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Comparative evaluation of the effect of preheating on the degree of conversion and depth of cure of three bulk fill composite resins

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Abstract
Aim- *This study was undertaken to evaluate and compare the effect of preheating on the degree of conversion and depth of cure of three Bulk fill resin compositesTetric EvoCeram® (Ivoclar Vivadent) , Filtek™ Bulk fill(3M/ESPE)*
And EverX Posterior (GC Corporation,Europe)using using Fourier Transform Infrared Spectroscopy (FTIR) and Vickers's micro hardness test respectively.
Materials and Methods:
Total of 120 customized molds with cylindrical recesses of 5mm diameter and 4mm depth were fabricated. 60 specimens were randomly divided into 3 groups of 20 each based on the composite resin used. Each group were further divided into two subgroups consisting of 10 samples each according to the temperature used Room temperature and Preheated at 50 ^{0} C. The remaining 60 specimens also were grouped similarly for evaluation of Vickers hardness test. In the designated groups, a composite warmer were (i-warmer) used for preheating the composite to 50^{0}C. All the samples were cured according to their respective parameters. Degree of conversion and depth of cure were determined by using Fourier Transform Infrared Spectroscopy (FTIR) and Vickers's micro hardness test respectively.
Results
The results indicated that preheated composite groups showed a higher degree of conversion and surface hardness than the room temperature composite groups.Among the tested materials, the highest value for the degree of conversion, was observed with group III A(EverX Posterior preheated)and the lowest mean percentage value was observed Group II B(Filtek ™ bulk fill room temperature)Similar trends were observed in surface hardness also.
Conclusion:*Within the limitations of the study, it was concluded that there was a significant increase in the degree of conversion and surface hardness for all the preheated composites when compared with the room temperature composites. Among the three bulk fill composites tested EverX Posterior displayed the highest degree of conversion and surface hardness.*
Keywords: *Degree of conversion; Fourier transform infrared spectroscopy; Surface Hardness; Bulk fill; pre-heating Exposure time.*

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I. INTRODUCTION

Resin-based composites are a crucial component of contemporary restorative dentistry. Advances in various aspects of composite materials and their versatile application modes have revolutionized the treatment of carious posterior teeth and enabled a minimally invasive approach[1]

However, Polymerization shrinkage is an inherent phenomenon of resin based composites(RBC), resulting in the generation of stresses in the material and at the tooth-restoration interface, and can generate defects at the adhesive interface, including tooth cracks, cusp deflection, marginal leakage, gap formation and reduced mechanical properties . Bulk Fill composite resin has been introduced to the dental market as a new restorative concept. According to the manufacturer, the material can be used in a single step to fill cavities of a 4 mm depth or greater without affecting physical and mechanical properties at this depth,[2]. This effect is achieved through various strategies including development of more translucent materials, using lower concentrations of fillers to facilitate deeper light penetration and using more efficient photoactivation systems.Nevertheless, bulk fill has its own drawbacks; shrinkage stress could be greater when using bulk fill. composites and also there was chances for incomplete polymerization of these composites in the proximal deep cavity[3].Recent studies have focused on the effect of preheating on the properties of composite resins. In 2019, Lempel E. et al., suggested that preheating

positively affects the conversion rate of bulk-filled resin-based composites. Preheating, in turn, increases the mobility of monomer molecules in the resin matrix, increasing the formation of free radicals, resulting in higher DC values and shorter cure times[4].

II. MATERIALS AND METHODS

In this in vitro study three brands of bulk fill composites, Filtek ™ Bulk Fill(3M ESPE, USA), Tetric EvoCeram® Bulk Fill(Ivoclar Vivadent ,India) EverX Posterior (GC Europe) have been used. The composition, brand, chemical composition of the materials and the manufacturer are described in Table 1.

