°C، المجموعة الثالثة (ج) تحفظ بدرجة حرارة -18°C والمجموعة الرابعة (د) تسخن الى درجة حرارة 40°C. تم تحضير نموذج على شكل قرص دائري قطره 8 ملم وسمكه 4 ملم. بعد اكمال عملية التصلب الضوئي تم حفظ النماذج في علب تحوي على لعاب اصطناعي، وتم خزنها في حاوية بدرجة حرارة 37°C لمدة 24 ساعة لاكتمال البلمرة بعد 24 ساعة من الخزن، تم قياس درجة البلمرة للوجه العلوي والسفلي لنموذج الراتنج المركب باستخدام جهاز مقياس الاطياف بالأشعة تحت الحمراء. وتم استخدام One-Way ANOVA لغرض التحليل الاحصائي عند (p=0.05). **النتائج:** اظهرت النتائج بوجود فرق معنوي في درجة البلمرة لمادة الراتنج المركب ذات التعبئة بالكتلة الواحدة باختلاف درجة حرارة التخزين. حيث ان رفع درجة حرارة مادة الراتنج المركب ادى الى زيادة درجة البلمرة. **الاستنتاجات:** رفع درجة حرارة مادة الراتنج المركب ذات التعبئة بالكتلة الواحدة قبل التصلب أدى الى زيادة درجة البلمرة.

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INTRODUCTION

Composite resin is among the most popular aesthetic restorative materials in dental clinical practice. The use of these materials has been on rise for the past several years to re-establish form, function, and aesthetic of teeth and thus, have become an integral part of modern, esthetic dentistry⁽¹⁾.

Since their Composite resin introduced in the dental market, their monomer chemistry, and filler technology have been continuously developed to improve their physio-chemical properties. A new class of resin base composite is introduced into the dental market with the purpose of time saving and simplification called bulk fill composite resin⁽²⁾.

Bulk- fill base composite resin claims to allow the use of material increment up to 4mm in thickness with control polymerization process and ensure a proper depth of cure. These advantages with bulk- fill composite may be due to enhanced light transmission properties due to reduction of light scattering at filler-matrix interface by either decreasing the filler amount or increasing the filler size⁽³⁾.

There are several factors affect the polymerization rate. Temperatures factor play an important role in the polymerization process, which alter the mechanical and physical properties of composite resin, composite is a visco-elastic material, which responds to an

increase or decrease in the external temperature.⁽⁴⁾ Dentists store composite resins in refrigerators to prolong their shelf lives usually at temperatures ranging from 2°C to 5°C as recommended by the manufactures⁽⁵⁾. Darnoch et al. (2006) reported that the degree of polymerization is decreased at 3°C and increased at a 60°C⁽⁶⁾. Other studies found no adverse effect from using materials directly from refrigerated storage⁽⁷⁾.

In addition, several dentists increase the temperature of composite resin to improve their handling characteristics. The preheating process of the composite lead to improve in mechanical properties of composite resin because of the increased rate of monomer conversion⁽⁸⁾.

Castro et al. (2013) stated that preheated resin composites could improve the mechanical properties of composite resins and attain the low viscosity⁽⁹⁾. Upon heating the composite resin, more free radical generation and addition polymerization leads to high DC. Upon heating, the resin viscosity is reduced due to the thermal vibration force that allows the monomers to slide easily to each other, which in turn improve adaptation of the material to the cavity walls, and reduces microleakage, thus diminishing the extrinsic staining of restoration.⁽¹⁰⁾

The relationship between pre-cure temperature and DC of composite resin have been studied in wide range, however

there are very limited studies investigated the effect of pre-cured temperature on DC of bulk-fill composite with no available data about the possible effect of -18 °C on degree of polymerization of bulk- fill composite .

Null hypothesis: No effect of different storage temperature on the DC of bulk- fill composite resin.

Aims of study

The objective of this study was to investigate the possible effect of different storage temperatures on DC of the bulk fill composite resins.

MATERIALS AND METHODS

Tetric EvoCeram bulk-fill composite resin (TECBF) of shade IVA, was used in this study. TECBF (ivoclar, Schaan, Liechtenstein) is a nano-hybrid composite for the fabrication of direct restoration in posterior teeth. TECBF has both camphorquinone, TPO and ivocerin initiators that allow polymerizing material at greater depth. ⁽¹¹⁾

Storage Temperature

The TECBF composite resin was divided into four groups according to storage temperatures as the following: ⁽¹⁰⁾.

1-Group A (control): Composite resin material stored in an incubator at room temperature $23 \pm 1^{\circ}\text{C}$ for 3days before sample preparation.

