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The effect of various pre-cured temperature of different resin materials on the degree of conversion and microhardness of cured composite resin

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الخلاصة

الأهداف : الاهداف: هدفت هذه الدراسة الى قياس تاثير درجة حرارة الحشوات االضوئية قبل تعريضها للتصلب على درجة التحول فى مادة الحشوات الضوئية بواسطة جهاز مقياس الاطياف بالاشعة تحت الحمراء Fourier Transform Infrared (FTIR, Bruker-TENSOR, Germany وعلى درجة شدة التصلب بواسطة جهاز قياسشدة التصلب (Spectroscopy- WOLPERT – WERKE –GMBH Germany) . **المواد وطرائق العمل** : تم عمل (45) انموذج على شكل قرص دائري قطره (8ملم) وسمك(2) باستعمال مادة الحشوات الضوئية من نوع Dentsply (CERAM.X shade D2بعد ان تم حفظ هذة المادة فى درجات حرارية مختلفة(5 ،25، 37) درجة سيليزية لمدة 24ساعة .تم حفظ العينات في داخل حاظنة في درجة حرارة 37 سيليزية لمدة 24 ساعة تعرضت للتصلب لمدة 40 ثانية. بعد ذلك تم قياس درجة التحول في مادة الحشوات الضوئية بواسطة جهاز مقياس الاطياف بالاشعة تحت الحمراء (FTIR) بينما تم قياس شدة التصلب بواسطة جهاز قياس شدة التصلب BaujahrTestor) (Germanyباستعمال فحص (Vickers Test) تم تحليل البيانات باستعمال .(Duncans (ANOVA) .**النتائج**: اظهر ا لتحليل الاحصائي (ANOVA)بانه توجد فروقات معنوية في درجة التحول وشدة التصلب بالنسبة لاختلاف درجات الحرارة وانه كلما زادت درجة حرارة مادة الحشوة الضوئية قبل تصلبها ازدادت درجة التحول وشدة التصلب لعينات الحشوة الضوئية .**الاستنتاجات** : ان رفع درجة حرارة مادة الحشوة الضوئية قبل تصلبها قد يحسن من درجة التحول وشدة التصلب للمادة بعد تعريضها للتصلب خصوصا إذا تم استخدامها في المناطق العميقة من الحشوات مما قد يزيد من معدل عمر الحشوة الضوئية.

ABSTRACT

Aims: The aims of this in vitro study were to evaluate the effect of different temperatures of the pre- cured composite resin materials on the degree of conversion and knoop microhardness of cured composite resin. **Materials and Methods** : A forty five disc-shaped specimens were prepared from (Ceram X, shade D2, Dentsply/ Caulk, USA) after stored for 24h in different temperatures (5,25,and 37°C) were light cured for 40s. The FTIR test was used to measure the degree of conversion for each specimen. The knoop microhardness was measured by the use of (WOLPERT-WERKE-GMBH - Baujahr Testor, GERMANY) (Vickers hardness test) for each specimen. Data obtained was analyzed using one-way analysis of variance (ANOVA) and Duncan's test at a 0.05 significance level.**Results** : The statistical analysis of the results (ANOVA) showed that there was statistically significant difference in the degree of conversion and in the microhardness of the prepared samples at the different temperature. As the temperature of the composite resin increase, there was an increase in the degree of conversion and increase in the micro hardness of the samples.**Conclusions** : The use of pre-warmed composite resin may help to improve the degree of conversion and the micro hardness of composite resin especially at the deeper areas of a restoration which could result in an increase in the expected life of a composite restoration.

Key words: Different temperature, Pre-cured composite resin, Degree of conversion, Microhardness.

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INTRODUCTION

Light-cured resin composites are widely used in dental restorations. These resins are polymerized by a light-curing unite (LCU) and the degree of conversion (DC) of dental resins can be determined using Fourier Transform-Infrared Spectroscopy (FTIR).[1,2] In addition, Knoop-microhardness has been shown to be a reliable method to determine how well a resin is cured [3] and a good correlation has been reported between the knoopmicro-hardness and both the DC [4,5] and the Young's modulus.[6] The overall rate of polymerization of resins is determined by the rates of the initiation, propagation and

termination phases of the reaction. The rate constant for initiation is independent of temperature, whereas the rate constants for both propagation and termination increase with temperature according to the Arrhenius dependence.[7] Using typical activation energies for propagation and termination, it has been estimated that the overall polymerization rate for dimethacrylate based resins will increase by 1.90% for each 1ºC rise in temperature. [8]

