EFFECT OF PREHEATING AND RADIANT EXPOSURE ON DEGREE OF CONVERSION AND MICROHARDNESS OF BULK FILL COMPOSITES: AN INVITRO STUDY

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CERTIFICATE

This is to certify that **DR.T. VAIBHAVI**, Post Graduate student (2017-2020) in the Department of Conservative dentistry and Endodontics, Adhiparasakthi Dental College and Hospital, Melmaruvathur – 603319, has done this dissertation titled **"EFFECT OF PREHEATING AND RADIANT EXPOSURE ON DEGREE OF CONVERSION AND MICROHARDNESS OF BULK FILL COMPOSITES: AN INVITRO STUDY "** under our direct guidance and supervision in partial fulfilment of the regulations laid down by the Tamilnadu Dr.M.G.R Medical University, Chennai – 600032, for MDS degree examination, (Branch-IV Conservative dentistry and Endodontics).

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DECLARATION

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PLACE OF THE STUDY	ADHIPARASAKTHI DENTAL COLLEGE AND HOSPITAL, MELMARUVATHUR – 603319
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I hereby declare that no part of the dissertation will be utilized for gaining financial assistance or any promotion without obtaining prior permission of the Principal, Adhiparasakthi Dental College and Hospital, Melmaruvathur – 603319. In addition, I declare that no part of this work will be published either in print or in electronic media without the guides who have been actively involved in dissertation. The author has the right to reserve for publishing this work solely with the permission of the Principal, Adhiparasakthi Dental College and Hospital, Melmaruvathur – 603319.

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ABSTRACT

AIM:

Aim of this study is to assess and compare the effect of preheating and variant radiant exposure on the degree of conversion and microhardness of three different bulk fill composites.

MATERIALS AND METHODS:

In this study 3 different bulk fill composites, Filtek ^{TM} Bulk Fill-3M, SureFil SDR flow-Dentsply, Palfique^{R} Bulk Flow- Tokuyama, were selected. Twenty-eight composite discs of 4mm thickness were prepared for each bulk fill composite. According to the method of polymerization four groups were formed,

Group1: High intensity, No preheating the composite (n=7 per composite)

Group 2: High intensity, Preheating the composite (n=7 per composite)

Group 3: Normal intensity, No preheating the composite (n=7 per composite)

All the samples were cured according to their respective parameters and degree of conversion and microhardness were determined by using Fourier Transform Infrared Spectroscopy (FTIR) and Vickers's microhardness test respectively.

STATISTICAL ANALYSIS AND RESULTS:

The statistical analysis was performed using SPSS software. A multivariate ANOVA (MANOVA) was done with 3 independent variables namely, intensity of curing light, preheating the composite and type of composite and 4 dependent variables namely Microhardness on top and bottom surface and Degree of conversion on top and bottom surface

of the composite samples. The combined values of dependent variables were used to assess the characteristic of composite.

CONCLUSION:

Within the limitations of the study it was concluded that all the three types of bulk fill composites achieved significant microhardness and degree of conversion with the high intensity and preheating parameters.

LIST OF ABBREVIATIONS

DOC or DC	-	Degree of conversion
MH	-	Microhardness
RBCs	-	Resin Based Composites
BFCs	-	Bulk fill composites
Bis-GMA	-	Bis-Glycol dimethacrylate
UDMA	-	Urethane dimethacrylate
Bis-EMA	-	Bis- ethoxylated dimethacrylate
EBPADMA	-	Ethoxylated bisphenol A glycol Dimethacrylate
TEGDMA	-	Triethylene glycol dimethacrylate
LED	-	Light Emitting Diode
FTIR	-	Fourier transform infrared spectroscopy

INTRODUCTION

Direct composite restorations in the posterior teeth have become an imperative element of recent era in dentistry[1]. The striking features of dental composites in relation to other restorative materials are its handling characteristics, aesthetic appearance and clinical durability[2]. However, a major hindrance in the usage of dental composites is its polymerization shrinkage, and its reverberations are poor marginal seal and secondary caries, postoperative sensitivity[3], recontamination and following failure of the endodontic treatment[4].

Resin based composites (RBCs) have been introduced in the market for many years which are in the surge of widely replacing the dental amalgam with the Minamata convention 2013 calling for its phase out[5]. The use of RBC as a restorative material in class I and class II cavities have shown clinical success according to various studies[6,7]. Enormous attempts have been made to improvise the mechanical properties by altering the composition of the composite.

Thickness of 2 mm for layering technique is the bench mark for composite resin placement and curing[8]. The technique sensitivity and time imbibing in cases of deeper posterior restorations or during coronal sealing of an endodontically treated tooth led to introduction of BULK FILL COMPOSITES. They are preconceived to reduce the shrinkage and the polymerization stress by using similar exposure time and light intensity used for normal composites[9]. These composites are available as low and high viscosity bulk filling composites, which usually have a higher translucency, and a modified initiator to establish better curing depth, as compared to conventional composites. The low viscosity material can be used as a base and they require an additional capping layer and the high viscosity material is used to fill the cavities. These materials are recommended to be used in 4 mm or even 5 mm in thickness

without stratification and are proposed to be used in class I, II, and class IV restorations[10]. These bulk fill composites have shown to reduce cuspal deflection[11].

Layering technique used in conventional composites are integrated with several disadvantages, such as i) Contamination and failure of bonding between the layers, ii) limitation to access in smaller cavities iii) time consuming.

The composition of bulk fill RBCs are almost similar to that of conventional RBCs[12]. The matrix of bulk fill RBCs are made up of monomers of Bis-GMA, UDMA, TEGDMA, and EBPDMA[12]. Filler content in these composite resins ranges from 60% to 80% by volume[13]. The inter-locking particle technology is the precedence for the bulk-fill composites where mixtures of different-sized filler particles are used. When the particles are packed together the larger particles mechanically interlock with the small particles[14]. In some cases, different monomers have been added and the classic Bowen monomer (Bis-GMA:2,2- bis [4-(2-hydroxy -3-methacryloxyprpoxy) phenyl] propane) has been modified[15].

Nonetheless, bulk fill has its own disadvantages; the shrinkage stress might be more when bulk-fill composites are used. The polymerization of these composites might be incomplete in the proximal deep cavity, leading to improper contact areas, which necessitates usage of adequate matrices[16].

SureFil SDR flow (Dentsply Caulk) emerged into the market in 2010 which was the first of its kind that endorsed the possibility of being used in the increments of up to 4mm[15]. The manufacturer of SDR have patented a resin of dimethacrylate urethane, which has greater molecular flexibility and avoids the stress generated at the time of curing. Hence it is named as the stress decreasing resin technology (SDR)[17].

3M ESPE affirms that Filtek Bulk fill is based on 4 monomers: BisGMA, UDMA, Procrylat, and BisEMA, having high molecular weight, which reduces the polymerization

shrinkage. In addition to that Procrylat monomer allows for greater fluidity which also lowers the polymerization stress[18].

Tokuyama asserts that the spherical fillers of the Supra-Nano particles used in PALFIQUE BULK FILL provide a uniform diffusion of light, allowing for a more forgiving shade match and superb blend to surrounding teeth. In addition, the spherical and round fillers provide low composite wear over time and safe for opposing dentition while causing less wear on opposing teeth. The catalyst technology adopted for PALFIQUE Bulk fill is the Radical Amplified Photo polymerization initiator (RAP technology). As a major feature, the initiator balances the high polymerization activity needed to cure the resin with short exposure times (1/3rd of that required by conventional products) and stability in ambient lighting[19].

One of the pertinent characteristics to be assessed for the bulk fill composite is its adequate curing depth in resin increments of 4mm or more as indicated by manufacturers. As per ISO 4049-2009 standard, the curing depth should not be less than 0.5mm than what has been established by the manufacturers[20]. A study conducted by ADA recently assessed the curing depth of 10 different bulk fill RBCs, which stated that SureFil SDR, Filtek Bulk Fill, has curing depth values equal or greater than what is required by the ISO in Bulk Fill RBCs[21].

Adequate marginal integrity is related to lesser polymerization stress; hence RBCs are expected to produce proper marginal integrity in hostile cavity conditions with a high C factor. Several studies have shown insignificant differences in marginal integrity of conventional and bulk fill RBCs[22].

The polymerization shrinkage which is the adverse effect of polymerization reaction is mediated by rigidity of the RBCs, its releasing ability, and its curing rate. Cuspal flexure, tooth fracture are the effects of polymerization stress which reduce the mechanical properties of the material[23]. The capacity of incremental technique in reducing the polymerization stress

have been questioned by many authors[46]. Studies evaluating shrinkage and polymerization stress in bulk fill RBCs are very less. Ilie et.al. stated that development of polymerization stress is lower in bulk fill RBCs, when compared to the conventional RBCs[17]. According to Garcia et. al., different bulk fill RBCs showed different values of polymerization shrinkage, either smaller, larger and similar to that of conventional RBCs[25]. Hence it was found that polymerization shrinkage varied significantly according to the product.

Vickers microhardness values at the surface and at certain depths have been proposed to determine the depth of cure[26] and additionally it also provides the information on material wear, polishing ability and abrasive effect on antagonist tooth[27]. The bulk fill composites containing the nano fillers were found to exhibit higher microhardness values due to more intimate contact of nanofillers with the resin matrix[28].

Preheating the composites prior to light curing is gaining popularity among the dentists as a method to improve the handling characteristics during its placement in a cavity[29]. It is known to reduce the viscosity of the material[48], augment the marginal adaptation[31] and decrease microleakage due to improved wetting of walls of cavity[32]. Preheating the composites is also known to amplify the monomer mobility resulting in higher conversion[33] which leads to an increase in the physical and mechanical properties of the materials[34].

The potency of polymerization is also affected by exposure time, intensity of curing light, distance between the light guide tip of the light cure unit and restorative material surface[35]. According to Selig et al. an exposure time of only 10 s and above gave a sufficient DC[36], thus increasing the light exposure time resulted in a higher radiant exposure reaching the RBC increment, particularly with conservative cavity preparation[37].

Hence this study was formulated to assess and compare the effect of preheating and variant radiant exposure on the degree of conversion and microhardness of the bulk fill composite.

