EGYPTIAN DENTAL JOURNAL

Vol. 64, 2717:2735, July, 2018

I.S.S.N 0070-9484

Fixed Prosthodontics, Dental materials, Conservative Dentistry and Endodontics

www.eda-egypt.org • Codex : 160/1807

INFLUENCE OF PRE-CURING WARMING ON MICRO-LEAKAGE, MICRO-HARDNESS AND DEGREE OF CONVERSION OF HIGH VISCOSITY BULK FILL COMPOSITE RESINS: AN IN VITRO STUDY.

Eman Mohamed Sobhy Elbahrawy* and Reham Mohamed Attia**

ABSTRACT

Objective: This study aimed to investigate the Influence of preheating on micro-leakage, micro-hardness and degree of conversion of high viscosity bulk fill and conventional nanohybrid composite resins

Materials and methods: For micro-leakage assessment, thirty extracted human molars were used. Sixty class II cavities were prepared in the mesial and distal surfaces of teeth, and were randomly divided into two groups according to type of composite resin **Group I** : nanohybrid high viscosity bulk fill composite resins (Tetric Evoceram bulk fill, Ivoclar Vivadent), and **Group II**: conventional nanohybrid composite resins (Tetric Evoceram,Ivoclar Vivadent). Each group was further divided into three subgroups according to the precure temperature of composite (subgroup A: 24 °C (control subgroups), subgroup B: °37C, and subgroup C: °68C). where subgroups (IA, IB and IC received high viscosity bulk fill nanohybrid composite resins), and subgroups (IIA, IIB and IIC received conventional nanohybrid composite). Micro-leakage was determined by dye penetration test with scoring criteria from 4-0. Sixty composite discs were used in Vickers micro-hardness test (VHN), and degree of conversion with the same experimental grouping as used in micro-leakage assessment .VHN was determined on top and bottom surfaces of the specimens using Vickers microhardnss indenter and monomer conversion was determined using Fourier transform infrared spectroscopy. Data were collected, tabulated and statistically analyzed by using ANOVA F-test, Mann-Whitney testand Wilcoxon Signed rank test ($p<0.05$).

Results: Concerning micro-leakage; There was no significant difference between the three subgroups for each type of the tested material. Concerning micro-hardness; results proved a significant difference in the top and bottom VHN among the two groups with the highest mean top and bottom VHN value observed in preheating both types of composite to °68C and the lowest mean top and bottom VHN value seen in control subgroups (composite resin stored at room temperature). Both composites attained bottom / top % ranges between 90-80 %. The degree of conversion of Tetric EvoCeram Bulk Fill was significantly increased ($p = 0.006$)with composite pre-heating, but no effect on monomer conversion of the other investigated material was observed. Tetric EvoCeram Bulk Fill achieved the significantly highest Vickers micro-hardness and monomer conversion, irrespective of the precuring temperature..

*Lecturer of Dental Biomaterials, Department of Dental Biomaterials, Faculty of Dentistry, Tanta University, Tanta, Egypt

** Lecturer of Operative Dentistry, Department of Conservative Dentistry, Zagazig University, Zagazig ,Egypt.

Conclusions: Both the composite material and the pre-cure temperature affect Vickers micro-hardness and degree of conversion. Pre-heating of composites prior to photo activation significantly increased the Vickers micro-hardness and degree of conversion but have no effect on micro-leakage.

KEY WORDS: preheating, Calset, bulk fill composite resins, micro-leakage, Vickers micro-hardness, monomer conversion.

INTRODUCTION

Today, composite resin has been considered the most popular and the best available esthetic restorative material being used. This has been facilitated by remarkable advances in their physical properties and improvements in their manipulative qualities and durability[1, 2].

In spite of enhancement in composite resin materials, some disadvantages still affect the durability and longevity of restorations of resin composite. The most commonly stated limitations are related to polymerization shrinkage and its stresses, low abrasion and wear resistance ,mismatch in thermal expansion, marginal leakage, toxicity and recurrence of decay[3].

To conquer these issues, challenges have been made to increase the filler content. However, this modification ends up in in higher paste viscosity and tougher extrusion of these highly filled materials from the delivery device[4].

One of the first clinical considerations is adaptation (both internal and marginal) and the resulting interfacial seal of composite resin to the preparation walls. In contrast to amalgam, highly filled composite resins can't be ''condensed'' by employing a heavier force to decrease porosity or to improve adaptation. Moreover, several up to date composite resins are also sticky and difficult to manipulate, leading to greater problems in placement [5].

A new brand of resin-based composite materials, so-called high viscosity 'bulk-fill' resin composites are introduced to the market recently, to simplify and accelerate the process of restoration. these materials can be adequately photo-polymerized in thick layers up to four or perhaps five millimeters, skipping the time needed for layering technique [6-9].

The bulk fill composite material used to restore large and deep cavities which could be restored simply and faster using these materials [10-12]. However, this modification results in higher paste viscosity which makes insertion and adaptation of the material to the cavity preparation walls troublesome [13].

Poor adhesion between restorative material and dentin causes marginal gap formation, which prompts micro-leakage. This is where oral microorganisms, liquids, and compound substances diffuse through the interface between tooth structure and restorative material. Fluids advancing through the micro-leakage area through dentin into the pulp result in post operative pain, recurrent caries, pulpal hyperemia, and restoration failure[14].

Numerous techniques for placing restorations have been advocated to improve the marginal integrity and seal of resin composite restorations, as incremental layering to reduce the C- factor [15] pulsed-curing and soft-start methods to modify the reaction rate and use of flowable composites prior to placement of the heavier filled material [16,17].

To achieve better marginal integrity; flowable composites, with their apparent fluidity, have been frequently recommended as stress absorbers and adaptation promoters [18-19]. However, attributable to the lower filler content of flowable composite materials, larger polymerization stress is anticipated

relative to standard resin composites , polymerization shrinkage, as well as demonstrate higher values of expansion and contraction with temperature change than do the higher filled materials [20,21]. This shrinkage results in stresses that can lead to de-bonding from de-mineralized tooth structure and result in increased micro-leakage, possibly resulting in restoration failure [18].