However, the rate of polymerization of filled dental resins is further accelerated by the Trommsdrof effect, where in the marked increased in the viscosity of the filled resin material during gel- ation increases the mobility of large polymer radicals. This effectively reduces the termination rate constant at a given temperature and accelerates the reaction. [9]

Kinetic studies of unfilled dimethacrylate resins using calorimetric techniques (10,11) have shown that for a given resin composition and light intensity, increasing the temperature up to 50 or 60ºC results in greater extent of conversion rate . In 2006, study by Daronch et al., [1] irradiation of Esthet X shade A2 with a QTH source emitting 630 mw/cm showed a marked increased in extent of conversion at the top and bottom surfaces of 2-mm thick samples when temperatures of the resin were increased from 3ºC to 60ºC.

The hardness of composite resin materials is influenced by several factors, such as organic matrix composition,[12] type of the filler particles,[13] and degree of conversion. [14] For many years, dentists were often asked to refrigerate their composite material before use. Although some studies found no adverse effects from using materials directly from refrigerated storage [15,1] on the others found this to probably be the worst possible course of action. [16,17] On the other hand, the warming of composites to body temperature, or somewhat higher, immediately before placement has been shown to improve composite properties and reduce curing time (18,19). Insertion temperature has been found to have an influence on the hardness of composite. The Vickers hardness of restorations inserted with the composite resin pre-warmed to 3 degrees warmer than body temperature (40ºC) was found to be significantly harder than the composite resin inserted at room temperature. [20] Moreover, Trujillo et al., [21] found an elevated temperature of composite resin during photo polymerization resulted in substantially higher immediate and final conversion values of all composite resin materials tested in their study along with a concomitant improvement in fracture resistance and a 50% or more reduction in curing time. Holmes et al., [22] also found the film thickness of microhybrid composite resin decreased by approximately 30% when the material was heated to 54ºC. Thus, it is possible to use a highly filled pre-warmed packable hybrid composite at gingival margins in a deep restoration while eliminating poor marginal adaptation.

The aims of this study were to evaluate the effect of different composite resin temperatures (refrigerated to 5ºC, room temperature at 25ºC, and body temperature at 37ºC) on the degree of conversion and the microhardness of the composite resin.

MATERIALS AND METHODS

The material used in this study was (Ceram X shade D2 Dentsply/Caulk, USA) restorative composite resin. A total of 45 disc-shaped specimens were prepared from composite resin by the use of disc shape (stainless steel) mold which was 2 mm in thickness and 8mm in diameter as shown in Figure (1), 15 specimens for each different temperatures as following:

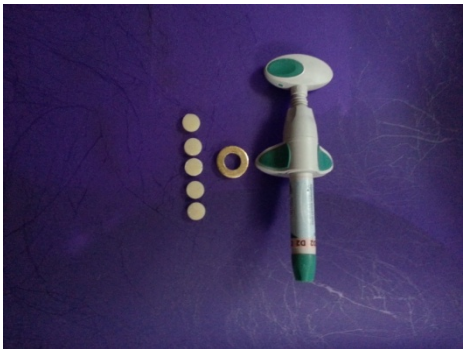

Figure (1) : Disc-shaped specimens.

Group1: Specimens prepared by the use of refrigerated tube of composite resin at 5ºC for 24h. before preparing the samples.
Group 2: Specimens prepared from the composite resin tube which was stored in an incubator at room temperature (25ºC) for 24h.
Group3: Specimens prepared from the composite tube which was stored in an incubator at 37ºC for 24h. .

The specimens were prepared by insertion of the composite resin inside the (stainless steel) mold and cover the materials by transparent matrix dental strip and two glass slide. A light pressure was applied over the mold to expel excess material. A total of(15) samples were tested for each temperature(10) samples for testing the degree of conversion and 5 samples were used for the microhardness test. The (10) samples divided into 5 samples cured and(5) samples uncured. After that the cured samples were polymerized with a light curing device (QTH,Astralis, VIVADENT, Austria) for 40sec from top and bottom surfaces of the samples according to manufacturer's instructions with the light quid held 1mm from the top surface of the specimen. All specimens were then immersed in distilled water for 24h. in dark incubator (Fisher scientific-isotemp/incubator/USA.)at37ºC. After 24h the samples were removed from the incubator ,the samples used to determine the degree of conversion were separately crushed and grinned manually by piston and mortar into a powder. After that the powder was mixed with potassium bromide at a weight percentage of 1:5. After mixing ,the resultant powder was poured into a metal mold and compressed into a disc shaped by pressure device (Burker–TENSOR, Germany) press at a load of 10 tons, and the samples become ready for measurement (Figure 2).