Material	Type	Shade	Organic matrix	Filler load wt% (vol%)	Photoinitiators	Manufacturer
Tetric EvoCeram® Bulk fill	Nanohybrid Bulk fill	A3.5	Bis-GMA, UDMA, Bis-EMA	80% (61%)	CQ, TPO, Ivocerin	Ivoclar Vivadent, Schaan, Lichtenstein
EverX Posterior	Fiber-reinforced bulk fill	Universal	Bis-GMA, PMMA, TEGDMA	74.2% (53.6%)	CQ, DMAEMA	GC Corp, Europe
Filtek™ Bulk Fill	Flowable bulk fill	A3	Bis-GMA, UDMA, TEGDMA	64.5% (42.5%)	CQ, EDMAB	3M ESPE, St. Paul, MN, USA

Bis-GMA: bisphenol A-glycidyl methacrylate, UDMA: urethane dimethacrylate, BisEMA: bisphenol A-ethyl methacrylate ,TEGDMA: triethylene glycol dimethacrylate, PMMA: poly(methylmethacrylate), CQ: camphorquinone, TPO: 2,4,6-tri-methylbenzoyl-diphenylphosphineoxid,DMAEMA:N,N dimethylaminoethyl-methacrylate, EDMAB: ethyl-4-N,N-dimethylaminobenzoate

A total of 120 customized molds with cylindrical recesses of 5mm internal diameter and 4mm depth were fabricated.
60 specimens were randomly divided into 3 groups of 20 each based on the composite resin used.
Group 1- Tetric EvoCeram® (Ivoclar Vivadent)
Group 2- Filtek ™ bulk fill (3M/ESPE)
Group 3- EverX Posterior (GC Corporation Europe)
Each group were further divided into two subgroups consisting of 10 samples each according to the temperature used
A) Room temperature
B) Preheated at 50 ^{0} C
Similar grouping were done for remaining 60 samples and the same procedure followed for evaluation of depth of cure.
Specimen preparation was performed at room temperature. The molds were positioned on a glass slab filled with various bulk fill composites in single increment. Excess material was extruded by application of pressure through the glass slide. Thereafter, the uncured RBC was covered with a polyester (Mylar) strip in order to avoid formation of oxygen inhibition layer which is an inhibitor of the polymerization. Immediately after that the specimens were irradiated from the top surface using Bluephase N LED Light curing unit (Ivoclar Vivadent) set at high intensity mode with an irradiance of 1200mW/cm ^{2} for 20 seconds according to manufacturer's instructions. The curing light guide was centrally positioned directly on the mold entrance and the tip of the light guide was ensured to be parallel to the sample.

In case of the pre-heated groups, the RBCs were preheated using a composite warmer
(i-warmer) that elevates composite temperature to 50°C. The mean time between removing composite from the device and light polymerization was approximately 40 seconds for all tests. The prepared pre-heated composite samples were photoactivated with the recommended irradiation time for each material, with the above described protocol.

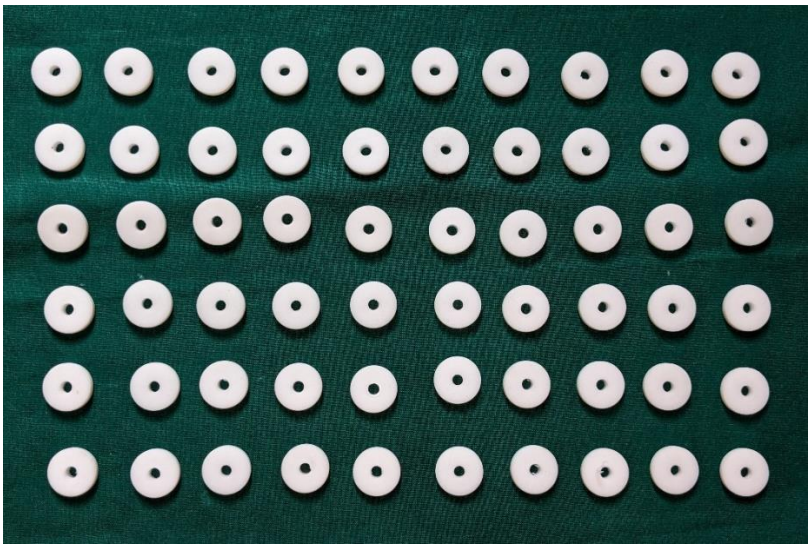


Fig No.1:60 Teflon mold (4 mm height and 5 mm internal diameter)