Precure warming of high-viscosity bulk-fill composites maybe an interesting approach to provide a transient viscosity reduction comparable to that of a flowable composite while not sacrificing the advantages of superior mechanical properties related to highly filled resin composites [22-26].

A device named Calset warmer (Calset TM AdDent, Inc. Danbury, CT USA) has been introduced to the market to warm composite resin before placement in the cavity and it absolutely was claimed that preheating the composite resin is also suggested to improve the physical and mechanical properties [27-30].

Micro-hardness has usually been used to evaluate the mechanical properties of restorative materials, and this property correlates well with the degree of conversion of resin composites [31-32]. Many factors influence the hardness of composite resin materials like organic matrix composition, type of the filler particles and degree of conversion [33]. There is a positive correlation demonstrated between increasing hardness and increasing degree of conversion. The Vickers microhardness test (VHN) has been widely used to evaluate the hardness of dental materials, as it is usually used for brittle materials and small film thickness materials [34-36]

The extent of polymerization of dental composite resins ranges between fifty and seventy five percent conversion, which has a direct effect on the physical and mechanical properties and hence the longevity of the restoration [32]. Incomplete polymerization leads to presence of unreacted monomer within the restoration that may leach into saliva resulting into increased diffusion of oral fluids. Both oral fluids and unreacted monomer act as plasticizers, reducing mechanical strength, dimensional stability and allow bacterial growth due to the ingress of oral fluids. Unreacted monomers can also cause allergic and sensitivity reactions [33].

Based on these considerations, the aim of our study was to evaluate the influence of composite pre-heating on micro-leakage, micro-hardness and monomer conversion of high-viscosity bulk-fill materials and a conventional nano-hybrid resin composite under the null hypothesis that pre-heating does not affect micro-leakage, micro-hardness and double bond conversion of the composite materials

MATERIALS AND METHODS:

I- Materials:

The materials used in the study are listed in Table 1 and shown in Figure 1

TABLE (1) The materials classification, composition & manufacturer.

Materials	classification	Resin matrix	Filler Type	Filler Loading	shade	Manufacturer
1-**TetricN-ceram Bulk-fill Composite:**	**Bulk-fill** [High –Viscosity Nano-Hybrid composite	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Bariumglass, Ytterbium trifluoride, oxides and prepolymer	81% by wt., 61% byvol	IVA	Ivoclar Vivadent AG,Schaan, Liechtenstein
2-TetricN-ceram Composite:	Universal Nano-Hybrid composite			76% by wt., 55% by vol	A2	

II-METHODS:

Micro-leakage assessment:

Micro-leakage assessment was made on thirty sound molars free of caries and restorations extracted due to periodontal causes. Molars were collected from the out-patient clinic of the Oral Surgery Department, Faculty of Dentistry Tanta University. All calculus deposits were removed with hand scaler. The teeth were cleaned and polished with pumice slurry and water using low speed hand piece, then rinsed thoroughly with tap water. The teeth were stored in 10 % formalin as disinfectant and preserving biologic specimens at 4°C to minimize bacterial contamination for 5 days, and then stored in distilled water at 23±1°C until they were required for cavity preparation [37-39].

Two Class II cavities were prepared in each tooth (mesial and distal) with a high-speed hand piece using water coolant and a #256 carbide bur (Komet H21314008 Lot 980042 Lemgo, Germanay). The actual width of each preparation was defined by the anatomy of each tooth. Pulpal floor was approximately 2 mm deep, and the proximal boxes were approximately 3 mm wide, 4 mm long and 1.5 mm deep. At least 1.5 mm of sound tooth structure remained occlusally between the two cavities. The amount of extension on the occlusal surface was typically about 3 mm [40, 41].

The cavities were grouped according to type of the material used as followed: **Group I**: 30 cavities were filled with Nano-Hybrid Bulk-fill composite resins [Tetric N-Ceram Bulk-fill]. **Group II**: 30 cavities were filled with Conventional Nano-Hybrid composite resins [Tetric N-Ceram]. Each group was subdivided into three subgroups according to precuring temperature as follow:

Subgroup A: 10composite specimens were stored at controlled room temperature at 24°C (room temperature composite) which was used as the control. **Subgroup B:** 10 composite specimens were preheated to temperature 37°C. **Subgroup C:** 10 composite specimens were preheated to temperature 68°C.

When testing at room temperature, the composite materials were allowed to stabilize to room temperature (24°C) for 24 hours [42], while Composite resins in the preheated subgroups were placed in a commercially available heating device (Calset TM Ad Dent, Inc. Danbury, CT USA). Which was preset to 37 or 68 °C (Figure 1).

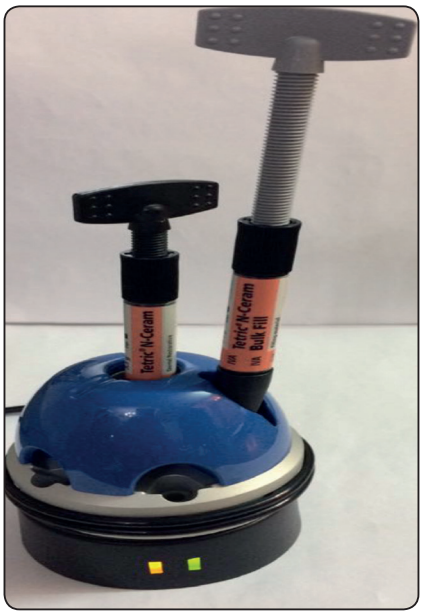

Fig. (1) Preheating of composite syringes in Calset device at (37-68°C)

A standardized volume of composite material was applied into the middle well of the heating device and maintained in situ for 5 min

For all subgroups, cavities were etched, bonded (TetricN-bond Self –etch, Ivoclar Vivadent ,AG,Schaan, Liechtenstein) and composite restorations were placed in the cavities and photopolymerized using LED curing unit (P11060012A LED P5 Guilin, Guangxi, Medical instrument CO., China) with a light intensity of 1200 mW/cm2, and with curing time of 20s according to the manufac-

turer's instructions. Composite restorations were finished and polished using diamond finishing bur (T&F hybrid points kit, Shofuinc, Japan) then polished with rubber points (KendaDental Polishers, Liechtenstein)[43].