Figure (2) Pressure device (press) (Bruker -TENSOR, Germany).

The uncured control samples were prepared by devolving the uncured composite materials with the carbon Quattro chloride (CCL4) (organic dissolvent), and placed on a special cell supplied by the manufacturers of the Fourier transform infrared spectroscopy (FTIR) (Bruker 27, TENSOR, Germany),and become ready for measurements. [23]

Measurements of Degree of Conversion :

The degree of conversion of the samples were measured by Fourier transform infrared spectroscopy (FTIR) (Figure3).The degree of conversion on the tested samples was calculated by the two frequency techniques using the net peak transmittance areas of C=C stretching vibration at 1638 cm-1 as analytic frequency and the aromatic C=C stretching vibration at 1608 cm-1 as reference frequency (Figure 4 A, B,C) .

Figure (3) Fourier transform infrared spectroscopy (FTIR) (Bruker27,TENSOR, Germany)

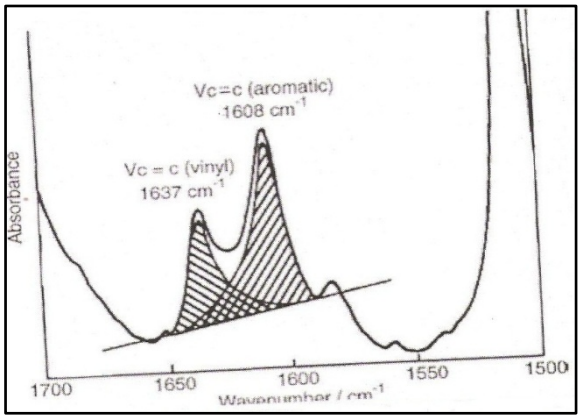

Figure (4 A) Typical IR spectrum obtained . The band at 1637 cm-1 was assigned C=C vibration ,and 1608 cm-1 to the vibration of aromatic rings.

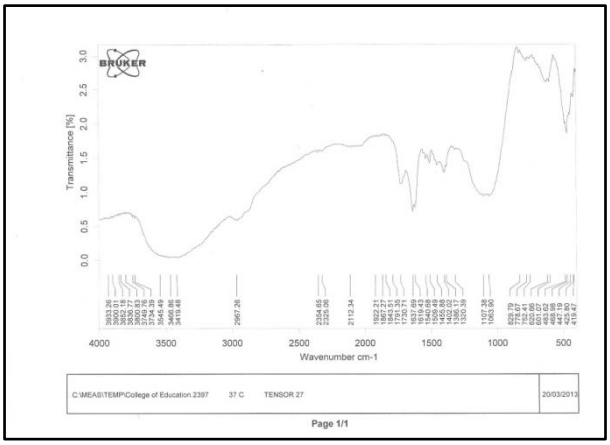

Figure (4 B) Spectrometer Chart of Prepared Composite Resin Sample with Aliphatic and Aromatic Peaks

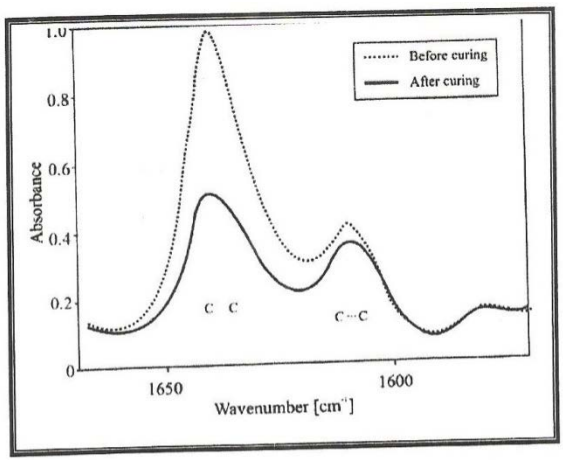

Figure (4 C) Stretching band of Aliphatic and Aromatic C=C

The degree of conversion was calculated according to the formula :

DC % = [1 – { (aliphatic C=C)/ (aromatic C=C) } of cured composite / { (aliphatic C=C)/(aromatic C=C) } of uncured composite] * 100 [(23)]. One way analysis of variance (ANOVA test) used to show the effect of temperature on the degree of conversion of the tested samples.