AIMS AND OBJECTIVES

AIM:

To assess and compare the effect of preheating and variant radiant exposure on the degree of conversion and microhardness of the three bulk fill composites and determine their characteristics.

OBJECTIVES:

1. To evaluate the effect of preheating on the degree of conversion of the three bulk fill composites.
2. To evaluate the effect of preheating on microhardness of the three bulk fill composites.
3. To evaluate the effect of variant radiant exposure on the degree of conversion of the three bulk fill composites.
4. To evaluate the effect of variant radiant exposure on microhardness of the three bulk fill composites.
5. To compare the effects of three bulk fill composites in respect to its degree of conversion and microhardness.

NULL HYPOTHESIS:

1. There was no effect of preheating the composite on degree of conversion and microhardness of the bulk fill composites.
2. There was no effect of variant radiant exposures on degree of conversion and microhardness of the bulk fill composites.
3. There was no significant difference in the performance of the 3 bulk fill composites investigated under the mentioned parameter.

REVIEW OF LITERATURE

Ferracane JL et al. 1985[38], determined the nature of the correlation between the Knoophardness and the degree of conversion of carbon double bonds, as evaluated by IR analysis,for unfilled dental restorative resins.

Sakaguchi et al.1992[39] described about the variables affecting light energy absorption by the composite and their effect on the polymerization contraction. Then onwards, the contraction due to polymerization stress is associated closely to the degree of cure of the restoration, this parameter served as an empirical indicator for the extent of polymerization. Variables which were included are shade of the composites, distance between the source of light and composite sample, and light intensity. Three resin composites were evaluated. Post-gel polymerization contraction was assessed using a strain gauge method. Curing light intensity reduced rapidly for distances greater than 2 mm between the tip of the light guide and material surface. A dependant relationship was shown between polymerization contraction and light intensity. The contraction due to polymerization of a micro filled composite and composites used for posterior teeth, using a curing time and light intensity which were constant. decreased linearly with increasing sample thickness. Output less than the optimal light output of the curing light source can be compensated by increasing application time within reasonable limits.

Imazato et al. 1995[40], elucidated the relationship between the degree of conversion and internal discoloration of light activated composite. The degree of conversion was estimated by Fourier transformation infrared Spectroscopy. The results indicate that the greater the degree of conversion, the less the discoloration of composite, and the correlation between the two factors were significant for light-activated composite.

Imazato et al. 2000[41], compared the efficacy of degree of conversion values using Differential thermal analysis (DTA) and Fourier transmission infrared spectroscopy (FTIR) for the light activated composites. DTA was considered convenient method and evaluated the usefulness of the DTA method.

Lale G Lovell et al. 2001[42] investigated the effect of cure rate on the mechanical properties of dental resin formulations. This study showed highly cross linked dimethacrylate systems exhibit similar network structure and properties as a function of double bond, irrespective of type of cure.

Ferracane et al. 2002[43] verified the influence of degree of conversion and speed of polymerization reaction on contraction stress by submitting one of the composites to different photo-activation times. Contraction stress was maintained for 10 minutes in a tensilometer. Fourier-transformed infrared spectrometry was used for evaluation of the degree of conversion. Volumetric shrinkage was assessed by means of a mercury dilatometer. Degree of conversion and volumetric shrinkage displayed a non-linear relationship with energy density. Degree of conversion showed a prominent influence on stress. Increased inhibitor concentration decreased curing rate and contraction stress in composites, without compromising the final degree of conversion.

Calheiros FC et al 2006[44], verified the influence of radiant exposure on contraction stress, degree of conversion and mechanical properties of two restorative composites. Results showed that contraction stress and microhardness were more sensitive to increasing radiant exposure. Degree of conversion was not affected.

Prasanna et al 2007[45], determined effect of the preheated resin composite heated to different temperatures on degree of conversion and residual stress and compared it to composite at room temperature. The results showed standard increase in the degree of conversion and residual stress with increase in preheating temperature.

Junkyu Park et al.,2008[46], studied the different techniques of composite placement in a cavity to assess its effect on cuspal deflection. This study showed that effective reduction in polymerisation shrinkage was seen with incremental layering technique.

Lohbauer U et al 2009[47], determined the monomer conversion and polymerization shrinkage of resin composites after various pre heating procedures. It was concluded that preheating of resin composite does not increase degree of conversion over time.

Lucey et al 2010[48], evaluated the effect of preheating resin composite on precured viscosity and post cured surface hardness. It was concluded that pre heating resin composites reduced the pre cured viscosity and enhanced its subsequent surface hardness.

Neeraj Malhotra et al. 2010[49], reviewed on bulk fill RBCs, explaining their compositions, advantages, and disadvantages, that are contemporary in today's clinical practice and those that are under research or in clinical trial phase.

Flavio F Demarco et al. 2012[50], assessed the longevity of the posterior composites and reviewed that a longer survival rate composite restoration depends on patient, material and operator factors.

Roggendorf et al.2012[51], evaluated the marginal integrity of bonded posterior resin composite to enamel and dentin. This study showed better performance of SDR as 4mm bulk fill dentin replacement.

Simon Flury et al. 2012[52], evaluated the accuracy of depth of cure determined by ISO 4049 when compared to Vickers hardness. The value of ISO was found to be overestimated when compared to Vickers Hardness.

Ruwaida Z. Alshali et al. 2013[53]**,** estimated the degree of conversion (DC)using FTIR for bulk-fill flowable resin composite materials and the conventional flowable and regular resin composite materials.

Liah Finan et al. 2013[54], determined the influence of irradiation potential on the degree of conversion and mechanical properties of two bulk-fill flowable RBC base materials. The declaration which states that the bulk-fill flowable RBC bases have a depth of cure in excess of 4mm can be confirmed but the differing chemistry of the resin formulations and filler properties contribute to statistically significant differences in DC and VHN data between the two materials tested.

Randolph et al. 2014[55], proved the null hypotheses that the resin composites which contain a photoinitiator of type 1 exhibited reduced DC or enhanced monomer elution at short curing

times compared to materials based on Type 2 ketone/amine system. Lucirin-TPO was found to be more efficient at absorption and convertion photon energy when using a curing-light with an appropriate spectral emission. The use of a set of curing protocols in this study has shown the potential of Lucirin-TPO and its impact for clinical applications, in replacement to materials using camphorquinone.

Karen V Ayub et al 2014[56], determined the effect of temperature on microhardness and viscosity of four resin composites. The results showed that preheating the resin composites increased the microhardness and decreased the viscosity of the samples.

Calheiros et al 2014[57]**,** tested the effect on degree of conversion and polymerization stress by increasing the temperature. They concluded that increasing the temperature allows for reduced exposure duration and lower polymerisation stress while maintaining or increasing degree of conversion.

Robert L. Erickson et al.2014[58], examined the effect of different parameters of curing on the depth of the cure within each configuration, for a specific resin-based composite (RBC) and found out a significant effect.

Dimitrios et al 2015[59], evaluated the microhardness of two composite resins, subjected to three different temperature and three different light curing times. The results showed that there is an increase in microhardness as the temperature of the composite is increased.

Taubock TT et al 2015[60], investigated the influence of preheating of high viscosity bulk fill composites on their degree of conversion and shrinkage force formation. Results showed that preheating the bulk fill composites reduced the polymerisation shrinkage without compromising degree of conversion.

Akshay Langalia et al 2015[61], stated that the greatest restraint in the use of compositeresins as a posterior restorative material is the polymerisation shrinkage during polymerization, which often leads to marginal fracture and subsequent secondary caries,marginal stains, displacement of restoration, teeth fracture and, or post-operative sensitivity.

Dimitrios Dionysopoulos et al 2015[62], evaluated the polymerization efficiency of bulk fill resin-based composites (RBCs) and the effect of their composition, temperature and post-irradiation polymerization on the results.

Kusai Baroudi et al.2015[63], presented the various current methods of decreasing viscosity of resin composite materials such as by using flowable composites, heating the composites and applying sonic vibration. These methods improved the handling properties and facilitated its bonding to cavities with complicated forms, decreased the time for procedure and improved marginal adaptation.

Alkhudhairy FI et al. 2017[64] investigated the effects of two curing light intensities on the mechanical properties (Vickers microhardness, compressive strength, and diametral tensile strength) of the bulk-fill resin-based composites (RBCs). A curing light with high intensity of

1200 mW/cm^2 had a better influence on the compressive and tensile strength of the bulk-fill RBCs used and microhardness of two materials tested compared to lower curing light intensity of 650 mW/cm^2. SDR cured with high-intensity light exhibited the greatest diametral tensile strength among the four materials.

Mariana et al. 2017[65], through his systematic review assessed the literature to determine the efficiency of polymerization of bulk-fill composite resins at 4 mm restoration depth. Regardless to the method performed in vitro, bulk fill RBCs were partially likely to fulfill the important requirement regarding properly curing in 4 mm of cavity depth measured by depth of cure and degree of conversion. The low viscosities Bulk fills performed better regarding polymerization efficiency compared to the high viscosities' bulk fills.

P Yu et al. 2017[66], evaluated the degree of conversion and polymerization shrinkage of a bulk-fill resin-based composites and giomer material. At all depths, SDR had the highest degree of conversion values. No significant difference in Degree of conversion was observed between depths at 2 mm and 4 mm for the bulk-fills, Degree of conversion at 2 mm was significantly greater than at 6 mm. For the conventional resin based, Degree of conversion at 2 mm was significantly higher than at 4 mm and 6 mm. Mean Polymerization shrinkage ranged from 1.48% to 4.26%. The DC at 2 mm and Polymerization shrinkage of bulk-fills were lower than the conventional resin based composites . At 4 mm, the Degree of conversion of giomer bulk-fills was lower than that of non giomer bulk-fill materials.

Jessica Dias Theobaldo et al 2017[67], evaluated the effect of composite preheating and polymerisation mode on degree of conversion, microhardness, plasticization and depth of polymerisation of a bulk fill composite. They concluded that composite preheating increased

the polymerisation degree of 4mm increment bulk fill and had no significant effect on microhardness of composites.

