THE COMPARATIVE EVALUATION OF THE DEGREE OF CONVERSION, MARGINAL ADAPTATION AND SURFACE HARDNESS OF FOUR DIFFERENT COMPOSITE RESINS BEFORE AND AFTER PREHEATING – AN IN VITRO STUDY

Dissertation submitted to

THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY

In partial fulfillment for the Degree of

MASTER OF DENTAL SURGERY

BRANCH IV – CONSERVATIVE DENTISTRYAND ENDODONTICS

2017-2020

RAJAS DENTAL COLLEGE AND HOSPITAL

THIRURAJAPURAM, KAVALKINARU JN – 627 105, TIRUNELVELI

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Affiliated to The Tamil Nadu Dr. M.G.R. Medical University, Chennai.

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This is to certify that this dissertation entitled **"The comparative evaluation of the degree of conversion, marginal adaptation and surface hardness of four different composite resins before and after preheating – an *in vitro* study"** *is a bonafide research work done by* **Dr. Mohanapriya R** *under my guidance during her postgraduate study period between* ***2017– 2020.***

This Dissertation is submitted to **THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY,** *in partial fulfillment for the degree of* **MASTER OF DENTAL SURGERY** in **CONSERVATIVE DENTISTRY AND ENDODONTICS - BRANCH IV .** *It has not been submitted partially or fully for the award of any other degree or diploma.*

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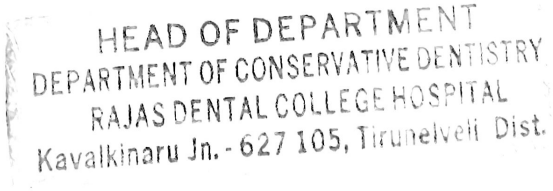

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LIST OF ABBREVIATIONS USED

ABBREVIATION	WORD EXPLANATION
Bis - GMA	Bis-phenol A diglycidyl ether dimethacrylate
TEGDMA	Triethylene Glycol dimethacrylate
C - factor	Configuration factor
DC	Degree of Conversion
FTIR	Fourier Transform Infrared Spectroscopy
SH	Surface Hardness
VHN	Vicker Hardness Number
SEM	Scanning Electron Microscope
CEJ	Cemento Enamel Junction
LED	Light Emitting Diode
SPSS	Statistical Package for Social Sciences
p - value	Probability value
SD	Standard Deviation
ANOVA	Analysis of Variance

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ABSTRACT

Background

Resin composites as direct posterior restorative material was associated with the polymerization contraction and microleakage. Different methods have been introduced to overcome these drawbacks by increasing the degree of monomer conversion and to minimize the polymerization shrinkage. Composite preheating is an innovative method to improve the handling and physical properties. So this study was done to evaluate the effect of prepolymerization warming of different composites on the degree of conversion and the marginal adaptation.

Materials and methods

Preheating of composites was done with the heating device and categorized accordingly as Group 1 - Delta composite warmer (preheating at 61˚C) and Group 2 – Room temperature composites (no preheating). Four resin composites were taken as Group 1A & 2 A – Bulk fill nanohybrid, Group 1B & 2B – Nanofill, Group 1C & 2C – Ormocer based composite and Group 1D & 2D – Microhybrid composites to assess the mechanical properties before and after heating of the composites.. For evaluation of surface hardness and degree of conversion, split mold of length 5 mm × width 5 mm × height 3 mm was taken. 160 composite blocks were prepared based on four composite resins for both preheated and control groups (n = 80). For preheating, composites were heated at 61˚C in the composite warmer and then light cured in a split mold. Prepared composite blocks (n= 80) were powdered to analyze the degree of monomer conversion to polymer using FTIR spectroscopy . Surface hardness was determined for 80 composite blocks with vicker microhardness tester . To analyze axial adaptation, class II cavities was prepared in 80 premolars and restored with respective composites. Samples were sectioned and analysed using

SEM at 200x magnification and marginal gap width was measured using Image analysis software.

Statistical Analysis

Data was entered in Statistical Package for Social Sciences version 21 Software for Windows. Data were executed in the form of mean and standard deviation and were analyzed with one way analysis of variance and Kruskal – Wallis tests.

Results

The results indicated that preheated composite group showed a higher degree of conversion and surface hardness than the room temperature composite group. Statistically significant differences was observed between Bulk fill nanohybrid, Ormocer, Nanofill and Microhybrid composites in terms of degree of conversion and surface hardness ($p < 0.05$). For degree of conversion, the highest mean percentage value was observed with Ormocer (74.35%) and the lowest mean percentage value was observed with bulk fill nanohybrid (44.44%). For surface hardness, the highest mean hardness value was observed with nanofill (110.58 VHN) and the lowest mean hardness value was with microhybrid (58.32 VHN).

For internal marginal adaptation, no statistically significant results were found. But the frequency of gap formation was comparatively higher in preheated group with increased MQ4 scores.

Conclusion

Within the limitations of the present study, it could be concluded that

1. Preheated group showed higher degree of conversion and surface hardness values with ormocer and nanofill ranked with the highest mean values respectively.

2. Preheated group showed poor internal marginal adaptation with increased frequency of gap formation.

Keywords

Degree of conversion, Fourier transform infrared spectroscopy, Surface hardness, internal marginal adaptation, Ormocer, Nanofill, Microhybrid, Bulk fill nanohybrid.

INTRODUCTION

The introduction of resin based composites dates back to 1960s. Due to increasing esthetic demands among the patient and better performance of these materials, many clinicians choose composites as a posterior restorative material. Eventhough these materials have excellent esthetic properties, it has its own negative characteristics like polymerization shrinkage, poor marginal adaptation and lack of wear resistance.

Posterior composite restorations undergo polymerization shrinkage that results in bulk contraction of the material. Dimethacrylate based composites accounts for 2 - 6% volumetric shrinkage [1]. Davidson *et al* investigated about the flow of composite materials in its early stage of setting, where more amount of polymerization contraction occur. Composites during its setting reaction, polymerization shrinkage will be compensated by the stress that induces the flow of the material [2]. Composite strain (flow) is affected by the confinement of the bonded material to the tooth that leads to manifestation of shrinkage itself as stress.

Polymerization contraction stress may cause tooth deformation, adhesive failure, might cause microcracking of the restorative material [3,4]. Various factors responsible for shrinkage stress include filler content, degree of conversion, elastic modulus of the material, water sorption and configuration factor. Polymerization shrinkage can be minimized by the presence of higher filler loading. It also improves the wear resistance and mechanical properties of the material.

There exist a significant correlation between the degree of conversion and shrinkage. A decrease in degree of conversion might result in inferior mechanical properties but lower the shrinkage stress. Ferracane *et al* evaluated about the effect of different resin compositions like the amount of diluents concentration

on the degree of conversion and mechanical properties of composite resin based materials. They stated that resins with lowered viscosity show increased degree of conversion[5].

There is a positive relationship between the stiffness of the material and shrinkage stress. Rigid material with high elastic modulus has shown the highest stress values. As the setting reaction of composite resin proceeds, it causes increase in elastic modulus of the material [6].