Measurement of Micro Hardness :

The micro hardness of the top and bottom surfaces of the specimens was determined by the use of (Baujahr Micromet 11 Germany) digital microhardness tester with a Vickers diamond indenter attached as shown in (Figure 5). A load of 300g was applied to the surface of the specimens for 12 seconds. Three indentations equally placed over a circle and not closer than 1mm to either adjacent indentations or to the margin of the specimens were made on the surface of each specimen. A statistical analysis using a one-way analysis of variance (ANOVA) was used to determined any significant difference in the hardness values of the composite resin at different temperature.

Figure (5): (WOLPERT – WERKE –GMBH Germany).

RESULTS

One-way (ANOVA) for the effect of pre-cured temperature on the D. of conversion of the composite resin (Table1) represent a statistically significant differences among group. Duncan multiple ray test (Table2) show that group(3)(63.86) revealed the higher D.of conversion then followed by group (2) (54.96) , group (1) (52.32) respectively this represent that the higher pre-cured temp. the higher the degree of conversion of composite resin.

In(Table3) represent the means and the standard deviations of V.H. values at different. pre-cured temp. One-way (ANOVA) for the effect of pre-cured temp. on the V.H. of the composite resin. (Table 4) represent a statistically significant differences among groups at (p=0.05) value. Duncan test (Table5) show that group(3)(83.24) show higher value of V.H. but with no statistically significant difference with group (2) (82.28). While group(1) (67.24) show the least value, this mean that V.H. value increased as the pre-cured temp. of composite resin was increased.

Table (1) One way ANOVA showing effect of temperature on the degree of conversion of the composite resin.

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	365.585	2	182.793	397.952	.000
Within Groups	5.512	12	.459		
Total	371.097	14			

Table (2) Duncan test showing the significant effect of temperature on the degree of conversion of the composite resin.

		Subset for alpha = 0.05		
Temperature °C	**N**	**1**	**2**	**3**
5	5	52.3200		
25	5		54.9600	
37	5			63.8600
Sig.		1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.
a. Uses Harmonic Mean Sample Size = 5.000.

Table (3) Mean Vickers hardness values (VHK) and standard deviations (SD) of composite resin specimens at different temperatures .

Temperture	Mean of TopVHN (SD)	Mean of Bottom VHN(SD)	*Pvalue*
Refrigerated to 5˚C	67.4 (4.4)	58.6 (1.4)	0.003
Room temperature to25˚C	82.2 (1.6)	73.4 (1.1)	0.011
Preheated to 37˚C	83.0 (1.6)	80.5 (2.8)	0.124

Table (4) One way ANOVA showing effect of temperature on the Microhardness of the composite resin.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	805.205	2	402.603	55.081	.000
Within Groups	87.712	12	7.309		
Total	892.917	14			

Table (5) Duncan test showing the significant effect of temperature on the Microhardness of the composite resin.

		Subset for alpha = 0.05	
Temperature °C	**N**	**1**	**2**
5	5	67.2400	
25	5		82.2800
37	5		83.2400
Sig.		1.000	.585

Means for groups in homogeneous subsets are displayed.
a. Uses Harmonic Mean Sample Size = 5.000.

DISCUSSION

Studies showed the advantage of preheating the composite for increasing the monomer conversion.[24] Resin polymerization occurs rapidly within the first 2s after the LCU is turned on and the ability to make three measurements a second using real time FT-IR is an improvement on previous reports that were based on one scan per second.[1,12] The first hypothesis was proven to be correct as the degree of conversion, rate of polymerization and knoop microhardnes at the bottom of the sample were all significantly increased by starting to the light cure the resin at simulated intra-oral temperatures compared to room temperature. The volume of resin contained within the 2-mm high, 8-mm diameter ring was similar to the volume of resin in a large restoration in a molar tooth. The overall polymerization rate is controlled by a competition between propagation (which increases the rate) and termination (which decrease the rate), and the rate of reaction decreases after maximum rate of polymerization as the monomer is consumed.[25,26]

Auto-acceleration of the rate due to a reduction of the constant termination occur due to the Trommsdrof effect .The increase in the viscosity as the polymer network is formed results in drastically reduced diffusion and mobility of large polymer radicals. This effectively reduces the termination rate constant at any given temperature and shortens the time at which the maximum rate of polymerization.[8] The second hypothesis that there would be a positive linear correlation between the degree of conversion and knoopmicr hardness measurements recorded in the same region of the specimens was also proven (Figure 6) shows that, within the rang of degree of conversion and knoopmicrohardness values recorded in this study, there was an excellent positive linear correlation between the degree of conversion and knoop micro hardness. This supports previous studies that have also shown a good correlation between the degree of conversion and the knoopmicrohardness of dental resins [5,7] and supports the use of microhardness as a means to evaluate the polymerization of dental resins. [6]