Alizadeh Oskoee et al. 2017[68], observed that Gap formation at the gingival margins of Class V cavities decreased due to preheating of silorane based composite resins.

Maan M et al. 2017[69], evaluated various factors influencing the polymerisation of resin-based composites. His results showed that the physical properties of clinically used RBCs enhanced by preheating the composites, through a specific device. Additionally, the use of LED unit, preferably the one with polywave system, covers a broader range and activate more photo initiator.

Mahdi Abbasis, et al. 2018[70], assessed the polymerization shrinkage of five BFCs composites and compared them with a conventional . The results showed that the bulk-fill composites tested had a polymerization shrinkage similar to that of the conventional composite.

Yousef T. Eshmawi et al. 2018[71], supervised the variation in composite degree of conversion and flexural strength for many curing lights and surface locations. It stated that the irradiance-beam profile from the different LCUs evaluated did not have a major effect on the DC and micro flexural strength for the investigated composite.

Lempel et al. 2018[72], assessed the (DC) degree of conversion of types of resin-based composites (RBC) in clinically relevant moulds, and investigated the influence of exposure

time and pre-heating on DC. The study determined that the increased exposure time improved the DC for each material. Pre-heating the low-viscosity RBCs reduced the DC% at the bottom. Pre-heating of the fibre-reinforced RBC to a temperature of 55°C increased the DC% at a higher rate than the extended curing time.

Zrinka et al. 2018[73] reviewed the various factors determining the DC, properties of composite materials which are dependent on the DC, as well as methods used to determine the DC. The DC is a basic attribute of a cured composite as it affects virtually all other material properties that are important for the clinical success of the restoration. Though the composition of contemporary composites is adjusted to attain optimal DC and the related properties if properly handled and light-cured, poor DC due to unfavourable curing conditions or operators' improper understanding of the curing procedure may affect critical material properties and increase the risk of clinical failure.

Meereis et al.2018[74] conducted a systematic review to determine the approach available to decrease and control polymerization shrinkage stress development in resin-based restorative dental materials. It was concluded that modification of the resin matrix made the largest contribution in minimizing stress development. The technology used for decreasing stress in the formulation of low-shrinkage and bulk-fill materials has shown to be a promising application for reducing and controlling stress development.

JK Gan et al. 2018[75], compared the consequence of cure on bulk-fill composites using polywave light-emitting diode (LED; with various curing modes), monowave LED, and conventional halogen curing lights. There was no significant difference in hardness ratios observed between curing lights/modes for Tetric N-Ceram bulk-fill, the hardness ratio obtained with Bluephase N Monowave was significantly higher than the hardness ratio obtained for Bluephase N Polywave Low for SDR.

Nikolaos Stefanos et al 2018[76], compiled all the laboratory trials regarding composite preheating and investigated their effects on the material. They concluded that preheating had a positive effect on the degree of conversion, marginal adaptation and microhardness of composite resins.

Leticia Nunes et al 2018[77], investigated the influence of preheating and post curing methods on microhardness and degree of conversion of fibre reinforced composites. The results showed that the mechanical properties were increased by preheating the composites but degree of conversion remained unaffected.

Carlos et al 2018[78], evaluated the effect of radiant exposure on physio-chemical and mechanical properties of micro hybrid and nano filled composites.it was concluded that increasing the radiant exposure had a positive effect on degree of conversion and mechanical

properties of material investigated.

Karacan et al 2019[79]conducted a study with the aim of measuring in vitro intrapulpal temperature effect when placing room temperature or preheated (54°C and 60°C) in bulk-fill composite. It was concluded that preheating does not pose significant problems in terms of intrapulpal temperature increase. Though the preheating process results in an increase in intrapulpal temperature, this is not the critical factor which causes harm to the pulp. Clinical significance of this study showed that preheating can improve material features.

Dhakshinamoorthy Malarvizhi et al. 2019[80], assessed that shrinkage cannot be eliminated completely but there are numerous methods to reduce it. Therefore, the clinician should implement any of these methods to improve the success rate and longevity of the composite resin restorations and reduce the polymerization shrinkage.

MATERIALS AND METHODS

ARMAMENTARIUM

1. Filtek ™ Bulk Fill-3M ESPE, USA
2. SureFil SDR flow-Dentsply, USA
3. Palfique^{R} Bulk Flow- Tokuyama, Japan
4. Light cure unit – I LED (Woodpecker) with intensity meter.
5. Composite warmer- Modified Glass bead sterilizer (Unikdent, India)
6. Glass slides
7. Teflon moulds (RS PRO PTFE, India)
8. Mylar Strips

EQUIPMENT

1.Vickers hardness tester

2. Fourier transform infrared spectroscopy (FTIR)

PROCEDURE

METHODOLOGY:

PREPARATION OF THE COMPOSITE RESIN SPECIMENS

In this in vitro study three brands of bulk fill composites of shade A2 have been used. The composition, brand, chemical composition of the materials and the manufacturer are described in Table 1.

The Filtek bulk fill is composed of Bis-Glycol dimethacrylate (Bis-GMA), Urethane dimethacrylate (UDMA), Bis- ethoxylated dimethacrylate (BisEMA), Procrylat resin as organic matrix. SDR contains modified UDMA, Ethoxylated bisphenol A glycol dimethacrylate (EBPADMA), Triethylene glycol dimethacrylate (TEGDMA) as organic

matrix. Tokuyama Palfique bulk fill consist of Bis-GMA, Bis-MPEP and TEGDMA as organic matrix. According to the polymerization method, the samples prepared were categorized into four experimental groups. In each group, 7 specimens from each material, were prepared. Table 2 shows the experimental groups according to the method of polymerization and the abbreviations of the investigated materials.

Teflon moulds of cylindrical shape, with 5 mm internal diameter and 8 mm height, representing deep proximal cavity or a pulpal chamber were constructed, according to the recommended thickness of the investigated materials. The schematic diagram of sample preparation is presented in Fig. 1. Specimen preparation was performed at room temperature set at 27◦C. Materials with recommended 4 mm layer thickness were condensed or filled with a canula into the 8 mm high mould part, which was positioned on a glass slide. Each sample was assessed for uniform thickness. 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 specimen was irradiated with a Light Emitting Diode (LED) curing unit [Woodpecker, Maximum intensity of 3000mW/cm2, Twin mode - p1 (high intensity mode) p2(normal intensity)] with an 8 mm diameter fiberglass light guide. The irradiance of the LED source was monitored before and after curing with a radiometer (Woodpecker). The curing light guide was centrally positioned directly on the mould 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 modified glass bead sterilizer as a composite warmer[81]. It is a simple device, in which common salt is used instead of glass beads. Though the glass beads retain heat, they stick to the syringe by aggregation. The glass bead sterilizer has a thermocouple inside the circuit which can be altered according to the temperature requirement with the help of an electrician. It takes 2-3 min to preheat the

composite. The prepared pre-heated composite samples were photoactivated with the recommended irradiation time for each material, with the above described protocol.

MEASUREMENT OF DEGREE OF CONVERSION

FTIR – FOURIER TRANSFORM INFRARED SPECTROSCOPY

Degree of conversion of resin composites, denotes the conversion of monomeric carbon=carbon double bonds into polymeric carbon–carbon single bonds[60] Increased degree of conversion culminates in high surface hardness, flexural strength and modulus, fracture toughness and diametral tensile strength and increased wear resistance. This improvement in its properties is known to be because of increased cross-linkage[61].

Amidst several methods to determine the degree of conversion (DC) of composites, Fourier transformation infrared spectroscopy (FTIR) has been authenticated to be a powerful technique and it distinguishes the C=C stretching vibrations directly before and after curing of materials[40,82]. Hence this device was selected for its accuracy.

The FTIR spectrometer (Avatar 360, Nicolet Analytical instruments) operated under the following conditions: 1680 and 1550 cm-1 at a rate of one per second, using 8 scans at 2 cm-1 resolution. After curing the samples were stored for 24 hours at 37^0C within a closed glass container to prevent water adsorption. For all the samples, DC was evaluated by determining the variation in the ratio of the absorbance intensities of aliphatic C= C peak at 1638 cm−1 and that of an internal standard peak of aromatic C= C at 1608 cm−1 of the uncured and cured samples. Due to the lack of aromatic C= C, internal standard peaks at 1600 cm−1 and 1720 cm−1 were used in the case of SDR[83]. The DC was determined by subtracting the % C=C from 100%, according to the equation:

$$DC\% = \{1 - (a / b)\} \times 100$$

a = absorption of aliphatic C–C / absorption of aromatic C–C (polymer)

b = absorption of aliphatic C=C / absorption of aromatic C=C (monomer)[41]

Measurement of Microhardness

VICKERS MICROHARDNESS TESTER

The Vickers microhardness technique utilizes the lengths of the cracks emerging from the corners of a hardness indentation to determine the fracture toughness of a brittle material[84]. This method has gained considerable recognition because of its relative ease of application, which unlike the conventional techniques, does not require extensive machining or sample preparation. Only a small sample size is required to estimate the fracture toughness of the material[85,86]. Sufficient hardness denotes that the placed restorative materials are resistant to in-service scratching, resulting from both mastication and abrasion. Indentations were conducted on the polished faces of the specimens using a Vickers diamond pyramid at various peak contact loads[87].