2-Group B: The composite resin material was subdivided into small increments and placed in well-seal container to prevent temperature variation

at sample preparation time. This material stored in the refrigerator at 5°C for 3 days before sample preparation.

3-Group C: The composite resin material was subdivided into small increments and placed in well-seal container to prevent temperature variation at sample preparation time. The composite resin material stored in the freezer at -18°C for 3 days before sample preparation.

4- Group D: The composite resin material was pre-warmed to 40°C using a Calset, composite warming unit (Ad Dent, Inc. Danbury,CT, USA). That was preset to 40°C . Composite resin applied into the heating device and maintained in place for 3 days. The storage temperatures were measured with a K-type thermocouple (TM-902C Digital sensor LCD thermometer, Hanau, Germany) ⁽¹¹⁾.

Sample Preparation

Forty-disc shape specimens 8mm in diameter, 4mm in thick prepared in dark room and verified by electronic digital caliper (digit-cal capau system, S/N 8R565806, China) ⁽¹²⁾.

The specimens fabricated by inserted composite resin in single incremental thickness 4mm into polytetrafluorethylene mold, which stood on a glass slide with transparent polyester strip. The top surface covered with another polyester strip to avoid oxygen inhibition, another glass overlaid the mold to ensure a smooth surface of samples with no need for finishing and polishing ⁽³⁾. Standard

pressure 1kg was applied to the glass slide for a duration of 15seconds to provide uniformity of sample surface by allowing the excess resin to extrude from the specimen surface Figure (1) ⁽¹³⁾. After the load removed, the light cure was applied

on the top surface of the specimen. The light cure tip was positioned perpendicular to the specimens' and the distance between specimen and light tip was standardized using 1mm glass slide ⁽¹¹⁾.



Figure 1: photograph for sample preparation and curing



Figure 2: FTIR-ART

All specimens were stored in an individual lightproof container with artificial saliva for 24 hours at 37C° in an incubator ⁽¹⁰⁾. Artificial saliva was prepared at the following proportion: NaCl 0.40G/L, KCl 0.40G/L, NaH₂PO₄ 0.79 G/L, CaCl₂·2H₂O 0.79G/L, Co (NH₂) 1.0G/L, Distilled water 1000ml. saliva PH= 7 measured by PH meter (Professional, Bench top, PH meter, Bp3001. Malaysia) ⁽¹⁴⁾.

Degree of Conversion (DC)

Attenuated total reflection Fourier transform infrared spectroscopy FTIR-ATR (Tensor 27, Bruker Optics, Ettlingen, Germany) was used to measure DC ^(15,16).

The DC of dimthacrylate composite resin was calculated by comparing the peaks derived from cured and uncured resin. The top and the bottom surface of each sample were tested for absorbance characteristics after 24 hours of polymerization ⁽¹⁶⁾. The sample was located in a close- fitting contact with the diamond crystal (Bruker Alpha); after infrared penetration the sample, the reflected beam is completely redirected from the boundary between the sample and crystal to detector in the infrared spectrometer. The absorbance rate was recorded at wavelength of 500-4000cm⁻¹ ⁽¹⁷⁾. Three samples from each group before cured were used to test for initial

absorbance by applying the uncured composite onto crystal. The mean of uncured composite was obtained and then used to compare with cured composite ⁽¹⁸⁾.

Degree of polymerization of resin composite measured by assessing the variation in peak height ratio of

absorbance intensities of carbon double bond peak at 1637cm⁻¹ and that of an internal peak at 1608cm⁻¹ (aromatic carbon double bond) during polymerization, in relation to the uncured material. The degree of polymerization was established according to the formula:

$$\%DC = \left[1 - \frac{Abs\left(\frac{1637cm^{-1}}{1608cm^{-1}}\right)_{polymerized}}{Abs\left(\frac{1637cm^{-1}}{1608cm^{-1}}\right)_{unpolymerized}} \right] \times 100$$

Abs 1637cm⁻¹ is aliphatic c=c absorbance at 1637cm⁻¹, Abs 1608 cm⁻¹ is aromatic c=c absorbance at 1608 cm⁻¹ ⁽¹⁹⁾

temperature for curing the bulk-fill composite.