Development of shrinkage stress depends upon the cavity configuration (C – factor). Flat and shallow cavity preparations favours the composite to flow freely in the early setting stage. It minimizes the shrinkage forces from creating the stress and favours the formation of strong composite - dentin bond. Feilzer described about the concept of configuration factor, usually less than one for the flat and shallow cavity designs. In class II designs, C – factor found to be two. When C – value increases, it result in restricted flow capacity leads to more shrinkage stress development [7].

It is generally agreed that resin based composites has a negative characteristics of polymerization shrinkage when used as posterior restorative materials. Factors involved to minimize shrinkage stress in direct posterior restorations depends on the choice of the material, choice of the restorative technique and polymerization strategies [8].

Flowable composites usually have reduced filler content with lower elastic modulus. Thus the reduced viscosity in flowable composites associated with inferior mechanical properties and increased shrinkage values. Eventhough the increased setting shrinkage of flowable composites can create heavily pre - stressed

interface, their reduced elastic modulus would produce less stress. Thus the low – viscosity composite functions by partially reduce the shrinkage stress. It appears that a thin film thickness of composites generates less contraction [9].

Researchers have focussed on the significance of preheating the composites and its influence on the material properties. Increasing the temperature upto 60ºc might increase the degree of monomer conversion into polymer. As the preheating enhances the molecular mobility and increases the activity of reacted species, but this reaction is self – limited due to rapid formation of more cross linked polymer with improved mechanical properties.

And also, heating the composites reduce its film thickness and viscosity that may partially relieve contraction stress and improve its marginal adaptation without hampering its mechanical properties [10].

The choice of restorative material and its placement technique has a role in management of shrinkage stress in direct posterior restorations. It is known that layering technique reduces contraction stress as it involves use of 2mm increment of material. Bulk placement technique involves placement of restorative material in 4mm thickness instead of 2mm incremental layer. However, it is unclear if bulk fill technique reduces contraction stress.

So this study was conducted to evaluate the effect of prewarming on the degree of conversion, marginal adaptation and surface hardness of four different composite resins.

AIM AND OBJECTIVES

AIM:

To evaluate the effect of prepolymerization heating on the degree of conversion, marginal adaptation and surface hardness of four different composites namely bulk fill nanohybrid, nanofill , ormocer and microhybrid composites.

OBJECTIVES:

1. To compare and evaluate the degree of monomer conversion among the four composites namely bulk fill nanohybrid, nanofill, ormocer and microhybrid composites after preheating with FTIR (Fourier Transform Infrared Spectroscopy)

2. To determine the surface hardness among the four composites namely bulk fill nanohybrid, nanofill , ormocer and microhybrid composites after preheating with Vicker hardness tester

3. To assess the axial margin adapatation among the four composites namely bulk fill nanohybrid, nanofill, ormocer and microhybrid composites in class II restorations after preheating with Scanning Electron Microscope analysis.

NULL HYPOTHESES:

There exist no significant differences among the four different composite resins namely bulkfill nanohybrid, nanofill, ormocer and microhybrid composites in terms of degree of conversion, marginal adaptation and surface hardness before and after preheating the composites.

REVIEW OF LITERATURE

It is desirable for a dental restorative resin to convert all of its monomer to polymer during the polymerization reaction. However, with Bis-GMA-based resins there is always a significant concentration of unreacted carbon double bonds remaining in the resin when it is cured at or near the oral temperature. This is believed to be due to limitations on the mobility of reactive species imposed by the rapid formation of a cross-linked polymeric network.

De Almida *et al* in 2018 [11] investigated about the influence of preheating and post-curing methods on diametral tensile strength (DTS), flexural strength (FS), knoop microhardness (KHN), and degree of conversion (DC) of an experimental fiber-reinforced composite. He stated that preheating promoted significantly higher values of FS and KHN. DC was not affected by both methods. Preheating and post-curing methods can be used to improve some mechanical properties of fiber reinforced composite resins but degree of conversion remains unaffected.

Yang *et al* in 2016 [12] evaluated the effects of preheated composite at different temperatures on microleakage. A total of 60 extracted non-carious human premolars were collected and class 1 cavity (1.5x 4x 3mm) was prepared in each and were randomly divided into three groups. Group 1 (n=20) was filled with microhybrid resin composite (Heraeus Charisma Smile) at room temperature. Group 2 (n=20) was filled with the same resin composite which was preheated to 50°C and Group 3 (n=20) was filled with resin composite preheated to 60°C. Using confocal microscope, microleakage was assessed using dye penetration method. The author concluded that sample with preheated composite restoration at 50°C showed an intact tooth-restoration interface with no micro leakage. However, the preheated composite at 60°C showed large amount of microleakage.

Par *et al* in 2015 [13]investigated about the "Raman Spectroscopic determination of Degree of monomer Conversion of Bulk-Fill Composites after 24 hours post polymerization". The author concluded that the Degree of conversion (DC) affects various physical properties and biocompatibility of a composite restoration. Adequate DC is especially important for bulk-fill materials, which are designed for placement in thick layers.

Dos Sonto *et al* in 2011 [14]evaluated microleakage in Class II cavities restored with dental composite and varying light-curing units and the temperature of the composite when subjected to a thermocycling test. Ninety cavities were prepared on the proximal surfaces of bovine teeth and randomly divided according to the light-curing mode (QTH-420 mW/cm2, LED 2nd generation-1100 mW/cm2, or LED 3rd generation-700 mW/cm2) and temperature of the resin composite (23°C, 54°C and 60°C). With the obtained result he concluded that the group preheated to 60°C showed no difference when compared to the group heated to 23°C. Preheating the resin composite (54°C and 60°C) did not improve the marginal seal when high-irradiance LED was used; however, it decreased the microleakage when a QTH with low irradiance was used.

Lucey *et al* in 2010 [15] evaluated about the "Effect of pre-warming the composite resin on the viscosity and microhardness of the material". The author stated that pre-heated composite resulted in significantly reduced viscosity and increased surface hardness compared to room temperature composite.

Wagner *et al* in 2008 [16] investigated about the "Effect of pre-heating resin composite on restoration microleakage". The results of this study indicate that preheating composites can improve adaptation of resin composites to tooth structure.

This technique significantly reduced microleakage. However, delaying light curing of the preheated composite after placement appears to be counterproductive and diminishes the positive effects from the preheating treatment.

The placement of composite at the elevated temperatures directly into a cavity preparation raises concern that it may impose detrimental temperature levels within the pulp, with the potential for iatrogenic damage.

Daronch *et al* in 2006 evaluated about "Clinically relevant issues related to preheating composites". The author stated that the temperature to which the composite is subjected during pre-heating in the heating device is between 50º c and 70º C. Composite temperature quickly decreases once a syringe or compuleis removed from the heating device and is injected into a tooth preparation.

Rueggeberg FA *et al* in 2006 [17] investigated about the "Effect of temperature on unpolymerized composite resin film thickness". The objective of this study was to compare the film thickness of a variety of commercial composite resins heated prior to light polymerization. The author concluded that Preheating conventional composite resin yields lower film thickness for some products, but flow cannot be attributed to composite resin classification, filler content, or shape. Preheated composite resin thickness was greater than that of all flowable composites.

Knight J and Norrington D in 2006 [18] studied about the "Effect of temperature on the flow properties of resin composite". He concluded that composite film thickness decreased with decreasing filler content, and increasing temperature.