SCHEMATIC DIAGRAM OF SAMPLE PREPARATION WITH TEFLON MOULD

Curing light guide tip

Teflon mould

(5mm internal

Diameter)

bulk fill composite

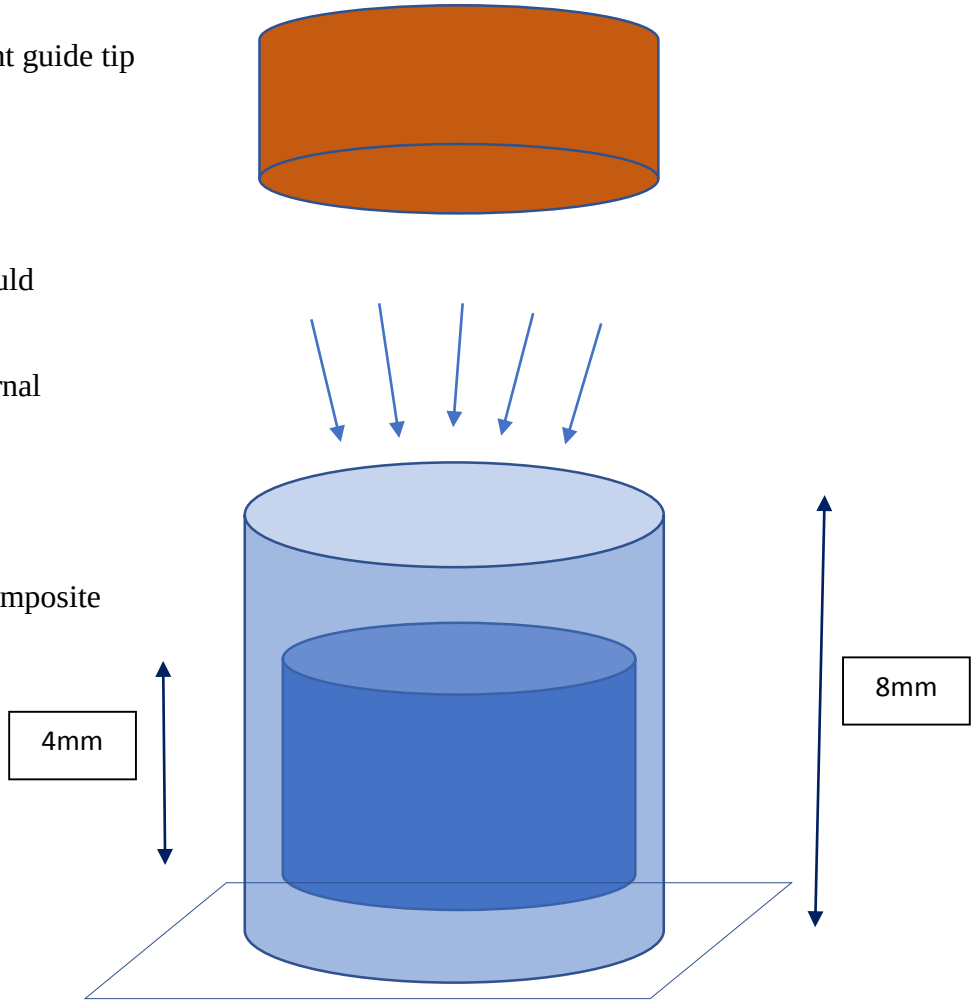


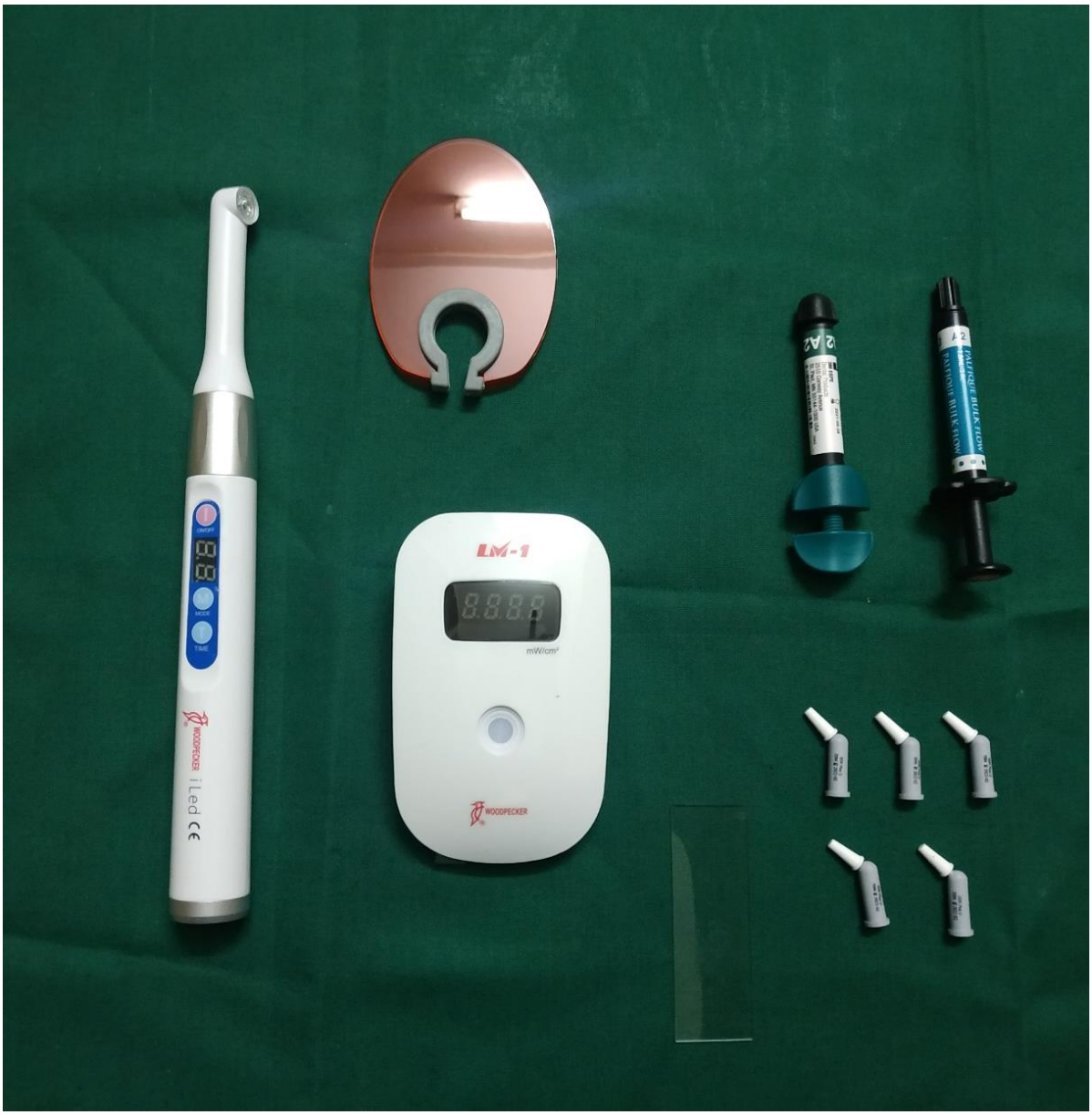
Table 1: MATERIALS MANUFACTURER AND COMPOSITION OF BULK FILL RBCs

NAME	MATERIAL LAYER THICKNESS	MANUFACTURER	SHADE	ORGANIC MATRIX	FILLER LOADING
Filtek	4mm	3M ESPE	A2	BisGMA, UDMA, BisEMA, Procrylat resin	64.5% by wt Zirconia /silica, Ytterbium trifluoride
SDR	4mm	Dentsply	A2	Modified UDMA, EBPADMA TEGDMA	68% by wt Ba-Al-F-B silicateglass, Sr-Al-F
Palfique	4mm	Tokuyama	A2	Bis-GMA, Bis-MPEP, TEGDMA,	Supra nano spherical filler 70% by wt SiO_2-ZrO_2

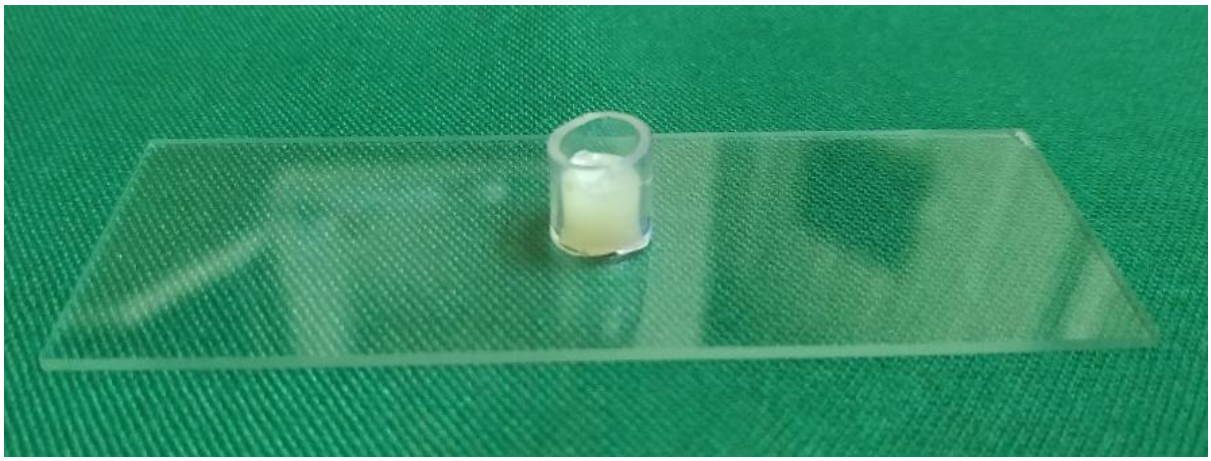
Table 2: EXPERIMENTAL GROUPS ACCORDING TO THE METHODS OF POLYMERIZATION

GROUPS	METHOD OF POLYMERIZATION	TEMPERATURE	EXPOSURE (1 sec exposure)
GROUP 1	High intensity, No preheating the composite	25^0C	P1 MODE ($2500mw/cm^2$)
GROUP 2	High intensity, Preheating the composite	55^0C	P2 MODE ($1000mw/cm^2$)
GROUP 3	Normal intensity, No preheating the composite	25^0C	P1 MODE ($2500mw/cm^2$)
GROUP 4	Normal intensity, Preheating the composite	55^0C	P2 MODE ($1000mw/cm^2$)