Teeth were stored in distilled water for 24 hours untill thermo-cycling. All samples were thermo-cycled for 500 cycles between 5°C and 55°C with a dwell time of 1 minute to simulate changes in temperature in the oral cavity using thermal cycling machine (Nova, Konya, Turkey). Before immersion in dye for micro-leakage assessment by dye penetration test; the apices of the teeth were sealed with sticky wax and the whole surface was coated with a nail polish 1 mm away from the restoration margins in order to reduce other leakage elsewhere that could lead to false positive results. The teeth were embedded in split copper cylindrical molds to make acrylic blocks where teeth were immersed in to facilitate the handling of the specimens, where teeth emerged 2mm above the cemento-enamel junction. Then molars were immersed in 0.5% methylene blue solution for 24 hours at room temperature. The blocks were immersed upside down where the restorations were immersed in dye to decrease leakage other than the tooth restoration interface [44].

To measure the extent of micro-leakage; teeth were sectioned in a mesio-distal direction through the restoration using IsoMet cutting machine (Isomet Low speed saw, Buehler, Lake Bluff, USA.) [20, 43].

The sectioned teeth were evaluated with a stereomicroscope (Olympus model no.SZ11. Japan) at (40x) magnification. The degree of cervical margin micro-leakage was determined through the extent of dye penetration and was scored according to scoring criteria (0 to 4) as follows: 0= No dye penetration [no micro-leakage), 1 = superficial Dye penetration not beyond the amelodentinal junction (ADJ), 2 = Dye penetration beyond the ADJ but not including the cervico-axial line angle, 3 = Dye penetration along the axial wall, 4=penetration into the pulp chamber [41-44] (Figure 2).

Micro-hardness test

Micro-hardness test was carried on sixty specimens of composite resin discs (6mm in diameter and 2mm in thickness.). The composite discs were grouped as mentioned previously in micro-leakage assessment. Composite discs were prepared using split Teflon molds with dimensions 6mm in diameter and 2mm in height. The resin composite was packed in the split Teflon mold using Teflon coated plastic filling instrument. A polyester strip was placed over the material and a glass slab was placed over the mold to obtain a flat surface. The glass slab was then removed and the light cure was placed directly onto the polyester strip touching it for 20 seconds according to manufacturer instructions. After the photo-activation, the samples were removed from the Teflon molds and their surface was polished with # 150, # 400, # 600, # 1200, and # 2000 grit water-proof abrasive papers. Then, they were immersed in distilled water at 37˚C, and placed into a dark container for 24 hours. [45,46].

The VHN analysis was performed on both top and bottom surfaces by means of micro-hardness tester (ZwicRoell, west Midlands, England) using a 100 gm load with a dwell time of 15 seconds [3,4]. For each side, three points were taken on both top and bottom of the composite resin specimens and the three indentations made by the square based diamond indenter of angle 136, the mean value was calculated for each top and bottom of each specimen and then bottom / top % was calculated. VHN was calculated by the following equation [47]:

VHN: HV=1.854 P/d2

Where, HV was Vickers hardness in Kgf/mm2, P was the load applied in Kgf and d was the length of the diagonals in mm and 1.854 was a constant number.

Degree of conversion measurements

Degree of conversion was measured using Fourier transform infrared spectrometer [Spectrum GX; PerkinElmer, Beaconsfield, UK). Ten specimens were prepared for each experimental subgroup. Following micro-hardness test [47] thin chips of composite discs were scrapped with a scalpel and pulverized into a fine powder. The composite powder was mixed with potassium bromide (KBr; Merck, Darmstadt, Germany) and pressed into a thin pellet (10-mm diameter)using a hydraulic press (Specac, Orpington, Kent, UK) with a load of 2.5 tons. Composite-KBr pellets were also prepared from the uncured material. The pellets were placed into a holder attachment in the optical compartment of the spectrometer for analysis. Infrared spectra were recorded in transmission mode in the 4000–400 cm^{-1} wave number range, and then converted into the absorbance mode. A total of 20 scans per specimen were measured at a resolution of 4 cm^{-1} . Using a standard baseline technique [48], the absorbance intensities (AI; peak heights) of the aliphatic C=C stretching vibrations at 1638 cm^{-1} and aromatic C . . . C stretching vibrations (internal standard) at 1610 cm^{-1} were determined for both the cured and uncured composites, and the degree of conversion (DC) was calculated according to the following equation [49] :

$$DC\ (\%) = \left[1 - \frac{[AI(1638\ cm^{-1})/AI(1610\ cm^{-1})]_{cured}}{[AI(1638\ cm^{-1})/AI(1610\ cm^{-1})]_{uncured}}\right] \times 100$$

Statistical analysis

To analyze the effect of precure temperature on resin composite micro-leakage, micro-hardness and degree of conversion statistical tests were performed. Statistical analysis of the data were achieved, where data were fed to the computer using IBM SPSS software package version 20.0., Qualitative data (micro-leakage assessment) were described using number and percent. Mann-Whitney test was used to compare between different tested groups for cervical microleakage at different precure temperature. Wilcoxon Signed rank test used to compare between different procure temperature for each material for cervical microleakage. The significance level was set at $P \leq 0.05$

The distributions of quantitative variables (micro-hardness and degree of conversion assessment) were tested for normality using Shapiro-Wilk test and D'Agstino test.

Quantitative data were described using mean and standard deviation for normally distributed data. Comparison among more than two populations were analyzed by F-test (ANOVA) and Post Hoc test (Scheffe). Significance test results were quoted as two-tailed probabilities. Significance of the obtained results was judged at the 5% level.

RESULTS

The results of the micro-leakage assessment are displayed in Figure. (2&3) and table (2& 3). The micro-leakage scores at cervical margins at the tooth and restoration interface revealed the following:

At 40X magnification, none of the materials used at the three different temperatures completely prevented micro-leakage. The restoration and tooth interface for all subgroups exhibited varying amount of micro-leakage along the entire interface of the restoration; figures (2 & 3).