Improving the adaptation of resin composites during placement is necessary to increase durability and reduce microleakage. Flowable resin liners have

been introduced to improve adaptation in composite restorations. An alternative way to improve sealing is to use conventional composites that have been heated to lower their viscosity.

Daronch *et al*in 2005 [19] evaluated about the "Monomer conversion of pre- heated composite". He stated that Monomer conversion increased significantly when compositewas pre-heated, compared to room temperature composite.

Jeffrey W. Stansbury *et al* in 2005 [20] investigated about the degree of conversion dependent contraction stress and strain in an attempt to control shrinkage stress and strain in dental composite restoratives. The two most widely used techniques to assess the extent of polymerization in dental composites have been the physical determination of surface hardness and the direct chemical analysis of conversion by mid-infrared (mid-IR) spectroscopy have been explained. Material approaches to reduced shrinkage stress and strain including changes in monomer structure or chemistry and changes in fillers or use of additives were discussed.

The placement of composite resin is affected by the shrinkage during polymerization of these materials. The resulting contraction stress can create cracks within the composite restoration, the tooth or at the restorative – dentin interface that lead to decreased clinical performance and poor esthetics.

Peutzfeldt A *et al* in 2004 [21] investigated about the polymer structure of dental resinous materials. He stated that extent of polymerization of Bis – GMA based materials depend upon monomer and filler composition, initiator system, and light-curing procedure. Polymers having similar conversion values may have different crosslink density. Thus, conversion alone may not prove to be a predictor of restoration performance.

Lovell *et al* in 2001 [22] evaluated about "The effect of polymerization rate on the mechanical properties of composite resins". This study investigated the effect of polymerization rate on the mechanical properties of dimethacrylate resin percentage variables using 75 wt% bis – GMA and 25 wt% TEGDMA. The degree of conversion rate of the samples were analysed by near-infrared spectroscopy. Samples were polymerized with UV and visible light systems. This study conclude that inspite of the method or rate of polymerization, more efficient cross-linked resins, such as bis-GMA and TEGDMA, show favourable polymer network structure and properties as a result of double bond conversion.

Lovell *et al* in 2001 [23] investigated the "Understanding of kinetics and network formation of Dimethacrylate dental resins". His work investigated the copolymerization behavior of bis – GMA and TEGDMA. Near IR – spectroscopy was shown to be a valuable tool for monitoring the kinetics of thick monomer samples that can be subsequently used for material testing. He stated that Increasing conversion produces higher surface hardness, greater flexural strength and rigidity, improved fracture toughness and tensile strength.

Tarle Z *et al* in 1995 [24] evaluated about the "Correlation between degree of conversion and light transmission through resin composite samples". The author stated that there are some relevant methods to determine the degree of monomer to polymer conversion of composite resin specimens like FTIR spectroscopy, Laser Raman spectroscopy, Electron Spin Resonance (ESR), Infrared Spectroscopy (IR), Dynamic Mechanical Thermal Analysis (DMTA), Attenuated Total Reflection (ATR).

Ferracane JL in 1985 [25] evaluated the "Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins". The purpose of this study was to evaluate the correlation between the Knoop hardness and the degree of monomer conversion, using IR analysis, for dental restorative resins. He concluded that there exist a positive correlation between the hardness and the increased degree of conversion. However, degree of conversion cannot be determined by using an relative hardness number. Mechanical properties are very dependent upon polymer network formation, which is not equivalent to conversion.

Cook WD *et al* in 1984 [26] described a review on "Resin-based restorative materials". The purpose of this paper is to review the development of composite resins in dentistry and discuss their properties. He stated that polymerization of dimethacrylate- based materials exhibits incomplete conversion of double- bonds (from 50 to 75%), leaving a significant proportion of methacrylate groups unreacted.

Bausch *et al* in 1981 [27]evaluated about the effect of temperature on the physical properties of composite resins. In this study, composite heating was done at various temperatures to evaluate the tensile strength and surface hardness. The author stated that mechanical properties were improved by increasing the temperature upto 60ºc and it results in highly cross linked polymer. Polymerization temperature also affects monomer conversion and, thus, polymer properties. And also, preheating of composite resins for clinical use was suggested as a means to improve mechanical properties and to reduce microleakage.

MATERIALS AND METHODS

MATERIALS USED:

1. Split mold
2. Extracted human maxillary premolars (n = 80)
3. Filtek Z350 XT Universal Nanofill Composite (3M ESPE AG, Brazil)
4. Tetric N Ceram bulk fill nanohybrid composite (Ivoclar Vivadent AG, Liechtenstein, Western Europe)
5. Admira (Ormocer based composite, VOCO GmBH, Cuxhaven, Germany)
6. Restofill microhybrid composite (Anabond Stedman Pharmaceuticals, Tamil Nadu, India)

INSTRUMENTS AND EQUIPMENTS USED:

1. No. 4 Round Diamond Bur (Dia – burs, Mani, Inc., Japan)
2. SF – 41diamond bur (Dia – burs, Mani, Inc., Japan)
3. NSK Pana Air FX hand piece (NSK Corporation, Japan)
4. Palodent V3 sectional matrix system (Dentsply Sirona, USA)
5. Ivoclar Bluephase 20i G2 LED curing light (Ivoclar Vivadent AG, Liechtenstein, Western Europe)
6. Diamond Disc of 0.10 thickness (Strauss & Co, Israel)
7. Typhodont maxillary jaw model (Nissin Inc, Japan)
8. Fourier Transform Infrared Spectroscopy (Shimadzu IR Tracer- 100 Infrared spectroscopy, Europe)

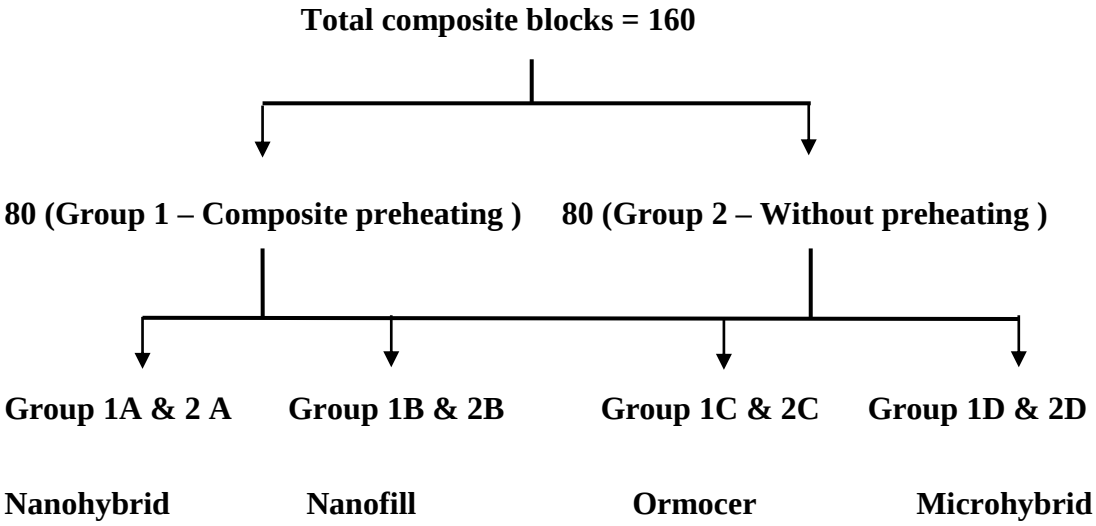
9. Vicker surface hardness tester (ASTM E 92 Standard calibrator, USA)