ARMAMENTARIUM



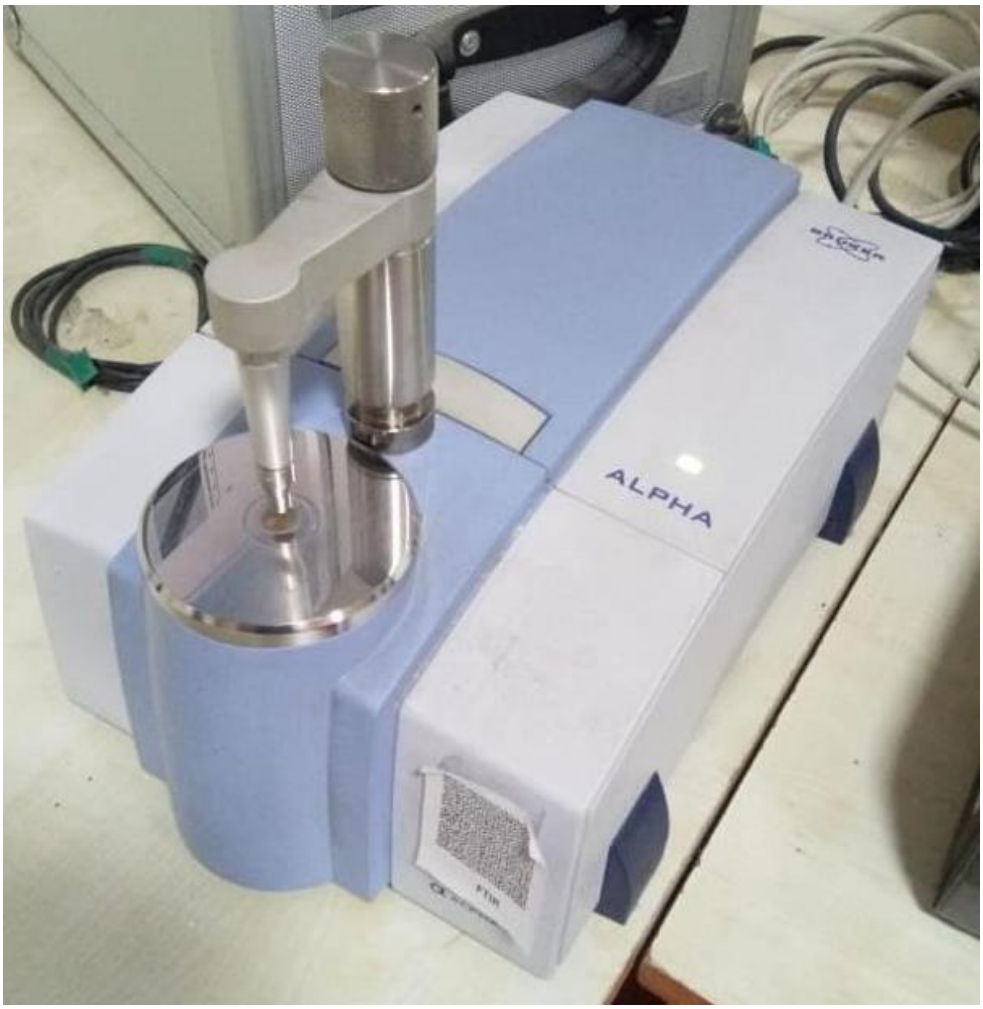
COMPOSITE SAMPLE – TEFLON MOULD



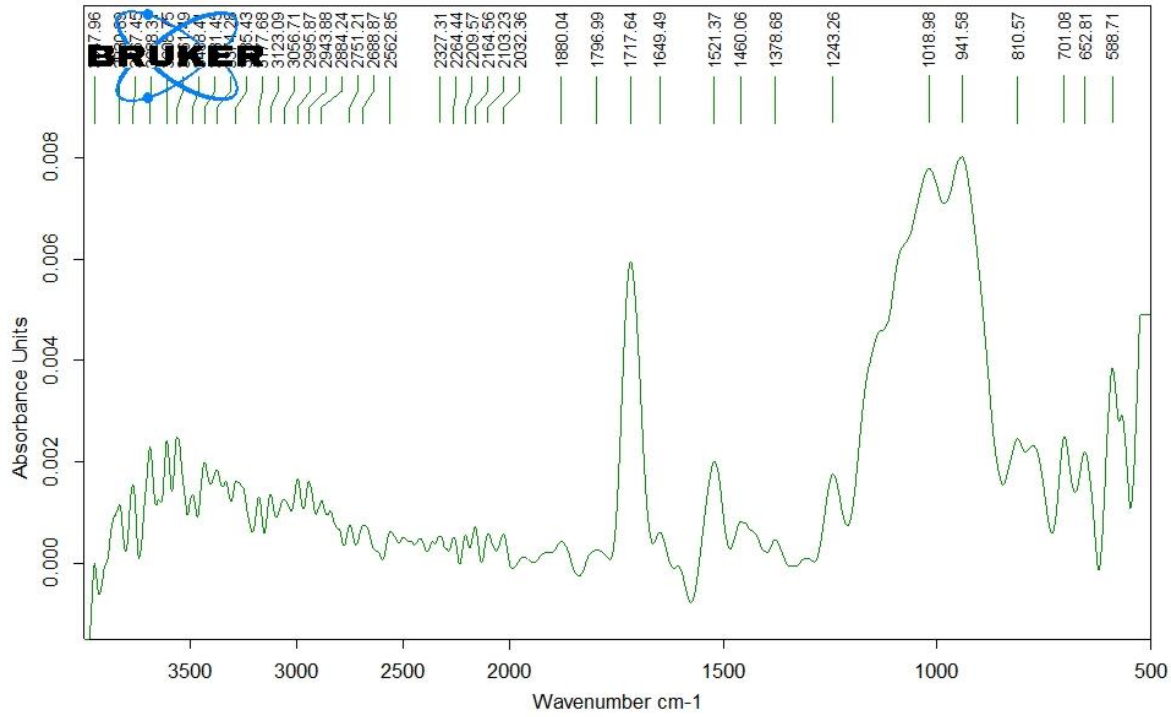
MODIFIED GLASS BEAD STERILISER

VICKERS MICROHARDNESS TESTER

FTIR – FOURIER TRANSFORM INFRARED SPECTROSCOPY



FTIR ANALYSIS RESULTS



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STATISTICAL ANALYSIS

The statistical analysis was performed using SPSS software. A multivariate ANOVA (MANOVA) was done with 3 independent variables namely:

- Intensity of curing light
- Preheating the composite and
- Type of composite

and 4 dependent variables namely

- Microhardness on top surface
- Microhardness on bottom surface
- Degree of conversion on top surface
- Degree of conversion on bottom surface of the composite samples.

The combined values of dependent variables were used to assess the characteristic of composite.

RESULTS

All the four dependent variables were normally distributed and assessed by Shapiro Wilk's test ($p>0.05$). There was Homogeneity of Covariances matrices as assessed by Box's test of equality of Covariances ($p>0.01$), but the variances were not homogenous as assessed by Levene's test.

There was a statistically significant interaction between three independent variables on combined dependent variables as assessed by Wilk Lamda test($p=0.032$)

Next a univariate 3-way ANOVA was performed. These showed statistically significant interaction effect among three factors.

- Tables 3-5 show the degree of conversion values according to the method of polymerisation, assessed for three types of bulk fill composites on top and bottom surfaces.
- Tables 6-8 show the degree of microhardness values according to the method of polymerisation assessed for three types of bulk fill composites on top and bottom surfaces.
- Table 9shows Descriptive status of composite type, intensity and preheating.
- Table 10 shows Multivariate tests.
- Graph 1 shows degree of conversion at the top surface and preheating and intensity of curing light
- Graph 2 shows degree of conversion at the bottom surface and preheating and intensity of curing light
- Graph 3 shows microhardness at the top surface and preheating and intensity of curing light
- Graph 4 shows microhardness at the bottom surface and preheating and intensity of curing light

Post hoc tests showed statistically significant values for degree of conversion and microhardness on top and bottom surfaces of all composite types.

TABLE3: **DEGREE OF CONVERSION(DOC) OF FILTEK BULK FILL COMPOSITES POST 24 HOURS OF CURING.**

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	65.8	57.8
2	68.5	58.4
3	67.4	59.4
4	68.9	63.5
5	62.6	57.4
6	59.7	52.3
7	67.5	59.4

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	69.7	64.9
2	70.6	65.4
3	72.5	64.3
4	69.9	59.4
5	69.6	62.4
6	70	61.2
7	68.8	59.6

GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	64.2	58.8
2	65.7	58.4
3	66.8	60
4	63.2	59.2
5	67.5	61
6	66.8	59.4
7	69.1	57.7

GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	67.9	58.9
2	68.9	59.6
3	70.1	60.7
4	68.5	60.2
5	67.5	59.7
6	69.3	60.2
7	67.4	59.6

TABLE 4: DEGREE OF CONVERSION(DOC) OF SDR BULK FILL COMPOSITES

POST 24 HOURS OF CURING

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	89.2	79.6
2	88.5	79.4
3	89.9	78.9
4	88.9	79
5	87.8	80.5
6	89	80.2
7	87.2	76.8

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	92.2	88.7
2	93.1	85.6
3	92.9	84.9
4	91	85.5
5	89	80.1
6	87.2	79.9
7	92.1	83.4

GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	84.3	75.4
2	81.3	72.5
3	87.4	76.4
4	85.7	75.2
5	88	75.7
6	89.1	78
7	88.4	76.7

GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	89.2	81.5
2	90.4	82.4
3	93.2	83.5
4	90.2	80.4
5	91.6	86.7
6	89.9	85.9
7	93.7	88.5

TABLE:5 DEGREE OF CONVERSION(DOC) OF TOKUYAMA BULK FILL COMPOSITES POST 24 HOURS OF CURING

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	78.5	69.6
2	79.5	70.2
3	77.5	69.5
4	80.3	76.4
5	74.8	70.2
6	79.3	69.3
7	78.9	69

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	85.2	79.8
2	86.7	76.8
3	88.8	78.5
4	85.3	77.6
5	84.1	75.4
6	84.2	73.9
7	84.6	74.3

GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	78.6	67.5
2	73.2	68.3
3	74.3	66.6
4	72.9	65.4
5	73.4	64.5
6	78.9	68.9
7	76.4	65.6

GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE DOC (%)	BOTTOM SURFACE DOC (%)
1	79.6	72.1
2	80.4	70.3
3	81.3	69.9
4	80.9	70.1
5	82.5	75.4
6	80.5	73.8
7	83.3	73.2