Fig No.2: Tetric EvoCeram® bulk fill composite

Fig No.3: Filtek ™ bulk fill composite

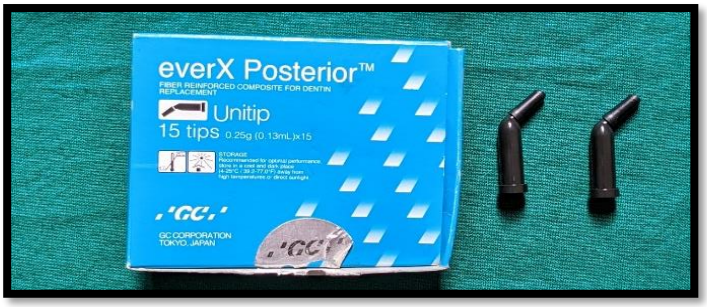
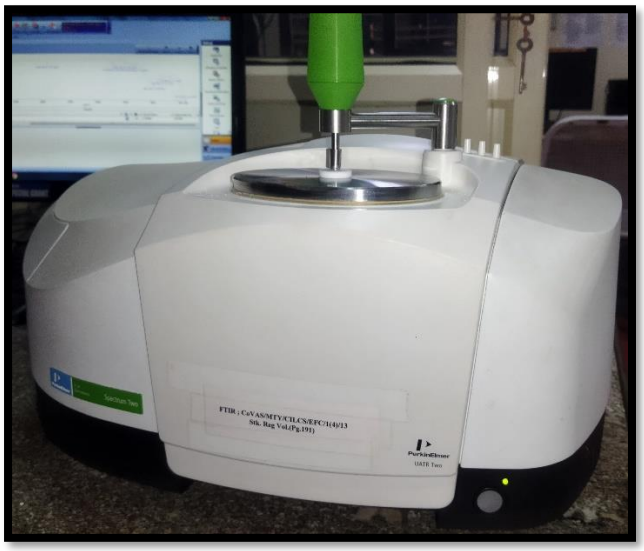


Fig No.4: EverX Posterior



FigNo.5: **FTIR (**Fourier transform infrared spectroscopy) Machine

Fig No.6: Vickers microhardness tester

Measurement of degree of conversion
Ftir – fourier transform infrared spectroscopy

The samples were stored for 24 hours at room temperature in a light-proof sealed glass container (to prevent water sorption on to the surface of sample and thereby preventing the potential source of noise in an FTIR analysis).The degree of conversion of bottom surface of the specimen were measured using Fourier transform infrared spectroscopy (FTIR) with an attenuated total reflectance (ATR) accessory (PerkinElmer UATR Two). Here the ATR crystal was placed in close contact with the bottom surface of the composite specimen and FTIR spectra ranging from 600 to 4000 cm^{-1} were documented. Total of 32 scans per samples were performed at a resolution of 4 $cm^{-}1$.The FTIR spectra of uncured RBCs were recorded at the start of the experiment, and DC was calculated using the following formula,

DC% = [1- (R Cured / R Uncured)] ×100

R corresponds to the ratio of the absorption intensities of the peak areas at 1638 and 1608 cm^{-1} in the spectra of the dimethacrylate-based composites.

Measurement of depth of cure
Vicker micro hardness test

The samples were stored in distilled water at room temperature for 24 hours, and the 24 hour post irradiation surface hardness were measured using Vickers micro hardness testing machine. The specimens will be loaded (500 g in 5 seconds) and Vickers hardness were calculated from the diagonals of the square-based pyramid impressions (Vickers intender).

Data was analyzed using the statistical package **SPSS 26.0** (SPSS Inc., Chicago, IL) and level of significance was set at **p<0.05**. **Descriptive statistics** was performed to assess the mean and standard deviation of the respective groups. Normality of the data was assessed using **Shapiro Wilkinson test**. Since the data was following normal distribution and parametric test were used for the data analysis. **Inferential statistics** to find out the difference between 2 groups was done using **Independent t test. One way ANOVA test followed by Tukey's HSD test** has been used to check the difference between three groups

III. RESULT

Degree of conversion

Mean and SD values of fracture toughness for each group are summarized in Table 2

There was significant difference regarding the mean degree of conversion values within room temperature and pre heated groups($p<0.05$). Regarding the three composite material tested reported significant difference between each other in the following manner (Everx posterior> Tetric EvoCeram® > Filtek^{TM})($P<0.05$) fig 1

Depth of cure

Mean and SD values of fracture toughness for each group are summarized in Table 2

The statistical analysis showed that significant difference regarding the mean depth of cure values within room temperature and pre heated groups($p<0.05$). Among the different material tested the everx posterior and tetric evo ceram showed comparable values while the filtek showed the laeast.