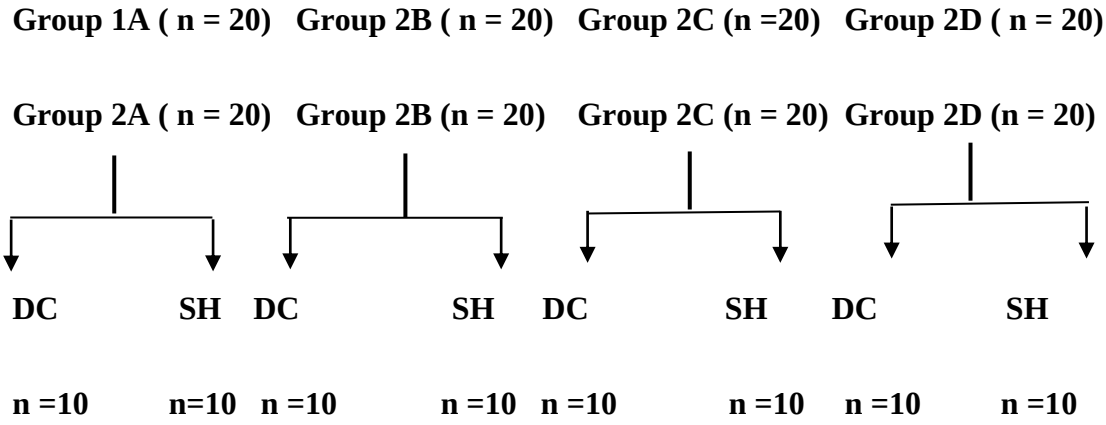
10. Scanning Electron Microscope (Zeiss DSM 962: Oberkochen, Germany)

SAMPLE ALLOCATION:

Sample grouping for degree of conversion and surface hardness

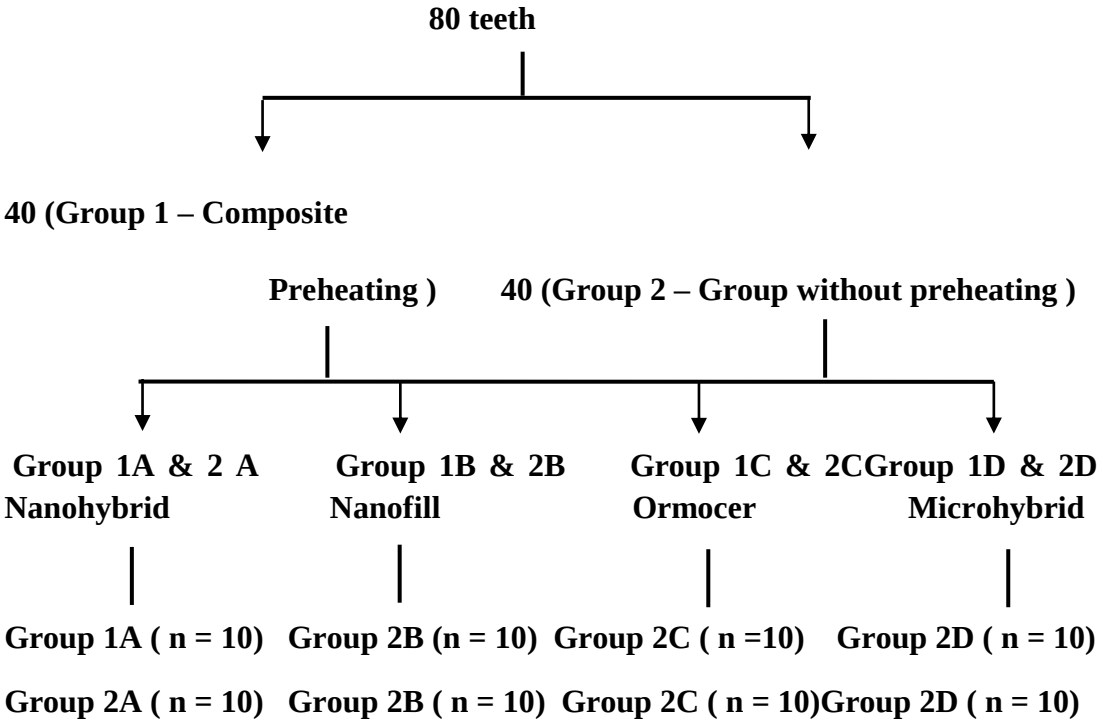
in group 1 and group 2





Sample grouping for internal marginal adaptation

in group 1 and group 2



DEGREE OF CONVERSION AND SURFACE HARDNESS ANALYSIS

160 composite blocks were prepared using a stainless steel split mold of length 5 mm × width 5 mm × height 3 mm (fig 1.1). Four different composite materials were used in the study (Fig 1.2). 160 Composite blocks were divided into 80 blocks based on the preheating method as Group 1 – Delta composite warmer (Fig 1.3) and Group 2 - group without heating the composites. In group 1, composites were preheated using composite warmer (Delta company, India) that elevates composite temperature to 61˚c (Fig 1.4 and 1.5). The mean time between removing composite from the device and light polymerization was approximately 40

seconds for all tests. In control group, the specimen preparation and testing was performed at controlled room - temperature (25º C).

Samples were further divided into 20 composite blocks according to the four different composite materials used. Group 1A [n = 20] &2 A [n = 20] - bulk fill nanohybrid, Group 1B [n = 20] & 2B [n = 20]- nanofill, Group 1C & 2C - ormocer and Group 1D & 2D – microhybrid composites (Fig 2.3 – 2.6). Degree of conversion (n = 10) and surface hardness (n = 10) were determined for each respective composites in both groups 1 & 2.

SAMPLE PREPARATION:

The split mold was placed over the glass plate, and each composite material was restored with incremental layering technique of 2 mm thickness (Fig 2.1). Each layer was light polymerised for 20 seconds using LED curing unit with a light intensity of 800 to1000 Mw/sq cm (Bluephase) (Fig 2.2). For group 1A & 2A, material was placed bulk inside the mold and light cured. Mylar strip was placed over the last increment and cured to minimise the formation of oxygen inhibited layer and to create a smooth surface. Samples were stored dry in dark container at room temperature for 24 hours before testing.

PROCEDURE FOR DEGREE OF CONVERSION ANALYSIS:

The FTIR analysis was carried out at the International Research Centre, Kalasalingam Academy of Research Institute, Tamil Nadu, India. Fourier transform infrared spectroscopy IR Tracer 100 - FTIR was used to evaluate the degree of conversion. Each of the polymerized samples (n =10) of each composite in both group – 1 and group - 2 was grinded into a fine powder with a mortar and

pestle (Fig 3.1). 50 µg of the powder was mixed with 5 mg of potassium bromide powder (Fig 3.2) and pressed to produce a thin disc (Fig 3.3), which was placed in a specimen holder and transferred to the spectrophotometer (Fig 3.4 and 3.5). The absorbance peaks were recorded using the absorbance mode of FTIR under the following conditions: 32 scans, over a wave length of 400 - 4000 cm^{-1} and a resolution of 4 cm^{-1}.