Using Mann-Whitney test: Intergroup comparison between the two investigated group of the study as regarding micro-leakage scoring criteria at the cervical margins at the three different temperatures discovered no significant difference between the two materials applied at the three different temperatures ($p > 0.05$).

Using Wilcoxon test; Intra-group comparison between micro-leakage scoring criteria at the cervical margins in the six subgroups of the study at the three different temperatures revealed no significant difference between the three temperatures and the both types of resin composite restorative materials at the cervical levels ($p > 0.05$).

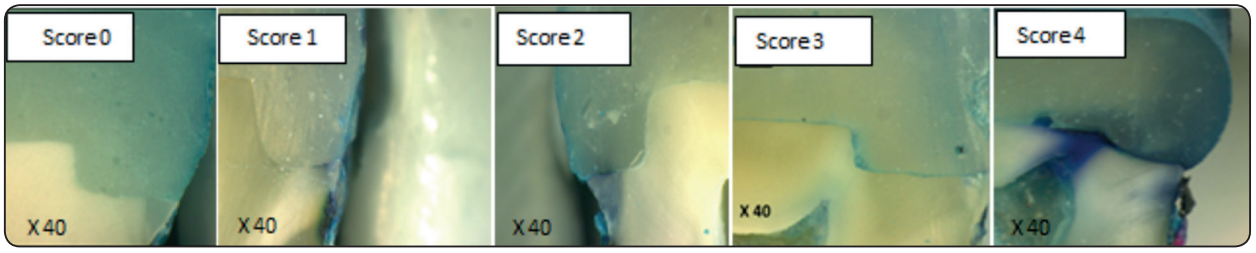

Fig. (2) Shows representative stereomicroscopic imagesof the samples with cervical micro-leakage at 40X magnification.

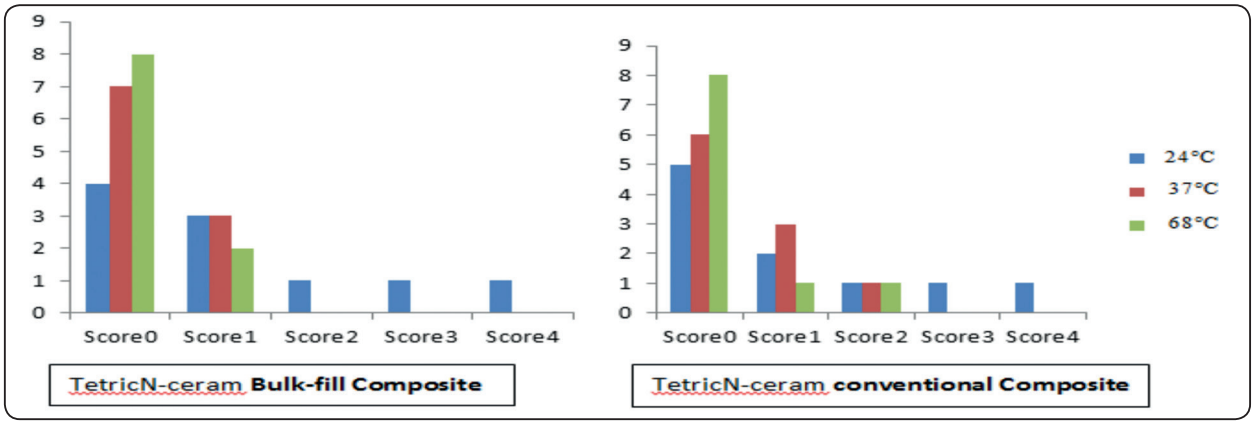

Fig. (3) Shows cervicalmicro-leakage scoring criteriain the two groups of the study at the three different temperatures

TABLE (2) Intergroup comparison between the two investigated materialsas regarding micro-leakage scoring criteria at the cervical margins at the three different temperatures

	TetricN-ceram**Bulk-fill Composite**					TetricN-ceram**conventional Composite**					**Mann-Whitney**	**P.Value**
24ºC	**Score0**	**Score1**	**Score2**	**Score3**	**Score4**	**Score0**	**Score1**	**Score2**	**Score3**	**Score4**		
	4 (40%)	**3 (30%)**	**1 (10%)**	**1 (10%)**	**1 (10%)**	**5 (50%)**	**2 (20%)**	**1 (10%)**	**1 (10%)**	**1 (10%)**	**0.00**	**1**
37 ºC	**7 (70%)**	**3 (30%)**	**0 (0%)**	**0 (0%)**	**0 (0%)**	**6 (60%)**	**3 (30%)**	**1 (10%)**	**0 (0%)**	**0 (0%)**	**0.224**	**.823**
68 ºC	**8 (80%)**	**2 (20%)**	**0 (0%)**	**0 (0%)**	**0 (0%)**	**8 (80%)**	**1 (10%)**	**1 (10%)**	**0 (0%)**	**0 (0%)**	**0.224**	**.823**

TABLE (3) Intragroup comparison between micro-leakage scoring criteria at the cervical margins in the six subgroups of the study at the three different temperatures

		Wilcoxon	P - value
TetricN-ceram**Bulk-fill Composite**	**24 ºCvs37 ºC**	23.500	0.390
	24 ºCvs 68 ºC	23.000	0.335
	37 ºCvs 68 ºC	27.500	1.000
TetricN-ceram**Composite**	**24 ºCvs37 ºC**	25.500	0.665
	24 ºCvs 68 ºC	23.000	0.335
	37 ºCvs 68 ºC	27.000	0.913

The dye penetration scores from (0-4) at the cervical margins where subgroups IC (68 ºC preheated TetricN-ceram Bulk-fill Composite) and IIC (68 ºC preheated TetricN-ceram conventional Composite) showed the least micro-leakage in 80% of cases while the highest micro-leakage was found in the control subgroups of both tested materials (room temperature composite)

Concerning top micro-hardness, as shown in table 4; results revealed that subgroup C in both groups(preheating at 68°C) obtained the highest mean top VHN where mean values of subgroups IC and IIC were 66.2340±3.15958and 59.2920±5.84295 respectively. This was followed by subgroup B (preheating at 37°) where mean values of subgroups IB and IIB were 65.3600±1.09417 and 51.4940±1.75499respectively. The lowest mean top VHN was recorded in subgroup A (control or room temperature storage) where mean values of subgroup IA and IIA were 57.7500±.86626and 47.5000± 1.25897respectively.