Table II- degree of conversion

	GROUP	ROOM TEMPERATURE	PRE HEATED	P VALUE (T TES)
FTIR	TETRIC EVO CREAM (1)	57.73±2.69	63.31±3.65	0.0001*
	FILTEK (2)	42.09±2.72	51.18±2.55	0.0001*
	EVERX POSTERIOR (3)	61.15±3.98	71.04±1.86	0.0001*
P VALUE (ONE WAY ANOVA TEST)		0.0001*	0.0001*	
POSTHOC TEST (P VALUE) TUKEY'S HSD TEST	1 v/s 2	0.0001*	0.0001*	
	1 v/s 3	0.0002*	0.0001*	
	2 v/s 3	0.0001*	0.0001*	

*P <0.05 is statistically significant

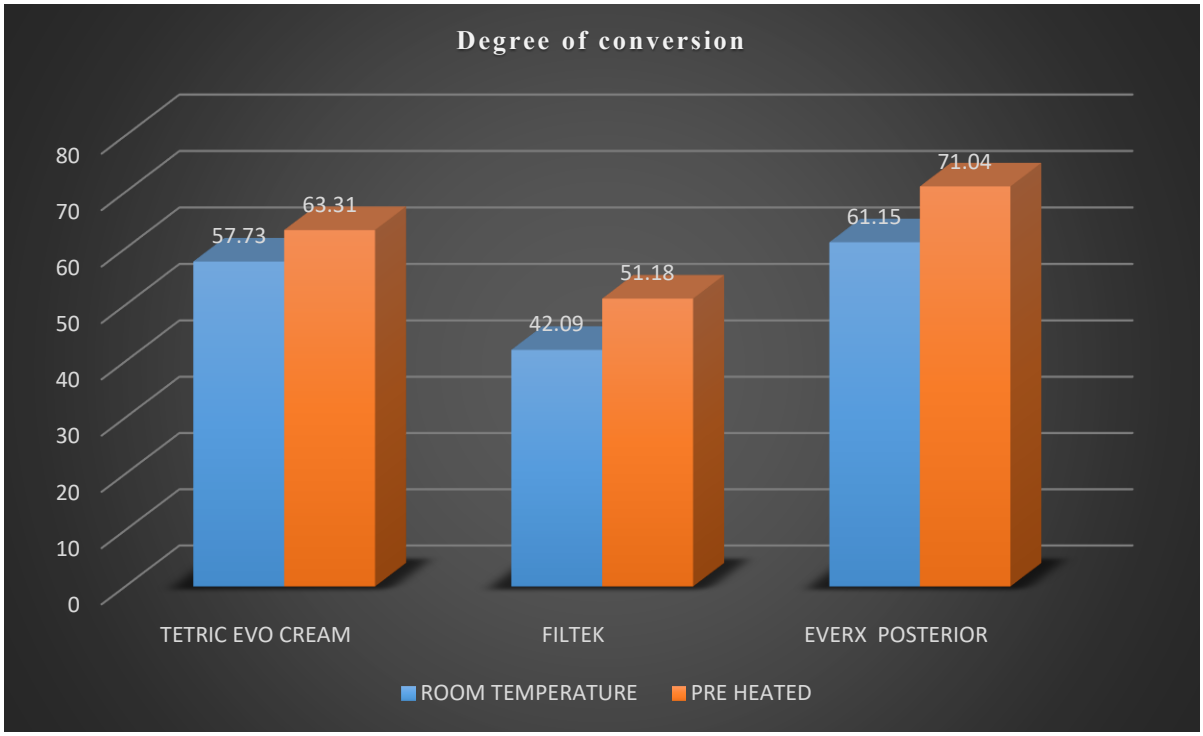
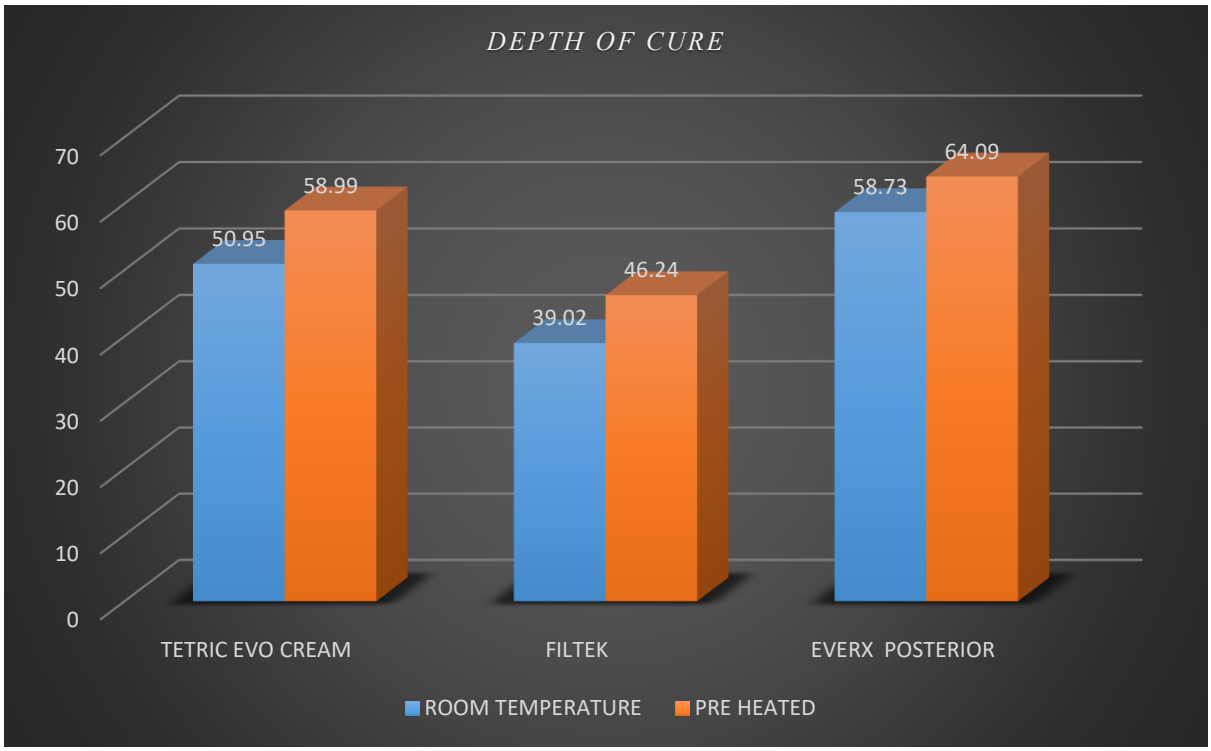