Unpolymerized specimens of each composite resin in both group – 1 and group - 2 were smeared onto thin potassium bromide discs, placed into a cell holder in spectrophotometer, and then a spectrum was obtained with the same parameters as for the polymerized specimens.

For dimethacrylate based composites, Degree of conversion was analysed by comparing the relative absorbance intensities of aliphatic carbon double bond peak at 1638 cm^{-1} against an internal standard peak of aromatic carbon bond at 1608 cm -1 during polymerization, in relation to the uncured material [28] (Fig 3.6, 3.7, 3.9). For Ormocer based composite, the internal standard was kept at the aromatic C – C stretching vibrations at the 1592 cm^{-1} (Fig 3.8)

DC % for each specimen was calculated using the following equation:

Degree of % conversion = { 1 - R cured / R uncured } × 100 %

where " R " is the ratio of absorbance peak intensities of the 1637cm^{-1} and 1608 cm^{-1}in the spectra of the dimethacrylate- based composites, or the 1637cm^{-1} and 1592 cm^{-1} absorbance intensities in the spectra of the ormocer-based composite.

EVALUATION OF VICKER HARDNESS:

The test was carried out at the Kalasalingam Academy of Research Institute, Tamil Nadu, India. Surface hardness was assessed on the top surfaces of each sample using a microhardness tester with a vicker pyramidal indenter under a 200g load for 30 seconds (Fig 4.1). Three indentations were carried out on the surface of each sample, 1mm away from each other. The average of 3 values was calculated as VHN (Vicker hardness number) value for each sample.

INTERNAL MARGINAL ADAPTATION ANALYSIS

80 extracted non – carious human maxillary premolars were collected that were extracted for orthodontic purposes (Fig 5.1). These teeth were stored in sodium azide solution until the experiment to prevent dehydration. Class II cavities were prepared on the proximal surface of all maxillary premolars, with the dimensions of 4 mm buccolingual width, 2 mm mesiodistal depth with the gingival margin at the cementoenamel junction (CEJ) (Fig 5.2). Cavosurface margin bevels were not placed. Margins were smoothened using hand instrument.

Then, the teeth were randomly divided into two groups of 40 each. Group 1 – Delta composite warmer and Group 2 - group without heating the composites. Samples were further divided into 10 composite blocks according to the four different composite materials used.

RESTORATIVE PROCEDURES:

Using typhodont jaw model, two teeth were mounted together in contact with each other using modelling wax. Sectional matrix was placed between the teeth to provide proper restorative material adaptation (Fig 5.3).

In all the groups total of 80 cavities, were etched with 37% phosphoric acid gel for 15 S, rinsed with water for 10 S and wet dried with cotton pellet for 2 seconds. Bonding agent (Tetric N - Bond, Ivoclar Vivadent) were applied onto the cavity surface, gently air dried and polymerized for 10 seconds, using LED curing unit with a light intensity of 800 to1000 mW/ cm2 (Bluephase 20i G2 LED curing light) (Fig 5.4).

The cavities were restored with the respective composite resins in 2mm increments in group 1B & 2B, 1C & 2C and 1D & 2D containing bulk fill nanohybrid, nanofill, ormocer and microhybrid composites. Each increment was light cured for 20 seconds. In bulk fill composites group 1A & 2A, bulk placement technique involve restoration in one increment to fill the entire proximal box with 4mm thickness and light cured for 11 seconds according to manufacturer's instructions (Fig 5.5). The matrix retainer and the adjacent tooth that were in contact with the restoration were removed after the restorative procedures. The same adjacent tooth was reused for all the samples to maintain contact.

SEM sample preparation:

Samples were sectioned using hard tissue – microtome longitudinally in the mesiodistal direction through the centre of the restoration. Buccal half of the sectioned teeth were taken uniformly in all the 80 samples. The tooth – restoration interface was analysed with Scanning Electron Microscope and images were captured at 200x magnification (Fig 6.1, 6.2 and 6.3). SEM evaluation was carried out at the International Research Centre, Kalasalingam Academy of Research Institute, Tamil Nadu, India. A criteria by Blunck and Zaslansky was followed to

evaluate the micromorphological qualitative assessment of the tooth – restorative interface [29].

MQ1 - Margin hardly visible; No or slight marginal irregularities; No gap

MQ2 - No gap but severe marginal irregularities

MQ3 - Gap visible (hairline crack up to 2 micrometer); No marginal irregularities

MQ4 - Severe gap (>2 micrometer); severe marginal irregularities

Internal margin micromorphology was assessed using Image J software to trace the complete width of the gap in three areas along the axial wall (Fig 6.4). Width of the gap was determined for MQ3 and MQ4 criteria (Fig 6.5 and 6.6). Mean average of the gap width in three areas were taken as frequency of scores for each composite in both the groups. Marginal irregularities was evaluated for MQ1 and MQ2 criteria (Fig 6.7 and 6.8). Margins were assessed twice by the same examiner to check reliability.

COLOUR PLATES

Fig 1.1 Split mold of length 5 mm × width 5 mm × height 3 mm

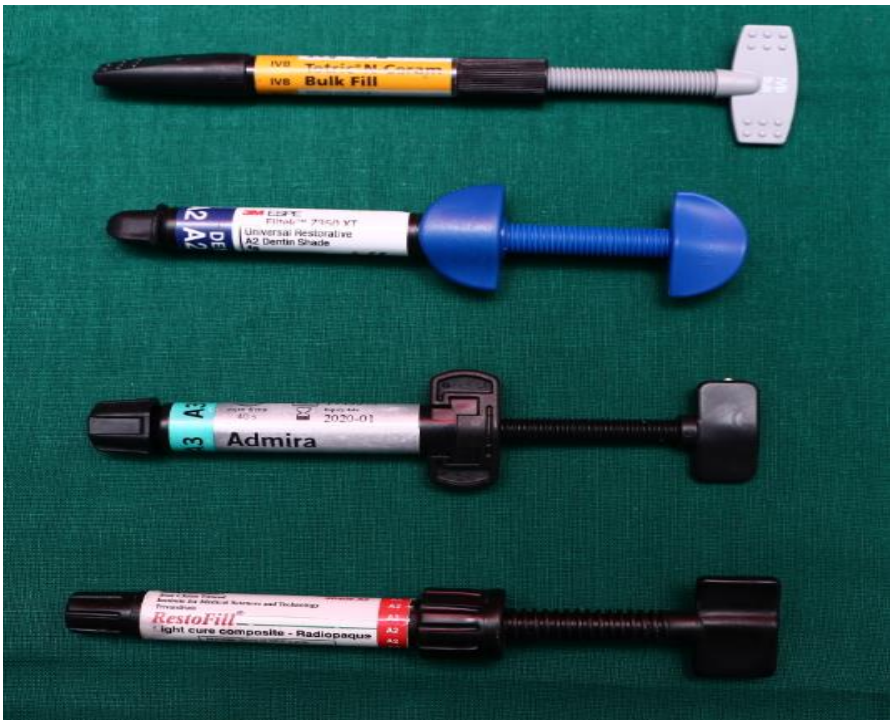


Fig 1.2: Tetric N Ceram bulk fill, Filtek nanofill, Admira (Ormocer), Restofill microhybrid composites