Anova F-test 44.548 was significant in the top VHN among the two groups (p=0.01 in group I & p= 0.015in group II). Post Hoc test (Scheffe) for pair wise comparison between subgroups showed that subgroupsIC and IB had significantly higher mean top VHN than subgroup IA but there was non-significant difference in mean top VHN between subgroups IC and IB. Also significant difference between subgroupsIIA and IIC and between subgroups IIB and IIC was found, while there was non-significant difference between subgroups IIA and IIB (Table 4).

Concerning bottom micro-hardness; as shown in table 4results showed that the highest mean bottom VHN value was observed in preheating both types of composite to 68 °C where the highest mean values of subgroups IC and IIC were 59.8780±3.13361and 50.4680±1.97195respectively. This was followed by subgroup IB (Bulk fill composite preheated to 37°C), subgroup IIB (conventional composite preheated to 37°C) where mean values were 57.6000 ± 1.37417and 45.4340±2.05669respectively. The lowest mean bottom VHN was recorded in both types of composite stored at room temperature(control)where mean values were 47.4100±.88591 and 38.7840±1.31977 insubgroup IC (Bulk fill composite stored at room temperature) and subgroup IIC (conventional composite stored at room temperature) respectively.

ANOVA F-test 64.575 was significant (p=0.0001) in the bottom VHN among the three subgroups. Post Hoc test (Scheffe) for pair wise comparison between subgroups showed significant relation in group I (Bulk fill composite)between subgroups except for subgroups IB and IC where there was no significant difference in-between(there was no significant difference between 37°C and 68°C preheating temperature). While in group II (conventional composite) there was significant difference between all subgroups.

Concerning Bottom/Top %:

As shown in table 4, results discovered that subgroup IIB and IC has the highest mean Bottom/Top %: where their mean values were 90.7600±0.77653 and 90.4000±1.81659respectively. This was followed by subgroup IIC and IB where their mean values were 90.0400±1.20333 and 87.8600±1.19499 respectively. The lowest mean Bottom/Top % was recorded in subgroup IA& IIA (control- room temperature) where their mean values were 82.1020±.1.40090 and 81.6400± 0.91815 respectively.

Anova F-test 44.548 was significant in Bottom/Top %among the two groups (p = 0.01 in group I & group II). Post Hoc test (Scheffe) for pair wise comparison between subgroups showed that subgroups IC and IB had significantly higher mean Bottom/Top %than subgroup IA. Also significant difference between subgroups IC and IB and between subgroups IIA and both IIB & IIC was found, while there was non significant difference between subgroups IIB and IIC (Table 4).

TABLE (4) Showing Comparison between the subgroups in both tested material regarding Mean top bottom VHN and Bottom/Top % at the three different temperatures.

Subgroup Mean VHN	**Subgroup IA** (TetricN-ceram-**Bulk-fill stored** at room temp.)	**Subgroup IB** (TetricN-ceram-**Bulk-fill** pre-heated to37°C)	**Subgroup IC** (TetricN-ceram-**Bulk-fill**preheat-ed to 68°C)	**Subgroup IIA** (TetricN-ceram**stored**at room temp.)	**Subgroup IIB** (TetricN-cer-ampreheated to 37°C)	**Subgroup IIC** (TetricN-cer-ampreheated to 68°C)
Top						
Mean ± S.D.	57.7500 ±.86626	65.3600 ±1.09417	66.2340 ±3.15958	47.5000 ±1.25897	51.4940 ±1.75499	59.2920 ±5.84295
ANOVA P		27.378 0.001*			13.904 0.015*	
P1		0.001*	0.001*			
P2			0.772			
P3					0.226	0.001*
P4						0.013*
Bottom						
Mean ± S.D.	47.4100 ±0.88591	57.6000 ±1.37417	59.8780 ±3.13361	38.7840 ±1.31977	45.4340 ±2.05669	50.4680 ±1.97195
ANOVA P		52.926 0.001*			52.250 0.001*	
P5		0.000*	0.000*			
P6			0.13			
P7					0.000*	0.000*
P8						0.02*
Bottom/Top %						
Mean ± S.D.	82.1020 ±1.40090	87.8600 ±1.19499	90.4000 ±1.81659	81.6400 ±0.91815	90.7600 ±0.77653	90.0400 ±1.20333
ANOVA P		40.529 0.001*			133.252 0.001*	
P9		0.000*	0.000*			
P10			0.048*			
P11					0.000*	0.000*
P12						0.498

Significance : * p ≤ 0.05

P 1 : Comparison between subgroup IA and both IB & IC regarding top VHN

P 2 : Comparison between subgroup IB and IC regarding top VHN

P 3 : Comparison between subgroup IIA and both IIB & IIC regarding top VHN

P 4 : Comparison between subgroupIIB and IIC regarding top VHN

P 5 : Comparison between subgroup IA and both IB & IC regarding bottom VHN

P 6 : Comparison between subgroup IB and IC regarding bottom VHN

P 7 : Comparison between subgroup IIA and both IIB & IIC regarding bottom VHN

P 8 : Comparison between subgroupIIB and IIC regarding bottom VHN

P 9 : Comparison between subgroup IA and both IB & IC regarding Bottom/Top %

P 10 : Comparison between subgroup IB and IC regarding Bottom/Top %

P 11:Comparison between subgroup IIA and both IIB & IIC regarding Bottom/Top %

P 12 : Comparison between subgroupIIB and IIC regarding Bottom/Top %

As regard to **top micro-hardness**, **bottom micro-hardness& Bottom/Top %:**

Table 5 shows that: at the three investigated temperatures TetricN-ceram Bulk-fill Composite showed significantly higher mean values than TetricN-ceram conventional Composite except for Bottom/Top % at 24ºC and 68ºC where the mean values of TetricN-ceram Bulk-fill Composite was non significantly higher mean values than that of TetricN-ceram conventional Composite ($p > 0.05$)

The results of the degree of conversion measurements are displayed in Table6 &7. One-way ANOVA revealed that the composite material ($p< 0.05$) and the interaction of pre-cure temperature and composite material ($p < 0.05$) significantly affected monomer conversion. Composite pre-heating significantly increased the degree of conversion of TetricEvoCeram Bulk Fill ($p < 0.05$), with no significant difference between preheating at 24°C &37°C but had no effect on monomer conversion of the other investigated material.Tetric EvoCeram Bulk Fill attained the significantly highest degree of conversion, irrespective of the pre-cure temperature.