TABLE III- DEPTH OF CURE -VICKERS HARDNESS TEST

	GROUP	ROOM TEMPERATURE	PRE HEATED	P VALUE (T TES)
VICKERS HARDNESS TEST	TETRIC EVO CREAM (1)	50.95±2.20	58.99±2.92	0.0001*
	FILTEK™ (2)	39.02±3.24	46.24±2.54	0.0001*
	EVERX POSTERIOR (3)	58.73±3.19	64.09±5.19	0.0001*
P VALUE (ONE WAY ANOVA TEST)		0.0001*	0.0001*	
POSTHOC TEST (P VALUE) TUKEY'S HSD TEST	1 v/s 2	0.0001*	0.0001*	
	1 v/s 3	0.0001*	0.0001*	
	2 v/s 3	0.0001*	0.0001*	



IV. DISCUSSION

Bulk-filled composites consist of a ceramic fiber resin integrated into an elongated network of fillers approximately 100 nm long [5] and are claimed to have cure depths of up to 5 mm The matrix of these RBCs consists of a photoactivated dimethacrylate resin with a high proportion of random or porous fillers, with a filler content of 60-80 volume percent [6].

In this study three types of bulk fill composites have been used. The samples were prepared in a Teflon mold measuring 4mm depth and 5mm diameter, clinically relevant dimensions. The three bulk fill RBCs used were Filtek™ bulk fill from 3M ESPE, USA, Tetric EvoCeram® Bulk Fill-Ivoclar Vivadent ,India and EverX Posterior –GC Europe. These bulk fill RBCs claim to be placed in a layer of 4mm in a deep cavity[7].Tetric EvoCeram® Bulk Fill contains dibenzoyl germanium derivative referred to as ivocerin as an additional photoinitiator besides camphoroquinone-amine initiator .Ivocerin, features a high absorption coefficient capable of polymerizing the material at greater depths [8]. Filtek™ bulk fill (3M ESPE) is composed of 4 monomers, BisGMA, BisEMA, UDMA, Procrylat and BisEMA, having high molecular weight for reducing the polymerization shrinkage. In addition, Procrylat monomer allows greater fluidity reducing the shrinkage stress [9].