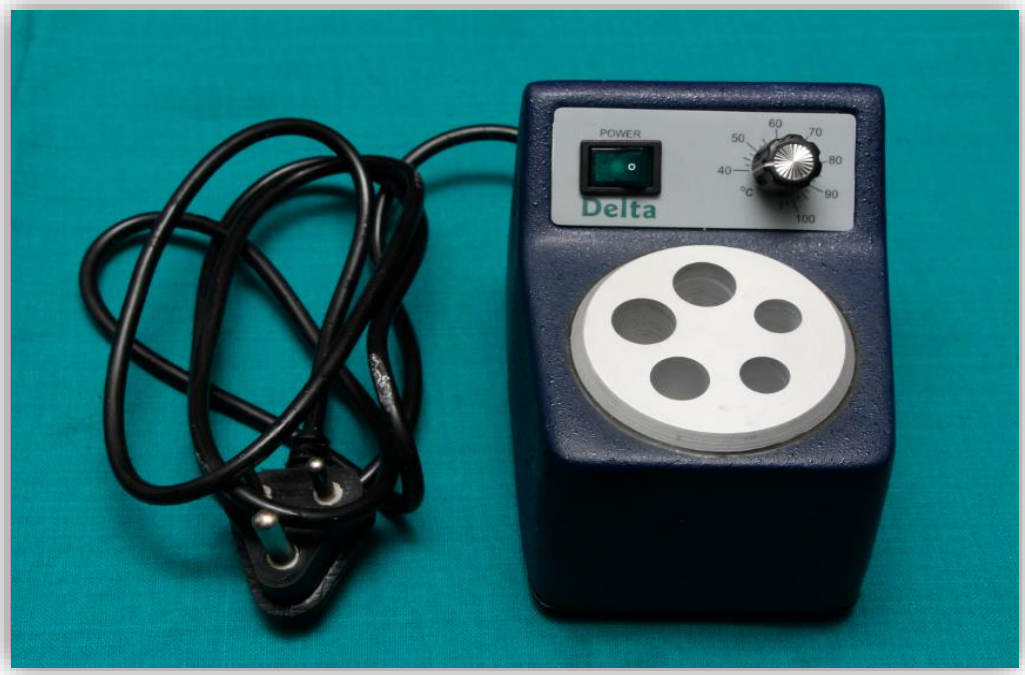


Fig 1.3 Composite warmer (Delta product)