TABLE (5) Showing Comparison betweenboth tested material regarding Mean top bottom VHN and Bottom/Top % at the three different temperatures

	Top VHN		**BottomVHN**		**Bottom/Top %**	
	TetricN-ceram **Bulk-fill Composite**	TetricN-ceram **conventional Composite**	TetricN-ceram **Bulk-fill Composite**	TetricN-ceram **conventional Composite**	TetricN-ceram **Bulk-fill Composite**	TetricN-ceram **conventional Composite**
24ºC MeanStd. Deviation	57.75 ± 0.866	47.65 ±1.401	**47.41 ±0.885**	**38.78 ±1.31**	**82.10 ±1.4**	**81.64 ±0.91**
T test	**13.359**		**12.135**		**0.617**	
P value	**0,001***		**0.001***		**0.555**	
54 ºC MeanStd. Deviation	**65.36** ±1.09	50.07 ±2.53	**57.6 ±1.37**	**45.4 ±2.05**	**87.86 ±1.19**	**90.76 ±0.77**
T test	**12.36**		**10.99**		**4.55**	
P value	**0.001***		**0.0001***		**0.02***	
68 ºC MeanStd. Deviation	66.23 ±3.15	56.04 ±1.69	**59.87 ±3.13**	**50.46 ±1.97**	**90.4 ±1.8**	**90.04 ±1.2**
T test	**6.356**		**5.683**		**0.369**	
P value	**0.01***		**0.01***		**0.721**	

Significance : * p ≤ 0.05

TABLE (6) Showing Comparison between the subgroups in both tested material regarding degree of conversion at the three different temperatures.

Subgroup Mean degree of conversion	**Subgroup IA** [(TetricN-ceram **Bulk-fill stored** at room temp.)	**Subgroup IB** (TetricN-ceram **Bulk-fill**pre-heated to 37°C)	**Subgroup IC** (TetricN-ceram **Bulk-fill**pre-heated to 68°C)	**Subgroup IIA** [(TetricN-ceram**stored**at room temp.)	**Subgroup IIB** (TetricN-cer-ampreheated to 37°C)	**Subgroup IIC** (TetricN-cer-ampreheated to 68°C)
Mean ± S.D.	56.9077 0.35856	60.5160 0.82099	65.7635 2.45759	50.6333 1.62583	51.9333 3.65120	52.5233 5.05348
ANOVA P	26.084 0.001*			0.203 0.822		
Pi		0.060	0.001*			
Pii			0.013*			

Significance : * p ≤ 0.05

P i : Comparison between subgroup IA and both IB & IC regarding degree of conversion

P ii : Comparison between subgroup IB and IC regarding regarding degree of conversion

TABLE (7) Showing Comparison between both tested material regarding degree of conversion at the three different temperatures

	TetricN-ceram**Bulk-fill Composite**	TetricN-ceram**conventional Composite**
24ºC Mean Std. Deviation	 56.9 ±0.35	 51.35 ±1.48
T test	**6.720**	
P value	**0.01***	
54 ºC Mean Std. Deviation	 **60.51** ±0.82	 **51.93** **±3.65**
T test	**3.972**	
P value	**0.017***	
68 ºC Mean Std. Deviation	 **65.76** **±2.45**	 **52.52** **±5.05**
T test	**4.081**	
P value	**0.028***	

Significance : * p ≤ 0.05

DISCUSSION

Improvements in resin composites throughout the last decade encouraged the use of composites more frequently for posterior restorations. However; many problems still hinder their use in large stress bearing areas. Efforts have been made to improve the clinical performance such as preheating resin composite. It was claimed to provide better handling characteristics which are similar to those of flowable composite [50-51].

Concerns have been raised regarding the ability of conventional nanohybrid composite and bulk–fill nanohybrid composite resins to make adequate adaptation to internal areas of the cavity walls and the cavo-surface margins. The high viscosity of such materials could increase the possibility of internal voids. To avoid these problems; some attempts have been suggested including the use of preheated resin composite [44].

Although the effect could be variable according to brand of the material; studies revealed greater flow of the preheated resin composite. Moreover, composites cured at elevated temperatures have shown increased rate of cure and a high degree of conversion. This could result in improved

mechanical properties. Whether preheating could improve the physical and mechanical properties or not of conventional nanohybrid composite (TetricN-ceram) and bulk–fill nanohybrid composite resins (TetricN-ceram Bulk-fill); it is a question needed to be studied and verified [52-54].

The high viscosity of heavily filled materials, as bulk–fill composite and conventional nanohybrid composite, may create a difficulty in producing a perfect marginal adaptation which may lead to void formation especially at the critical gingival margin [53].

Therefore, nanohybrid composite(bulk–fill and conventional types) was selected for the current study as it was suggested that preheating increased its flow properties, and hence improved its handling characteristics as reported by knight et al, in 2006 [55].

In this study, resin composite was preheated for the time recommended by the manufacturer. It was maintained in the device for five minutes so as to accomplish the most extreme preset temperature as Daronch et al, in 2006 suggested [56].

For this study, three temperatures were evaluated. 24°C was chosen because it represents the typical dental office temperature. 37°C was chosen because it was produced by the Calset device, and because it is close to the normal intraoral temperature of the mouth, and 68°C was chosen because it is the maximum temperature produced by the Calset unit [49].