Everx posterior used as a 4 to 5mm bulk-fill dentin replacement in conjunction with a conventional composite as enamel replacement on top. According to manufactures the short fibers are incorporated to prevent and stop crack propagation through the restoration ; this fiber-reinforced composite is particularly recommended for large-sized cavities so that it can be used to restore also severely weakened teeth in a more economically affordable manner, such as teeth with extremely deep cavities, endodontic cavities, large amalgam cavities, and cavities with missing cusps [10].In this study samples of three groups were preheated before curing to a temperature of 50^0c. The aim was to determine the effect of preheating on the degree of conversion and depth of cure of three bulk fill composites in 4 mm deep clinically simulated cavities. After preheating, a statistically significant increase in micro hardness and degree of conversion were seen in all the three types of bulk fill composites used in this study.For prewarming composite resins, Calset composite warmer (AdDent, Inc., Dandury, CT, USA) was extensively studied in the literature to preheat the resin that operates at 54°C and 68°C. The efficiency of these warmers was studied by Daronch and co-workers[11]. Due to its increased cost and limited availability in our country, other preheating devices that are simple and easy to use can be tried.In the present study .The composites were preheated with a composite heating device(i-warmer),a manual device in which depending on the choice of material and personal preference ,temperature can be set up to 40^0C to 70 ^{0}C [12]. For this study the temperature was set at 50^0C and preheated for 15 min. The average preheating temperature found in the literature is 54°C -68 °C, considered a safe temperature for some authors . However, Knezevic et al. when assessing cellular toxicity resulting from preheating of resins at 68 °C suggested that this procedure may not be safe. Bortolotto and Krejci showed that, with a light cured composite, when temperatures were raised from 5°C to 40°C, significant increases

in Vickers Hardness were found at a curing depth of 0.5 mm [13]. Sharafeddin et al in 2015 also concluded that preheating the resin composite at 45˚C improves flexural strength and modulus of nanohybrid and silorane-based resin composite [14]. Another important consideration about preheating is the required time to achieve good fluidity and improvement of restorative material properties .There is a wide variation regarding this parameter in the literature, the minimum and maximum times found were 40 seconds to 24 hour. However, a reasonable clinical time is approximately 15 min, as used in this study similar to be used by Lucey et al and by Muhammadi et al .After preheating the composite, there is a time lapse between removing it from the heating device, placing it in the cavity, contouring it and light curing it. Lohbauer et al. confirmed that RBCs temperature rapidly falls to physiological level on removal from heating device .So in the present study The total insertion time from the material removal from the syringe to the insertion of the composite resin in the mold in one increment was limited to 40 seconds this is in accordance with the study of Sharafeddin et al. Decrease in paste viscosity was clinically examined in the study due to heating. Only moderate transient viscosity reduction was observed with 50ºC,15 minute preheating.

The alternative repeated preheating and cooling of composite that occurred during the preparation of all composite samples also might have influenced the material's properties and thereby the results, but D' Amario M et al in 2013 stated that repeated preheating cycles of 20 at 45°C did not affect the mechanical properties of composite materials tested [15] .

The minimum percentage of degree of conversion acceptable for restorative material at occlusal layers is 55% according to Soares et al In this study, the mean DC values obtained for each composite materials in room temperature were between 42.09% for Filtek™ 57.73 % for Tetric EvoCeram® and 61.15% for EverX Posterior .Preheated group were between 51.18 % for Filtek™ 63.31 for Tetric EvoCeram® and 71.04% for Everx posterior. So among both the conditions EverX Posterior showed highest monomer conversion values followed by Tetric EvoCeram® and Filtek™ bulk fill composites.