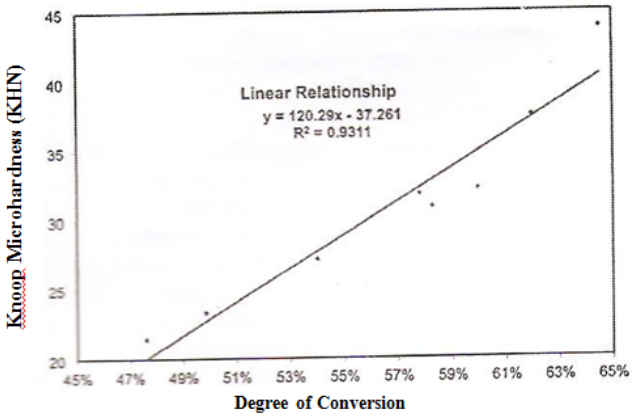

Figure (6) Regression analysis between mean degree of conversion and mean knoop Microhardness of the specimens obtained at each temperature

The extent of conversion affects both the physical and mechanical properties of the polymer, both of which depend on the polymer network formation. [26,27] Hence the degree of conversion of the refrigerated and preheated composite was evaluated in this study. The degree of conversion of samples at 5ºC was found to be (52.3) while composite at room temperature 25ºC was (54.96) where as the composite at 37ºC was (63.86). The increased degree of conversion can be due to several reasons. The viscosity of the system increased with increase in temperature and this enhances radical mobility. The collision frequency of unreacted active groups and radicals also increases with elevated curing temperature when it is below the glass transition temperature. [24,26] In the clinical, when the composite is preheated, there are two factors to be considered: one is the temperature of the composite resin when it is placed in the oral cavity and the second factor is the time delay between

dispensing it from the syringe and placing it into the preparation, contouring, and light –curing it.

The methods to evaluated the degree of conversion include FTIR spectroscopy, physical determination of surface hardness, Raman spectroscopy, and photo- differentiated scanning calorimetry. FTIR spectroscopy was chosen for this study because of its accuracy.[24]

Hardness is the resistance of material to identation and it correlate well to the materials strength and rigidity [27,28] Vickers hardness testing was selected for this study because of its relative simplicity and favorable correlation to the degree of conversion using infrared spectroscopy [23,24]. Asmussen and Ferracane[20,27] found a correlation between the increase in surface hardness and an increase in the degree of conversion of resin materials .Materials with low surface hardness contain a high amount of unreacted methacrylate group. The best surface hardness (top and bottom) in this study were obtained using pre-warmed composite resin at 37°C. On the other hand, the lowest surface hardness (top and bottom) resulted from using refrigerated composite resin. Freedman et al.,[28] stated the use of pre-warmed composite resin will enhance the physical properties of the final restoration, reduce polymerization time and increase the degree of conversion. A short irradiation combined with a high, uniform conversion and low stress is desirable in clinical practice.

Practitioners are faced with the dilemma of improving the degree of improving the degree of conversion worsens marginal continuity. It must be emphasized energy is attenuated in deeper areas of restoration, thus full conversion can be at risk [25]. Therefore, the use of pre-warmed composite resin might help improve conversion, especially at the deeper areas of restoration, which improves its physical properties and reduces its film thickness. Thus, it is possible to use pre-warmed hybrid, or packable composite, at the gingival margin to achieve better marginal adaptation and to attain better and faster conversion in areas where isolation control is not optimal .

Finally, elevating composite resin temperature during polymerization will reduce curing time, consequently, saving chair time . A concern might arise regarding the effect of using high temperature composite on the dental pulp causing iatrogenic damage. However the maximum intra pulpal temperature rise from application of composite resin heated to 57.2°C was approximately 1.6°C which is well within the pulp tolerance of more than 10°C [28].

CONCLUSIONS

Under the condition of this in vitro study, when the initial temperature of the resin was increased from (5,to25,37)°C, the degree of conversion and knoop microhardness values recorded in this study were increased. This may be revealed that there was a positive linear correlation between the degree of conversion and the knoopmicrohardness and the degree of pre-cured resin temp. and between the degree of conversion and miccrohardness at the same temp. and this support the use of knoop microhardness as a useful mesurement parameter.

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