To provide the higher maintained temperature, the heating unit was very close to the cavity restored or for the specimens prepared for hardness and degree of conversion testing to allow quick application and to allow minimum amount of heat to be dissipated during manipulation. This came in agreement with Daronch et al in 2006[56]. Many authors prompted the clinicians to work with the composite resin keeping in mind the end goal to guarantee the slightest temperature drop conceivable and accomplish the best clinical execution [57]. Several pilot studies were done to fix the time of resin composite application to be as quickly as possible (8-10 seconds). A previous invitro study showed that when a compule of composite resin was preheated, the definite pre-delivery composite temperature was less than the temperature chosen and expressed on the device of heating. Also, during the placement of preheated composite, the temperature of the composite dropped quickly upon syringe expulsion from the unit of heating until its placement on the prepared tooth [55].

In the present study, regarding micro-leakage assessment, the null hypothesis, stating that the difference is not significant between the samples of composite resin that have been preheated and those cured at room temperature with no preheating, should be validated.

Micro-leakage was investigated by the dye penetration test that permits seeing the extent of leakage occurring between the tooth restoration interface. Failure of the restoration to achieve an adequate seal may lead to marginal discoloration, unfavorable pulp reactions, recurrence of caries and post-operative hypersensitivity. In the present study, the samples were subjected to thermo-cycling in order to simulate the intraoral environment; all the test specimens were thermo-cycled for 500 cycles between temperature of 5°C and 55°C with a dwell time of 1 minute in the thermo-cycling machine. The teeth were sectioned longitudinally through the center of the restoration; therefore, the micro-leakage scores could be evaluated as two dimensional. This method was preferred in the current study because it was easier and less expensive than other techniques [32,57].

During the procedures of this study, the flowable behavior of preheated resin composite increased slightly at 37°C and markedly at 68°C. This was in agreement with Knight et al [55], in 2006, who found that the photo-activated composites showed better flowability when the temperature was elevated closer to the oral temperature. Freedman [58] in

2003 also stated that the viscoelastic materials as composites exhibit decreased viscosity when the temperature was increased, which affected the rheological properties of the material, and that was in agreement with our study.

In the present study, it was observed that least micro-leakage scoring was obtained in preheated Bulk-fill nanohybrid composites [TetricN-ceramBulk-fill) followed by preheated conventional nanohybrid composites [TetricN-ceram]. The same was observed in the control subgroup of both types of composite specimens that were stored in room temperature, but in overall groups there was no significant difference between the six subgroups. This indicated that the temperature change does not prevent micro-leakage but it may affect the extent of micro-leakage through the tooth restoration interface [59].

Although preheating could decrease the viscosity of the nanohybrid composites and might enhance composite adaptation to the internal walls, there was no significant difference between the micro-leakage scores of preheated and room temperature nanohybrid composite in the present study. This could be attributed to the possibility of contraction due to thermal changes that could have occurred when curing of composite was carried out either immediately or delayed at higher temperature. Higher temperatures could cause the material to go back more quickly to a preceding shape. This was accepted to happen in the current study because of the viscoelastic properties of the composite, which were responsible for pull away of the composite from the tooth preparation walls. There are two types of viscoelastic deformations which are deformation related to viscosity and retarded elastic deformation which come into play when placing the composites resin [44]. Viscous deformation is reliable for the most of the forming of the resin composite. The second type which is retarded elastic deformation happens at the same time as viscous deformation, and it is responsible at some point for shaping of the restoration of the composite. On the other hand, the retarded elastic deformation is characterized by temporary effect and the composite resin slowly tries to revert to its previous format. This means that composite resin material has a "memory". The retarded elastic deformation is not immediate, rather it happens gradually, depending upon various factors, including temperature [49,60-62]. Higher temperatures could make the material to try to return more quickly to a previous shape, which was demonstrated in the study made by Wagner et al [20] who demonstrated that delayed curing increased micro-leakage.

Cervical micro-leakage was chosen to be evaluated in this study because it was demonstrated by previous studies that micro-leakage is more cervical than occlusal. This could demonstrate that better sealed interfaces are found at the occlusal margins than at the cervical margins. This may be explained by the fact that the enamel amount at the occlusal margins is greater; which allows enhanced sealing and decreases the micro-leakage. Finally, the rheological properties related to the restoration can affect the easiness of insertion of the composite between the cervical and occlusal margins. Similar agreements were also reported byWagner et al [19], Arslan et al [28], Lohbauer et al [63] and Karaarslan et al [64].

Majority of the Class II carious lesions extend up to or beyond the CEJ and into deeper dentin along the proximal pulpal wall. Consequently, the cervical margins of composite resin restorations placed at cementum or dentine surfaces may result in a weaker marginal seal than at the enamel surface[19,63].

Micro-hardness of a composite resin is usually interrelated to the rigidity, physical strength and occlusal degradation resistance of the restoration in the environment of the oral cavity. Earlier studies have revealed that there was a direct correlation between hardness values and degree of monomer conversion [46].

Although bulk-fill composite materials allow the restoration build-up in thickness of 4–5 mm[11],

the thickness of specimen in the current study was 2 mm only to allow accurate and valuable comparison of degree of conversion and hardness of the bulk-composites and a conventional composite (TetricEvo Ceram). Unlike bulk-fill composites, TetricEvo Ceram is used only in layers of max. 2-mm thickness as recommended by the manufacturer, therefore TetricEvo Ceram could not polymerize appropriately at 4–5-mm thickness.

According to the results reported here, the null hypothesis, which states that the difference is not significant between mean post curing micro-hardness regarding the samples of resin composite that have been preheated and those cured at room temperature without preheating, should be rejected.

Tetric EvoCeram Bulk Fill showed higher hardness in comparison with conventional Tetric EvoCeram, although it has a similar resin composition as it is the bulk-fill complement. The recorded results might be due to the elevated filler content and complete cure of the bulk-fill materials due to the presence of the patented light activator Ivocerin which is responsible for ensuring the curing of the composite resin [11].