EverX Posterior, which is a Fiber-reinforced bulk fill composites showed a range of DC values about 71.04%in preheated group and 61.15% in group without preheating. This composite resin is intended to be used as base filling material in high stress bearing areas especially in large cavities of vital and non-vital posterior teeth.**[16]** It consists of a combination of a resin matrix, randomly orientated E-glass fibers and inorganic particulate fillers. The resin matrix contains bis-GMA, TEGDMA and PMMA forming a matrix called semi-Interpenetrating Polymer Network (semi-IPN) (net-poly(methyl methacrylate)- inter-net-poly(bis-glycidyl-A-dimethacrylate) which provides good bonding properties and improves toughness of the polymer matrix[17].It has been demonstrated by Lehtinan et al in 2008 that refraction indices and extinction coefficients change during polymerization of BisGMA-TEGDMA monomer systems of fiber reinforced composites which enhance light-induced polymerization to occur Similarity between the fiber/matrix refractive indices may allow light penetration into the deeper parts of the material. Le Bell et al. have shown that fiber-reinforced composites conduct and scatter the light better than conventional composite resin. Goracci et al. concluded in their experiment, that EX exhibited DOC over 4 mm, the maximum thickness recommended for bulk placement

Statistical analysis also revealed that DC values of Tetric EvoCeram® bulk fill composites were comparable with that EverX Posterior. The results are in agreement with the the study conducted by Jafarzadeh et al in 2015 who stated that silica nanoparticles and nanoclusters in nanohybrid composites have a light scattering effect thereby exhibited more variations in degree of conversion and microhardness . Due to its higher filler loading, its light intensity might be reduced by the effect of dispersing the light. Another reason for lower DC in tetric bulk fill composites might be due to its lower light transmission inside the bulk of the composite [18]. Since this bulk fill composites have a potent photoinitiator called Ivocerin to provide adequate depth of cure interestingly the results of our study are contradictory to it .Similar results were obtained by Taubock et al that In contrast to bulk-fill composites, Tetric EvoCeram® is indicated for use only in layers of max. 2-mm thickness, and would therefore not polymerize properly at 4–5-mm thickness .Additional photoinitiator (Ivocerin®) besides conventional camphoroquinone/amine initiator systems In Tetric might have affected pre-heating efficacy and contributed to the observed increase in monomer conversion of Tetric EvoCeram® Bulk Fill at elevated pre-cure temperature.

Among bulk-fill materials in the present study, Filtek bulk fill composite has the lowest filler loading and the highest translucency parameter. Despite of these advantageous parameters, Filtek bulk fill composite failed to reach the DC of the other investigated materials, In accordance with previous studies, the DC values for Filtek bulk fill composite were lower compared to the conventional flowable RBCs or to the other investigated bulk-fill materials[19]

In case of Filtek, besides Procrylate resin, BisGMA was combined with UDMA and BisEMA, instead of TEGDMA. Although the viscosity of UDMA is much lower than that of BisGMA, when it is mixed with the high

molec-ular weight BisEMA, it can significantly restrict the mobility of mobility of UDMA monomers and decrease their reactivity and conversion value.

Micro hardness of a RBC material does not reflect only the extent of polymerization, but other factors such as filler content and filler size, largely affect hardness results. Consequently, the comparisons among the tested materials do not concern the degree of conversion but their hardness as a mechanical property

The outcomes of the present study indicated that EverX Posterior showed the highest Vickers hardness at 4 mm. Most striking was the translucency of Everx, this must most likely be associated with the glass fibers with an average length of 1.3–2 mm. Previously, the presence of these fibers in experimental fiber-reinforced composite materials has been associated with improved flexural strength and load-bearing capacity, and reduced shrinkage stress and microleakage[20].Among the tested material Filtek™ showed the lowest value for micro hardness. This findings can be understood by the nature of its organic matrix .Among the modification proposed by the manufacturer, different monomers were incorporated into its composition such as high molecular weight aromatic di methacrylate (AUDMA) capable of increasing the rigidity of polymer chain formed. It decreases the number of reactive groups on the organic matrix and hinders their mobility during polymerization reaction[21]. It was also demonstrated by Habib and Waly in 2018[22]

V. CONCLUSION

In this study the effect of preheating on degree of conversion and depth of cure of three bulk fill resin composites; Tetric EvoCeram® Bulk fill(Ivoclar vivadent)Filtek™ bulk fill(3M/ESPE) and GC EverX Posterior composite were evaluated by FTIR and Vickers micro hardness test .
Within the limitations of the present study, it could be concluded that

1. There was a significant increase in degree of conversion and surface hardness for all the preheated composites when compared with the room temperature composites

2 Among the three bulk fill composites Everx preheated composites displayed the highest degree of conversion and surface hardness.

3. The degree of conversion and depth of cure of Tetric EvoCeram® were comparable with other tested bulk fill composites

4. Bulk fill Filtek™ composites showed the lowest degree of conversion the lowest surface hardness

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