Data Collection and Statistical Analysis

The following statistical methods were used to analyze and assess the result via SPSS v.19 for windows

- 1- Descriptive statistics include, mean and standard deviation.
- 2- Analysis of variance (ANOVA) was used to find the effect of different storage temperatures on degree of conversion followed by Duncan test to find the best

RESULTS

Composite temperatures before photoactivation had a significant influence on DC (p<0.05). When the temperature was elevated above the room temperatures, the DC was increased. Mean value of polymerization for temperature factor of all groups can be seen in Table (1). The highest mean value of DC was observed in D top and bottom 70.3±3, 63.97±4.0 % respectively. The lowest mean value of DC was found in C top and bottom surface 56.7±3.5, 47.76±3.6 % respectively.

Table (1): Mean value and standard (±SD) deviation of DC of composite resin (%) at different storage temperatures.

Group	N	Mean ± SD Top surface	Mean ± SD Bottom surface
A	10	66.60±3.8	59.10±3.2
B	10	60.85±2.9	53.71±3.4
C	10	56.7±3.5	47.76±3.6
D	10	70.3±3	63.97±4.0

The degree of polymerization was increased with increasing in the temperatures, and it showed a significant difference $p < 0.05$. This confirmed by One-way analysis of variance (ANOVA) degree of conversion. Further tests

performed using Duncan Multiple Analysis Rang Test that showed significant difference among treatment groups. The results of the Duncan test can be seen in Table (2).

Table (2): A comparison between the top surfaces of resin samples at different storage temperatures.

Temperatures	p-value	Duncan
A		c
B	.000*	b
C		a
D		d

According to ANOVA $p < 0.05$ for each group, means with different small letters vertically have a significant difference at $p \leq 0.05$ according to Duncan multiple analysis rang test. A= (23±1 °C), B= (5 °C), C= (-18 °C), D= (40 °C).

DISCUSSION

The effect of the storage temperatures on DC was detected in the in this study, which varies with different temperatures levels. Different treated temperatures have a significant effect ($p < 0.05$) on the DC compared with the control group. The null hypothesis is rejected.

The result in this experiment demonstrated that higher temperature would have a strong and positive influence on the polymerization kinetics of composite resin. As the temperatures of composite resin increased in this study from room temperature 23±1°C to 40°C the degree of conversion of composite resin was increase. Several researchers have found that, high storage temperatures accelerate the polymerization reaction and increase degree of conversion of

composite resin. This occurred because the viscosity of the composite resin augment upon increased in temperatures, which enhances the molecular mobility, the diffusion –controlled propagation rate increased, diffusion-controlled termination rate decreased and auto-acceleration occurs due to the trommsdorf effect, which leading to an increase in monomer conversion⁽²⁰⁾. Our finding is in line with Awliya (2007), who recorded that a higher monomer to polymer conversion as the pre cure temperature increases from 5°C to 37°C⁽²¹⁾.

In addition, the results in this study are in agreement with Alshaafi (2016), who reported the greater monomer conversion, when the composite temperatures increase from 23°C to 33°C⁽²²⁾.

Additionally, the results in this study are in agreement with Tauböck et al (2015), who found only one of the five tested composites resin (TECBF) showed a significant increase in polymerization rate upon elevated storage temperatures from room temperatures to 68°C⁽¹¹⁾. We expect this is due to TEC photoinitiators system. Tetric evocearam is containing additional photoinitiator ivocerin and TPO besides camphorquinone. The ivocerin initiators compared to CQ yield high conversion and may be affected by pre-cured temperatures.

In this study the bulk fill composite resin stored at 5°C showed a value of degree of conversion lower than those composites resin store at room temperature or pre-heated. The low DC at low storage temperatures occur because the low temperatures increase the viscosity of composite resin that affects the movement of molecules during the polymerization reaction⁽²³⁾. This finding is in agreement with Mary et al (2019), Who reported that the degree of conversion of composite resin decreases 10°C and increases as the increased of the per –cure temperatures to 68 °C⁽²⁴⁾.

In addition, these results are in agreement with Puspitasari et al (2019), who found that a higher means value for degree of conversion of the Tetric N Ceram bulk fill composite resin when stored at 35°C but the lowest mean value occurred in the 5°C⁽¹⁰⁾.

In this study, the low DC in-group C is due to the vibration of the atomic molecules that decreases as the storage temperatures decrease and the distance between the atoms gets closer. When the temperature reaches absolute zero, the atom will stop moving. The decrease atomic motion will increase the viscosity of the material and the material may appear cloudy which is an indication of the resin crystallization. If this continues to occur, there will be a change in the material structure⁽²⁵⁾.

CONCLUSION

This following can be concluded from this study:

- Preheating and precooling can affect the polymerization of resin composite.
- As the composite resin temperatures increased before photo-activation, the DC of the top and bottom surfaces of composite specimens increased.

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