Regarding the two types of resin composites used in the current study; preheating led to increase the mean top VHN. Also, bottom VHN of the nanohybrid composite samples was the highest after preheating at 68°C. These findings were in agreement with previous studies done by Fróes-Salgado et al [33], Daronch et al [56], and Tatbirojn et al [35]. However, Saade et al .,[67] concluded that preheating of the resin composites had no effect on Vickers hardness values of microhybrid resin composite.

Preheating significantly increased the top and bottom VHN of both tested materials. The present study indicated that the micro-hardness achieved at the top surface of samples is greater than that the bottom VHN. This can be explained by the reduction of light (because of reflection, absorption and dispersion phenomena) as it passes through the composite. In the current study, measuring of micro-hardness at the top and bottom surfaces was done at 2 mm depth, which is the suggested increment thickness for placement of the composite. At a depth of 2 mm, light attenuation may reduce irradiance to approximately 75% of that reaching the top surface. It has been attributed that, on average, resin composites can achieve 50% to 70% conversion of monomers at room temperature [65-67].

To indicate an appropriate polymerization of the composite, the hardness values at the bottom should be between 80 and 90% of the hardness at the top surface as reported by previous studies. In the current study, the exposure duration recommended by the manufacturer led to bottom-to-top-surface micro-hardness ratios between 80 and 90% for the tested material [32,58,59].

In this study; micro-hardness of composite resin was found to be significantly affected by change in temperature where the preheated composite resin specimens at 68°C were significantly higher than room temperature specimens in top Vickers micro-hardness measurements. This comes in agreement with the results of Cohen et al [68], who concluded that the exposure of the specimens from a 5- to 20-fold longer time than that indicated by the manufacturer is needed to attain 70%- 80% bottom-surface hardness to the top. In addition, Osternack et al [69] indicated that using a longer curing time is needed to raise the energy density at the bottom of the layer and enhance the degree of conversion.

Degree of monomer conversion of composite resin is correlated to mechanical properties and biocompatibility of cured resin material [33]. Other studies showed that temperature plays an important role and has a significant effect on degree of conversion values of resin composites [27, 68]. In the present investigation, however, only one of the tested resin composites (TetricEvoCeram Bulk Fill) showed a significant increase in monomer conversion upon pre-heating, whereas no difference was found in the degree of conversion between

pre-heated and non-pre-heated groups for the other materials. Accordingly, the null hypothesis, stating that there is no significant difference in mean post-curing conversion between samples of resin composite that have been preheated and those cured at room temperature without preheating, could not be rejected [8].

Photo-initiator system used has a great role and affects the extent of polymerization of TetricEvoCeram Bulk Fill, producing marked increase in conversion of double bond due to pre-heating. This may be due to presence of (Ivocerin) in addition to conventional camphorquinone/ amine initiator systems. The presence of this germanium-based initiator with a higher quantum yield conversion compared to camphorquinone may have marked pre-heating effectiveness. This participated to increase the double bond conversion of Tetric EvoCeram Bulk Fill at raised pre-cure temperature [11].

Preheating nanohybrid composites to 37-68°C produces higher conversion rate. This elevated reaction rate may lead to an elevation of stress formation and accelerate expansion of the verification point which leads to damage of the bond at the resin/tooth interface [57]. Preheated composite showed increase in degree of conversion which resulted in superior properties of restoration such as micro-hardness as was suggested by Caneppele et al.,2011[70]. On the contrary, Didron et al [59] demonstrated that preheating composite resins have no significant effect on micro-hardness.

In the studies demonstrated by Dranoch et al, 2005 [27]; preheating composite before curing enhanced conversion rate without speeding up the time at which greatest rate of cure takes place at the top and at 2mm depth. This upgrade might be accomplished by amplified molecular mobility which resulted from increasing the temperature. The delay of diffusion, control of propagation and reaction diffusion controlled extinction and auto-deceleration, allowing the system to reach higher limiting conversions before verification. Consequently, a superior cross linked polymer network or oligomeric network is created. As a result, improved physical and mechanical properties might be predictable from resin composites when they undergo preheating to temperatures exceeding the room temperature.

The results of the current study may be attributed to the fact that increasing the temperature of composite resin material leads to elevation in the rate of the free radical mobility. This enhances extra polymerization, auto-acceleration, auto-deceleration and final conversion reaction, then cross-linking and mobility are reduced. The viscosity of the system becomes increased until the polymerization reaction stops due to polymer verification. Beginning of verification happens as diffusion reaction become very slow due to formation of the polymeric network. Thus, a slowdown in the polymerization processes takes place determining the final degree of conversion; therefore less unreacted residual monomer remains free producing better mechanical properties [27].

This comes in agreement with Daronch et al [27], Osternack et al [69] and Jim and Kim [71]. It was not in agreement with Torres et al [70] who stated that the preheating composite resins have no significant effect on micro-hardness, and demonstrated the profound impact of composite resin temperature on polymerization contraction of resin composites. Also, Didron PP et al. [59] stated that preheating of resin composites to higher temperature leads to marked elevation of the rate of polymerization and polymerization contraction stress. The increased stress at elevated temperature seems to be a consequence of the system thermal contraction rather than an increase in materials conversion, since the composites mechanical properties were not significantly improved at elevated temperatures[57,69].

Therefore, it was concluded that increasing temperature improves both radical and monomer mobility, leads to superior overall conversion and accelerates reaction rate diffusion; thus better physical and mechanical properties.

CONCLUSIONS

Under the conditions of this invitro study; it was concluded that the preheating to 37°C-68°C did not affect the micro-leakage of the tested resin composites. It was noticed that it affected the marginal adaptation of the composite resin materials to the cavity walls. It lowered the viscosity of the resin composite materials where it facilitated its introduction to the cavity by increasing its flowability.

Preheating could affect the hardness and conversion of the resin composite materials but it mainly depends on type of composite resin used, the amount of filler, depth of cure and the type of light curing unit used. So this will need further investigations. Also; more investigations are required for preheating and its effect on pulp vitality and intrapulpal pressure.

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