Fig 1.4 Composite warmer at 61˚ c

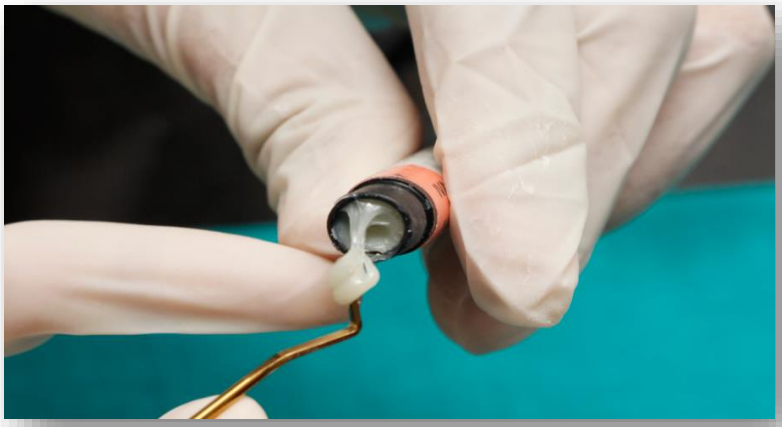


Fig 1.5 Lesser viscosity of composite after preheating in warmer

2.PREPARATION OF COMPOSITE BLOCKS

Fig 2.1 Placement of composite into the mold

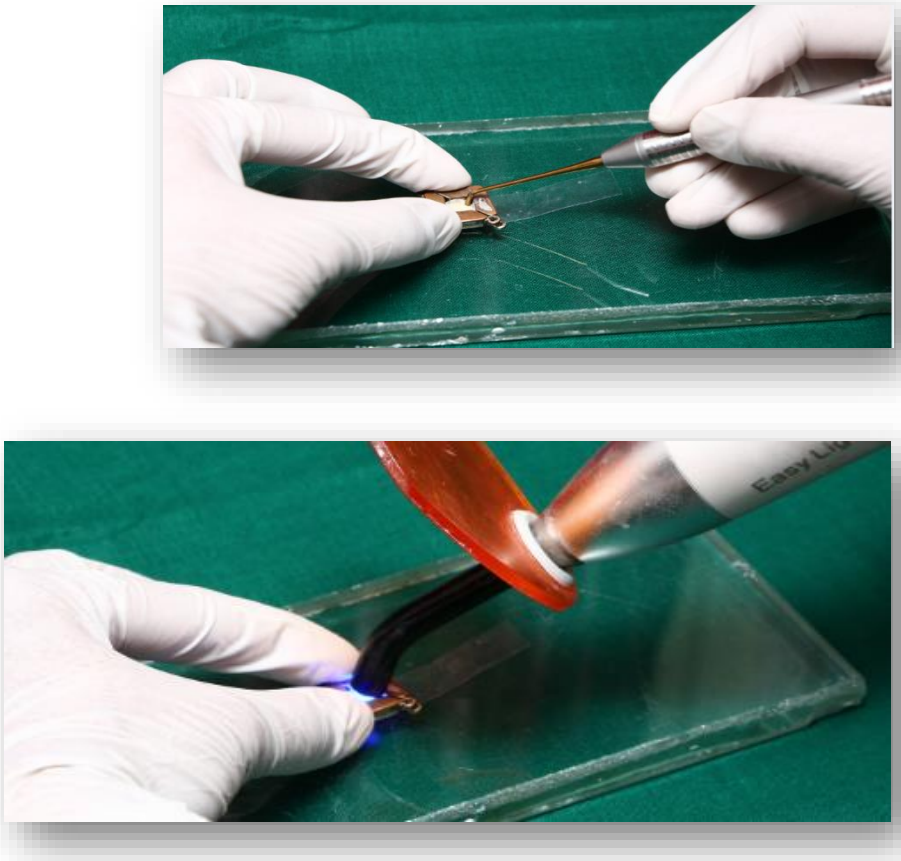


Fig 2.2 Light curing of composite block

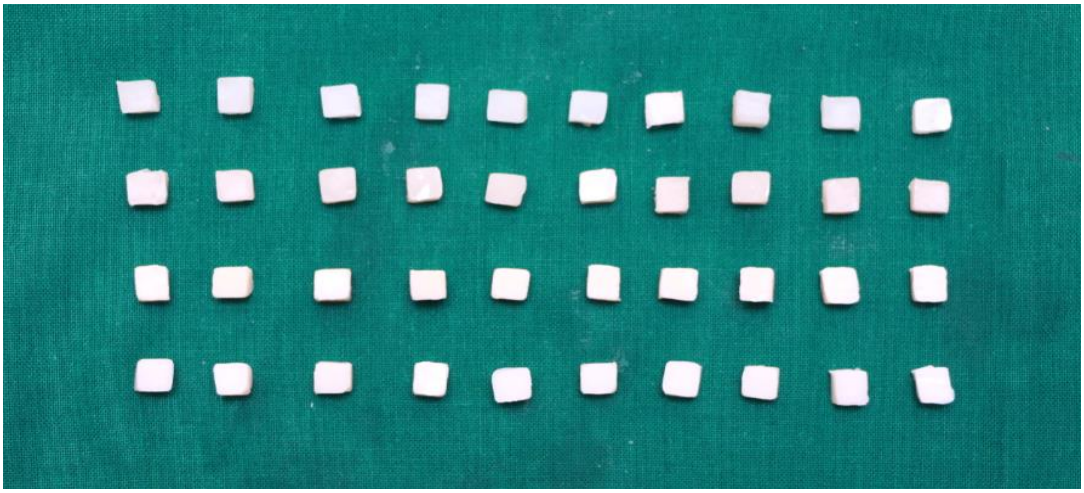


Fig 2.3 Tetric N Ceram bulk fill composite blocks

Fig 2.4 Filtek nanofill composite block

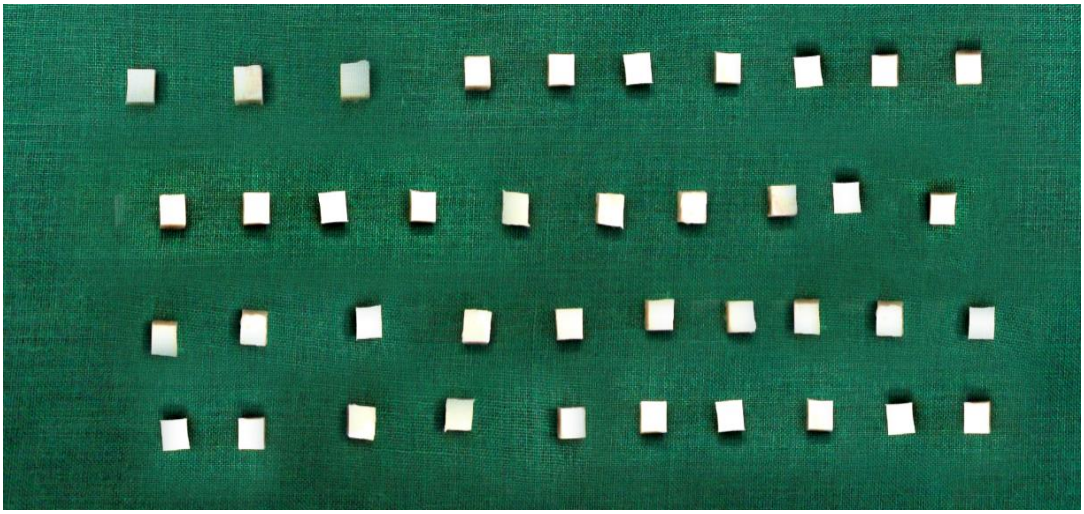


Fig 2.5 Admira (Ormocer) composite blocks

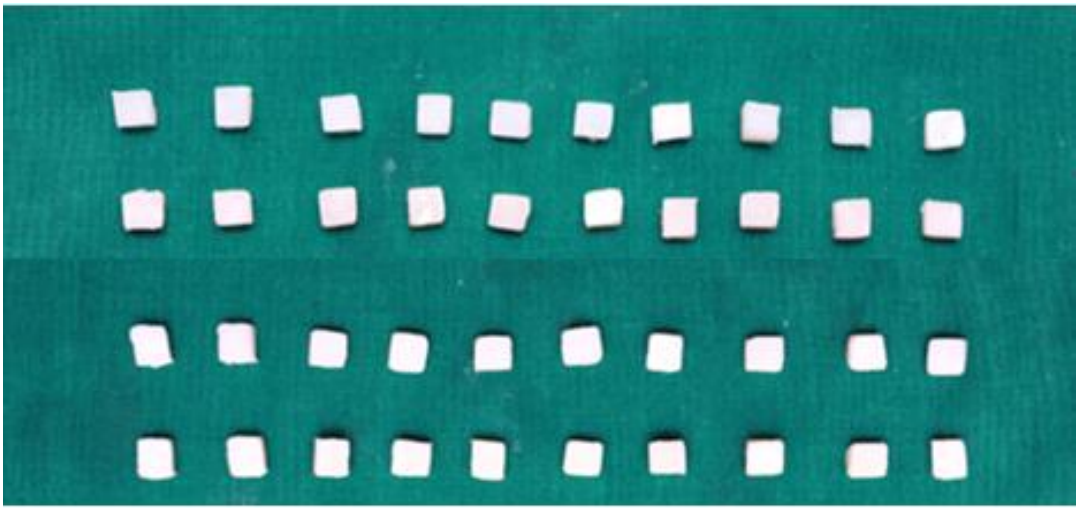


Fig 2.6 Restofill microhybrid composite blocks