TABLE: 6 MICROHARDNESS (MH) OF FILTEK BULK FILL COMPOSITES POST 24 HOURS OF CURING

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	32.2	26.6
2	32.4	25.9
3	34.2	25.6
4	30	25.8
5	33.4	23.5
6	32.4	26.7
7	33	26.8

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	39.6	32.2
2	37.5	33.4
3	38.4	32.1
4	37.6	33.4
5	38.7	33.5
6	39	35.4
7	36	34.3

GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	28.2	26.2
2	28.5	24.5
3	28	24.8
4	28.3	26.7
5	28.5	28
6	28.2	26.4
7	28.4	26.7

GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	30.4	29.4
2	31.4	28.4
3	32.3	29.6
4	32.5	26.4
5	31.8	27.9
6	32.2	29.2
7	32	28.4

TABLE:7 MICROHARDNESS (MH) OF SDR BULK FILL COMPOSITES POST 24 HOURS OF CURING

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	48.4	44.1
2	48.9	43.8
3	45.6	44.1
4	48.7	43.5
5	48	44.7
6	47.8	46.6
7	46.9	46.6

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	52.4	48.2
2	52.3	48.1
3	52.6	48
4	51.2	47.9
5	52.9	48.2
6	52.8	47.5
7	50.2	47.9

GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	35.6	33.2
2	34.9	33.3
3	34.5	32.8
4	35.1	29.6
5	35.5	28.5
6	34.7	29.4
7	35.3	30.4

GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	35.1	30.7
2	35.2	30.6
3	35	31.6
4	36.4	31.6
5	37.4	30.6
6	38.3	33
7	35.6	32.5

TABLE: 8 MICROHARDNESS (MH) OF TOKUYAMA BULK FILL COMPOSITES

POST 24 HOURS OF CURING

GROUP I: HIGH INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	38.3	35.6
2	38.6	35.7
3	38.5	37.8
4	33.5	28.5
5	33.5	28.5
6	32.7	28.5
7	36.2	34.9

GROUP II: HIGH INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	40.6	37.5
2	40.8	37.6
3	40.9	38.2
4	42.6	40.1
5	38.7	35.7
6	37.9	34.4
7	39.1	35.2

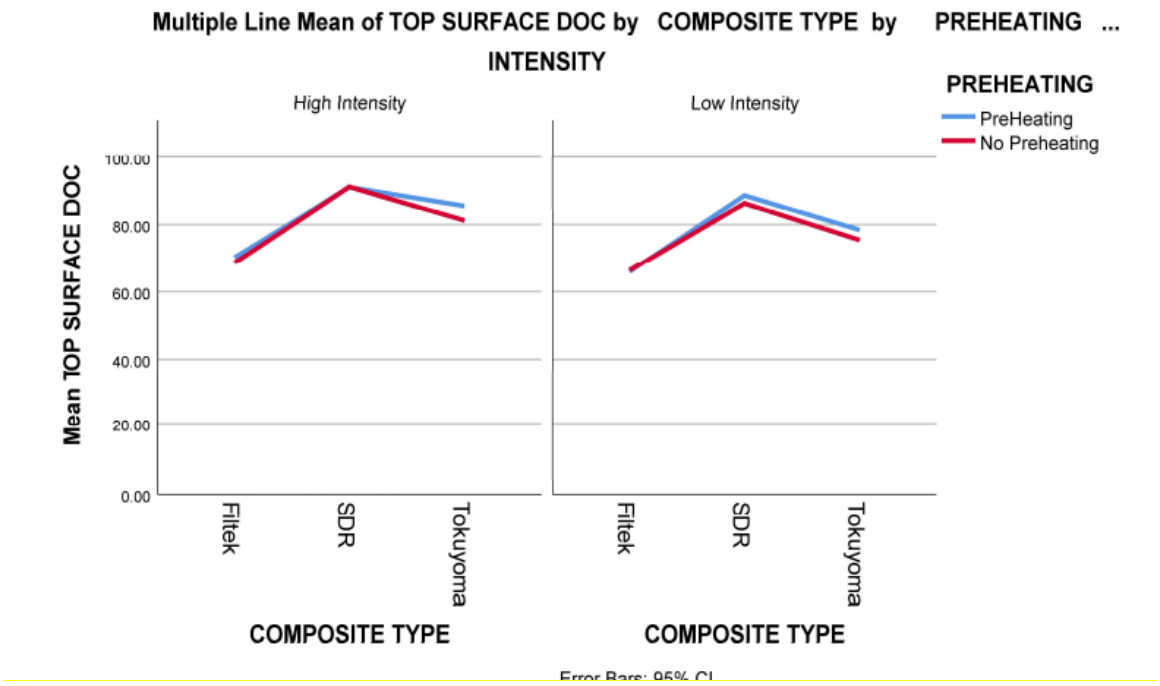
GROUP III: NORMAL INTENSITY, NO PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	35.4	30.2
2	35.5	32.2
3	35.4	30
4	34.7	32.1
5	35.7	33
6	36.1	32
7	37.1	33.4

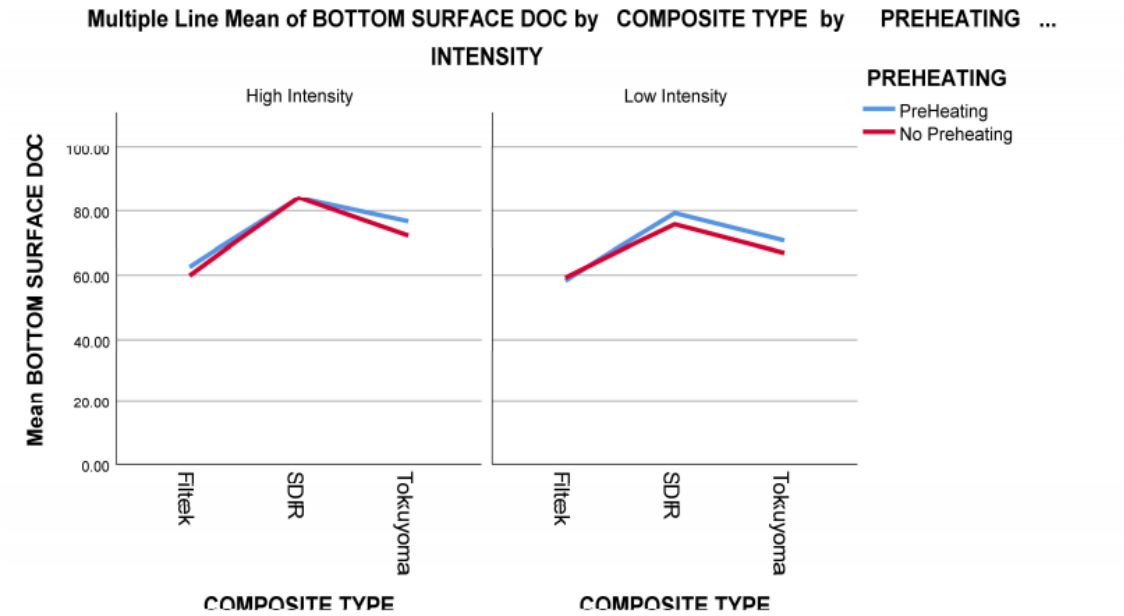
GROUP IV: NORMAL INTENSITY, PREHEATING THE COMPOSITE

SAMPLES	TOP SURFACE MH	BOTTOM SURFACE MH
1	38.3	33.6
2	38.4	31.3
3	36.3	32.5
4	39.2	33.4
5	38.6	33.2
6	39.4	34.2
7	38.9	33.7

GRAPH 1: GRAPHICAL REPRESENTATION OF DEGREE OF CONVERSION AT THE TOP SURFACE AND PREHEATING AND INTENSITY OF CURING LIGHT



GRAPH 2: GRAPHICAL REPRESENTATION OF DEGREE OF CONVERSION AT THE BOTTOM SURFACE AND PREHEATING AND INTENSITY OF CURING LIGHT



GRAPH:3: GRAPHICAL REPRESENTATION OF MICROHARDNESS AT THE TOP SURFACE AND PREHEATING AND INTENSITY OF CURING LIGHT

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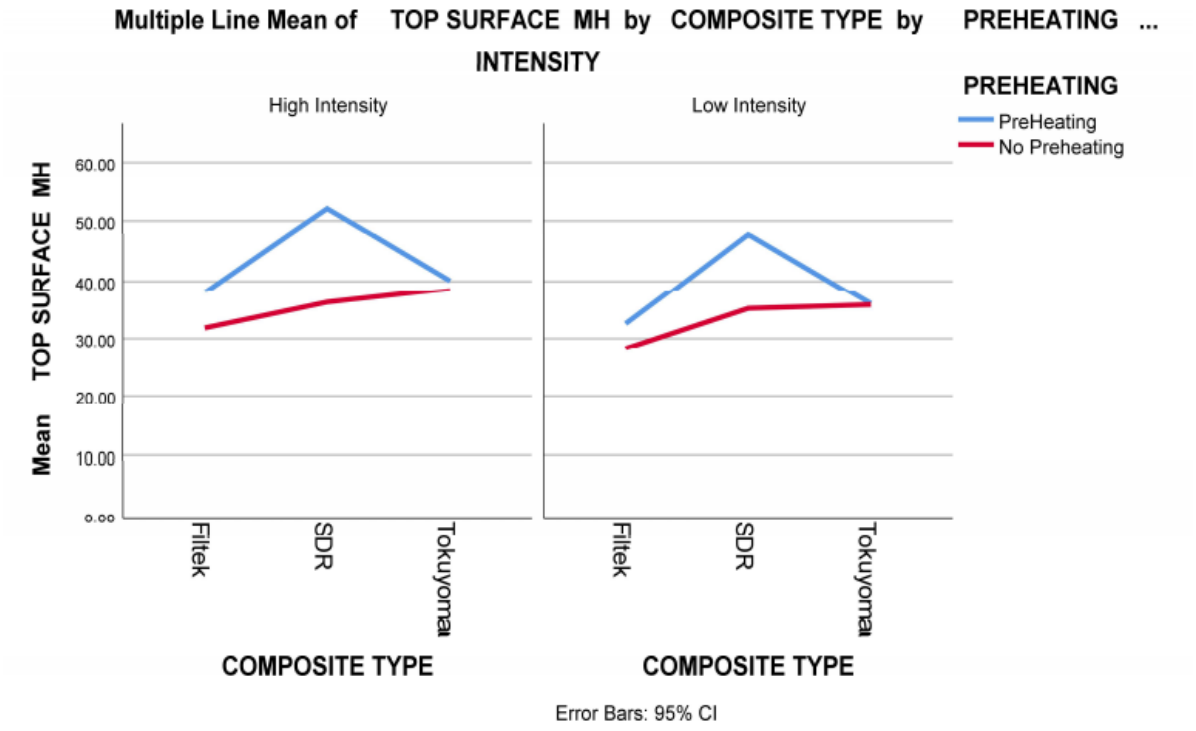

GRAPH 4: GRAPHICAL REPRESENTATION OF MICROHARDNESS AT THE BOTTOM SURFACE AND PREHEATING AND INTENSITY OF CURING LIGHT

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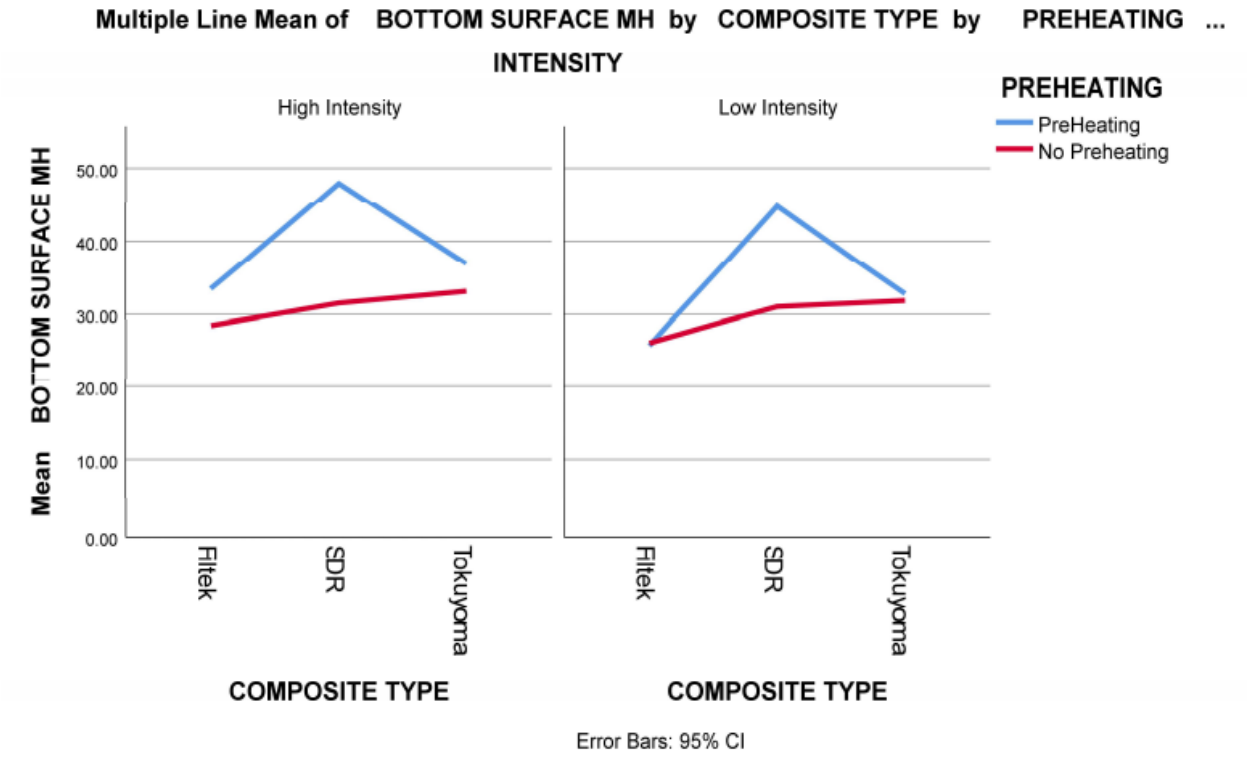

Table 9: DESCRIPTIVE STATUS: COMPOSITE TYPE, PREHEATING, INTENSITY

95%Confidence Interval

Dependent Variable	Composite type	Preheating	Intensity	Mean	Std. deviation	Lower bound	Upper bound
Top surface MH	Filtek	Preheating	High	38.114	1.19224	37.167	69.061
			Low	32.5143	1.31076	31.567	33.461
		No preheating	High	31.800	.71414	30.853	32.747
			Low	28.300	.18257	27.353	29.247
	SDR	Preheating	High	52.0571	.99307	51.110	53.004
			Low	47.7571	1.15882	46.810	48.704
		No preheating	High	36.1429	1.28304	35.196	37.090
			Low	35.0857	0.40999	34.139	36.033
	Tokuyama	preheating	High	40.0857	1.60357	36.139	41.033
			Low	35.9000	2.63502	34.953	36.847
		No preheating	High	38.4429	1.02771	37.496	39.390
			Low	35.7000	0.74610	34.753	36.647
Bottom surface MH	Filtek	Preheating	High	33.4714	1.14850	32.178	34.765
			Low	25.8429	1.13850	24.549	27.136
		No preheating	High	28.4714	1.10259	27.178	29.765
			Low	26.1857	1.19921	24.892	27.479
	SDR	Preheating	High	47.9714	0.24300	46.678	49.265
			Low	44.7714	1.30092	43.478	46.065

		No preheating	High	31.5143	0.95991	30.221	32.808
			Low	31.0286	2.02049	29.735	32.322
	Tokuyama	Preheating	High	36.9571	1.97219	35.664	38.251
			Low	32.7857	4.10546	31.492	34.079
		No preheating	High	33.1286	0.95867	31.835	34.422
			Low	31.8429	1.29596	30.549	33.136

Dependent Variable	Composite type	Preheating	Intensity	Mean	Std. deviation	Lower bound	Upper bound
Top surface DOC	Filtek	Preheating	High	70.1571	1.16456	68.641	71.673
			Low	65.7714	3.41063	64.255	67.288
		No preheating	High	68.5143	0.99403	66.998	70.031
			Low	66.1857	2.00286	64.669	67.702
	SDR	Preheating	High	91.0714	2.20130	89.555	92.588
			Low	88.6429	0.90343	87.127	90.159
		No Preheating	High	91.1714	1.71922	89.655	92.688
			Low	86.3143	2.76009	84.798	87.831
	Tokuyama	Preheating	High	85.5571	1.67815	84.041	87.073
			Low	78.4000	1.81016	76.884	79.916
		No Preheating	High	81.2143	1.28378	79.698	82.731
			Low	75.3857	2.57516	73.869	76.902
Bottom surface DOC	Filtek	Preheating	High	62. 571	2.48721	60.763	64.151
			Low	58.3143	3.32988	56.620	60.009

		No Preheating	High	59.8429	0.57982	58.149	61.537
			Low	59.2143	1.07770	57.520	60.909
	SDR	Preheating	High	84.0143	3.16461	82.320	85.709
			Low	79.2000	1.20968	77.506	80.894
		No Preheating	High	84.1286	2.97361	82.434	85.823
			Low	75.7000	1.70098	74.006	77.394
	Tokuyama	Preheating	High	76.6143	2.19502	74.920	78.309
			Low	70.6000	2.59551	68.906	72.294
		No Preheating	High	72.1143	2.12401	70.420	73.809
			Low	66.6857	1.62217	64.991	68.380

Table 10: MULTIVARIATE TESTS

Effect	Wilks' Lambda Value	F	Hypothesis Df.	Error Df.	Sig.	Partial Eta Squared
Intercept	0.000	50516.422[b]	4.000	69.000	0.000	1.000
Composite type	0.023	95.720[b]	8.000	138.000	0.000	0.847
Preheating	0.097	159859[b]	4.000	69.000	0.000	0.903
Intensity	0.197	70.184[b]	4.000	69.000	0.000	0.803
Composite type * preheating	0.084	42.247[b]	8.000	138.000	0.000	0.710
Composite type * intensity	0.604	4.947[b]	8.000	138.000	0.000	0.223
Preheating * intensity	0.725	6.554[b]	4.000	69.000	0.000	0.275
Composite type * Preheating * Intensity	0.790	2.157[b]	8.000	138.000	0.035	0.111

DISCUSSION

Bulk fill composites (RBCs) are into the market for past the two decades, introduced as packable and condensable composites[16]. Their use for the restoration of posterior teeth is being sought after due to their mechanical properties and aesthetic needs[88]. These new composites have been manufactured with the aim of decreasing the working time by reducing the layers that have to be cured during their placement in a cavity, and at the same time care has been taken to minimise polymerisation shrinkage[9]. In an attempt to reduce the polymerisation shrinkage, a change has been made in the composition that is by altering filler matrix composition and improving the translucency or by changing the photo initiator system[89].

Layering technique of composite placement in the cases of deep cavities is associated with several drawbacks, including time consumption and contamination in between the layers[39,90]. Compared to incremental layering techniques, bulk fill composites exhibited reduced cuspal deflection and improved marginal integrity[11].

Bulk fill composites consist of ceramic fibre resin consolidated into the elongated filler network of about 100nm in length[91] and are claimed to have a curing depth up to 5mm[92]. These RBCs are recommended to be used in class I, II and VI restorations. The matrix of these RBCs is composed of light activated, Di-Methacrylate resins with a higher percentage of either irregular or porous fillers with filler loading ranging from 60%-80%by volume[93].

In this study three types of bulk fill flow composites have been used. The samples were prepared in a Teflon mould measuring 8mm depth and 5mm width which resembles the deep proximal cavity or a cavity for post endodontic restoration. The three bulk fill RBCs used were Filtek bulk fill flow from 3M ESPE, SDR bulk fill flow from Dentsply, and Palfique bulk fill flow from Tokuyama. These bulk fill RBCs claim to be placed in a layer of 4mm in a deep cavity.

Filtek bulk fill flowable (3M ESPE) is composed of 4 monomers, BisGMA, BisEMA, UDMA, Procrylat and BisEMA, having high molecular weight for reducing the polymerisation shrinkage. In addition, Procrylat monomer allows greater fluidity reducing the shrinkage stress[18].

SDR (Dentsply) is based on "Stress Decreasing Technology"[94]. This allows for greater molecule flexibility, hence preventing polymerisation stress.

Tokuyama claims that the spherical fillers of the Supra-Nano particles used in PALFIQUE BULK FILL provide a uniform diffusion of light, allowing for a more forgiving shade match and superb blend to surrounding teeth. In addition, the spherical and round fillers provide low composite wear over time and safe for opposing dentition while causing less wear on opposing teeth. The catalyst technology adopted for PALFIQUE Bulk fill is the Radical Amplified Photo polymerization initiator (RAP technology). As a major feature, the initiator balances the high polymerization activity needed to cure the resin with short exposure times ($1/3^{rd}$ of that required by conventional products) and stability in ambient lighting[19].

In this study samples of two groups were preheated before curing to a temperature of 55^0C. The aim was to determine the degree of conversion in 8 mm deep clinically simulated cavities. After preheating, a statistically significant increase in microhardness and degree of conversion on top and bottom surface were seen in all the three types of bulk fill composites used in this study. The composites were preheated with a modified glass bead sterilizer, invented by Dr.Arora[81], in which glass beads are replaced with common salt. The unit has a thermocouple inside in the circuit which can be modified according to clinician's requirements. For this study the temperature was set at 55^0C. It took 10 minutes to pre heat the unit and 2-3 min to warm the composite. The syringe, pre-loaded guns can be directly used.

Preheating decreases the viscosity of the restorative material, enhances the marginal adaptation and decreases microleakage[95] and at the same time maintains or increases degree of conversion and cross linking by improving free radical and monomer mobility, by enhanced collisions among molecules[96,33].

After preheating the composite, there is a time lapse between removing it from the heating, placing it in the cavity, contouring it and light curing it. Lohbauer et al. stated that RBCs temperature rapidly falls to physiological level on removal from heating device[78]. Polymerisation is an exothermic process and the heat released accelerates the photoreaction. The heat generated decreases the viscosity of the material by increasing the system temperature and increases molecular mobility and postpones diffusion-controlled propagation known as auto deceleration, hence increases degree of conversion[33]. During cooling the polymer formation, there is an excessive heat loss which deprives the system of its energy necessary for chain propagation. The gel phase might decreased leading to auto deceleration and early vitrification decreasing degree of conversion. The polymerisation reaction is influenced by several factors such as environmental temperature, curing time, filler loading and nature of filler[97].

The minimum DC% acceptable for restorative material at occlusal layers is 55% according to Soares et al[98]. In this study DC% were assessed on top and bottom surfaces post 24 hour curing by FTIR device. All the three bulk fill composites exhibited a DC% of more than 55% under all parameters. FTIR spectroscopy allows for direct detection of amount of unreacted C=C in the matrix.

DC% of a composite material is very pivotal in determining its mechanical properties such as strength modulus, hardness, solubility and biocompatibility[38]. Additionally, evaluating degree of conversion during polymerisation is considered essential in understanding

polymerisation kinetics using different curing techniques. The DC% values are known to be dependent on chemical structure of dimethacrylate monomer and photo initiator concentration and polymerisation factors[99]. Bulk of the post irradiation polymerisation is known to occur in the first few minutes or one hour after the removal the curing guide tip[100] with a gradual increase up to a maximum of 24 hour after irradiation[101]. The two main properties of a monomer which affects the DC% are the initial monomer viscosity and flexibility of its chemical structure[102].

DC% of various monomer system increase in the order of:

Bis-GMA< Bis-EMA< UDMA<TEGDMA[103]. Polymerisation stress can be efficaciously minimised by decreasing the rate of polymerisation either by controlling the initial curing light intensity or by modifying the polymerisation inhibitor concentration[104].

Light curing a RBC is a complicated process as its depth of cure is influenced by composition, layer thickness, irradiance, curing time and many other factors[69] and an incomplete curing leads to early wear of the restoration leading to its failure and the presence of free monomers may cause health hazards[8].

For sufficient polymerisation, a traditional composite should receive a radiant exposure of 16-24J/cm^2 range. Radiant exposure, also known as energy density is calculated by multiplying the irradiance level of the light cure unit by its duration. Curing time is dependent on the irradiance value of the Light cure unit. Selig et al. stated that an exposure duration of 10 seconds and above gave an adequate DC[105]. Hence increasing the exposure duration resulting in high radiant exposure. In this study I-LED light cure unit from Woodpecker has been used. The specifications of the unit are that it cures 2mm of resin in 1 second (mode P1). It has got maximum intensity of 3000mw/CM2 and operates on twin mode P1 (High intensity mode); P2 (normal intensity). It comes along with a digital intensity meter, with an unbreakable probe

with 360-degree rotation. The samples were irradiated under high and low intensity for 1 and 3 seconds respectively.

Microhardness and depth of cure are the most important properties of the RBCs which play a role in comparing and assessing the characteristic of a dental restorative material. Microhardness is used as an indirect measure for degree of conversion which also determines the efficiency of curing light. The improper monomer conversion and limited depth of cure are the issues associated with photo polymerisation of composites[106]. Low microhardness is also associated with poor wear resistance leading to failure of restoration[107].

Quite a few studies have been conducted assessing the microhardness of bulk fill composites. There arose a concern when the studies mostly showed low microhardness for bulk fill composites especially SDR and Filtek bulk fill flow[108].Leprince also stated that SDR and Filtek bulk fill showed very low microhardness[99].

Hence with the previous studies showing the effect of preheating and high intensity having favourable results on microhardness and degree of conversion in conventional composites, this study was formulated to assess the effect of preheating and increased curing intensity in the bulk fill composites.

In this study the Vickers microhardness for both the top and bottom surface was calculated after post 24hour curing. The microhardness at top surface was more than that of bottom surface, this may be because of decrease in the light reaching the bottom surface as it travels through the composite or due to scattering of light through the filler particles[109]. All the three types of bulk fill composites used in this study showed a statistically significant increase in the microhardness at the top and bottom surface on preheating the composite and its relation with the high and low intensity was statistically not significant.

The shade of the bulk fill composite is known to affect the microhardness of the restorative material. Thome et al. state that the resins with lighter shade exhibit higher microhardness values than that of darker shade, as the latter requires more exposure time and thinner increments[110]. In this study, A2 shade for all the composites have been used to standardize the sample and decrease its influence on polymerisation.

Limitations of this study are, that this is an in vitro study and the specimens were irradiated in "occlusal" direction. In clinical cases, there is a possibility to irradiate the composite resin restoration from a buccal or lingual aspect as well, to enhance the DC. Though indirect polymerization of the RBCs through a substance (Teflon mould) reduces the radiant exposure delivered to the material significantly, the tooth absorbs the energy originated from the photocuring device and also, the DC values do not provide information about the mechanical properties or the development of contraction stress in the materials in response to recommended or doubled duration exposures and pre-heating. Although, direct relation exists between hardness and DC, additional mechanical testing, like three-point bending is planned to get more information about the relation of DC and mechanical characteristics. Finally, further investigations are required to clarify the negative effect of pre-heating on the DC of flowable RBCs.

CONCLUSION

- The degree of conversion and microhardness values were significantly higher on top surface on the sample that that measured at the bottom.
- The degree of conversion for the bulk fill composites after preheating was better than that of the value obtained for bulk fill composites cured at room temperature.
- The degree of conversion for the bulk fill composites after increasing the curing light intensity was better than that of the value obtained for bulk fill composites cured with normal light intensity.
- Among the three bulk fill composites, the DC values were maximum for Palfique and SDR, which achieved similar results to that of lab processed indirect composites.
- The Vickers microhardness for the bulk fill composites after preheating them was better than that of the value obtained for bulk fill composites cured at room temperature.
- The Vickers microhardness for the bulk fill composites after increasing the curing light intensity was better than that of the value obtained for bulk fill composites cured with normal light intensity.
- Among the three bulk fill composites, the microhardness values were maximum for SDR.

Hence within the limitations of the study, it was concluded that in a clinical situation, where deep cavities are encountered or post endodontic restorations are required, using bulk fill composites after preheating and/or using height curing light intensity increases the degree of conversion and Vickers microhardness values.

SUMMARY

This study was carried out with an aim to assess and compare the effect of preheating and variant radiant exposure on the degree of conversion and microhardness of the bulk fill composites.

3 different bulk fill composites were selected. Twenty-eight composite discs of 4mm thickness were prepared for each bulk fill composite. According to the method of polymerisation four groups were formed with n=7.

Group1: high intensity, no preheating the composite

Group 2: high intensity, preheating the composite

Group 3: normal intensity, no preheating the composite

Group 4: normal intensity, preheating the composite

All the samples were cured according to their respective parameters and degree of conversion and microhardness were determined by using FTIR and Vickers's microhardness test.

A multivariate ANOVA (MANOVA) was done with 3 independent variables namely, intensity of curing light, preheating the composite and type of composite and 4 dependent variables namely Microhardness on top and bottom surface and Degree of conversion on top and bottom surface of the composite samples. The combined values of dependent variables were used to assess the characteristic of composite.

The three types of bulk fill composites achieved significant microhardness and degree of conversion with the high intensity and preheating parameters.

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This Ethical Committee has reviewed the research Protocol submitted by Dr. T. Vaibhavi, Post Graduate Student, Department of Conservative Dentistry and Endodontics, under the title "Effect of preheating and radiant exposure on degree of conversion and microhardness of bulk fill composites: An invitro study". Ref no.: 2017-MDS-BrIV-10/APDCH under the guidance of Dr. B. Hema Sathya for consideration of approval to proceed with the study.

This Committee has discussed about the Material being involved with the study, the Qualification of the investigator, the present norms and recommendations from the Clinical Research Scientific body and comes to a conclusion that this Research protocol fulfils the Specific requirements and the Committee authorizes the proposal.

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