3. SAMPLE PREPARATION FOR DEGREE OF CONVERSION

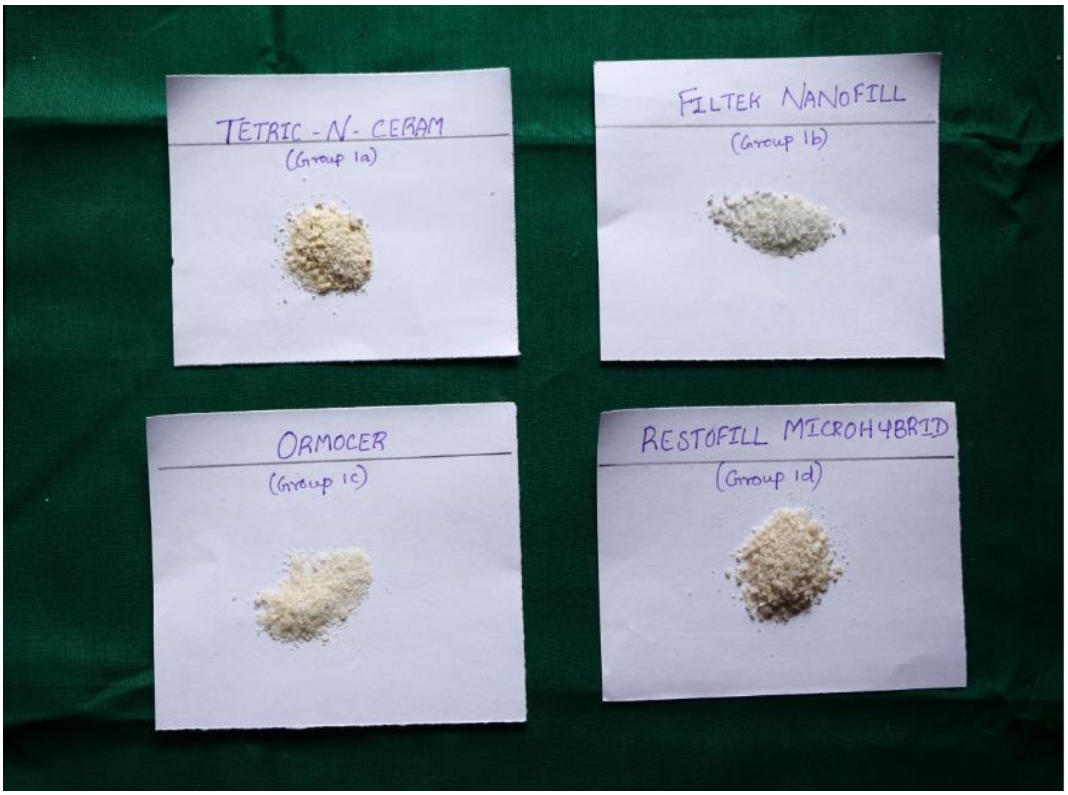


Fig 3.1 Samples in powdered form

Fig 3.2 Potassium bromide addition to the fine powdered sample

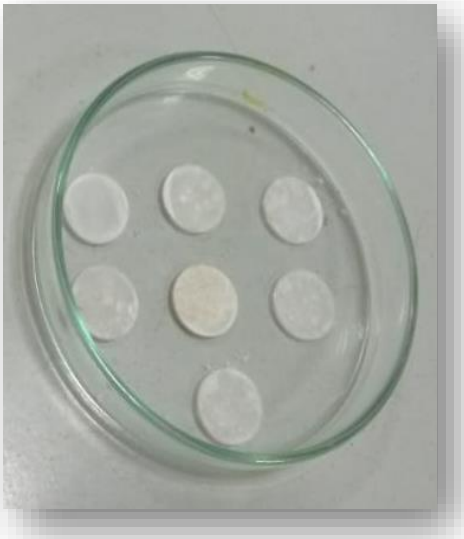
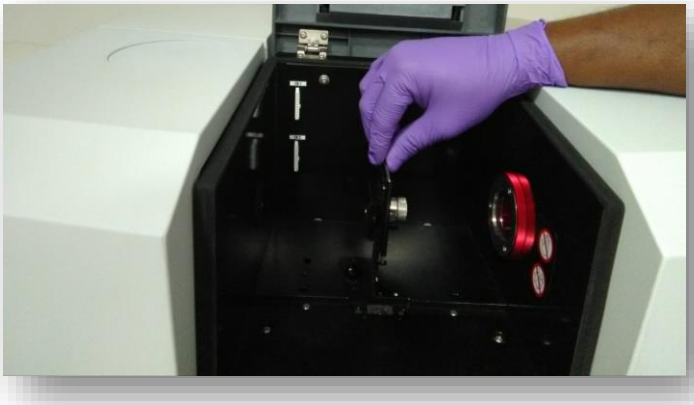


Fig 3.3 Preparation of thin disc using hydraulic press maker

Fig 3.4 Specimen inside cell holder



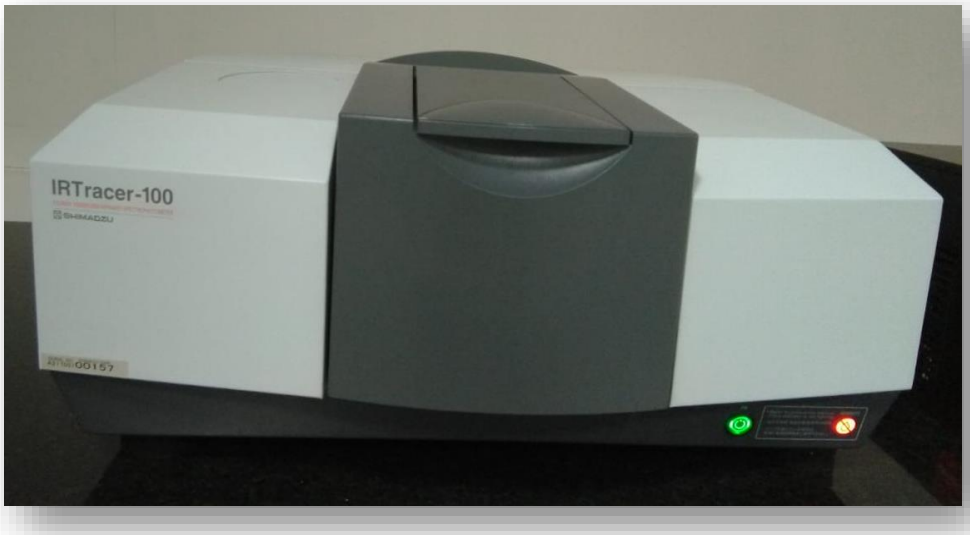


Fig 3.5 Fourier Transform Infrared Spectrophotometer

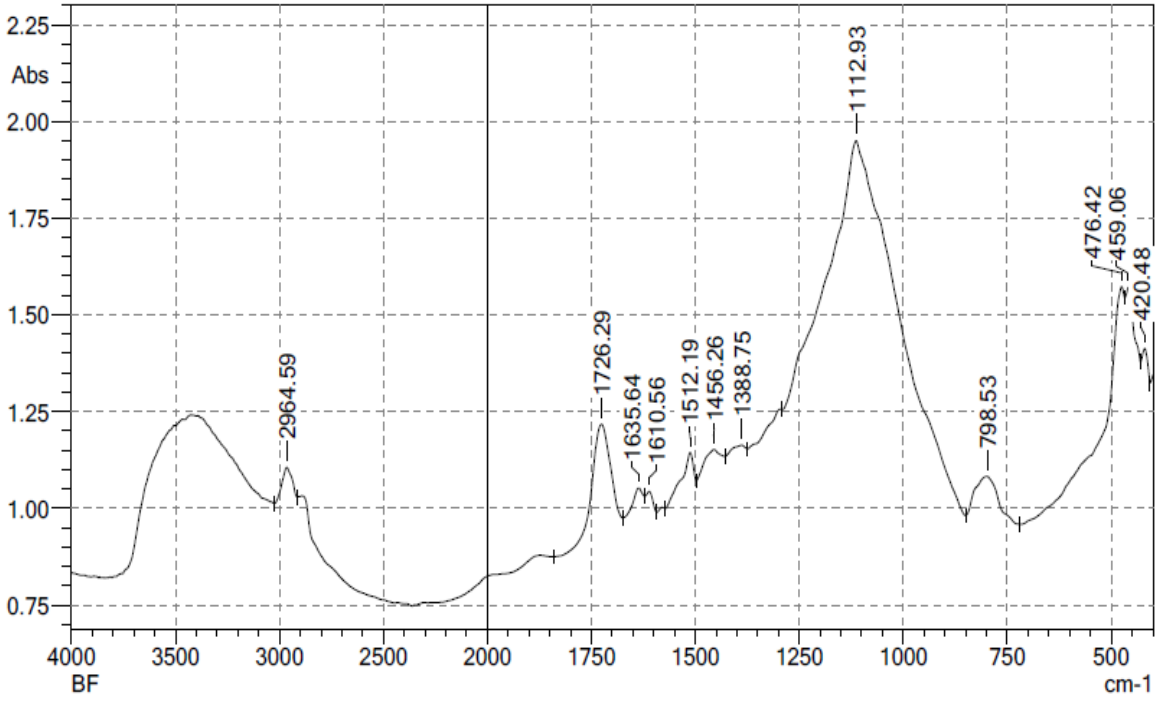


Fig 3.6 Graphical representation of FTIR for bulk fill nanohybrid composite

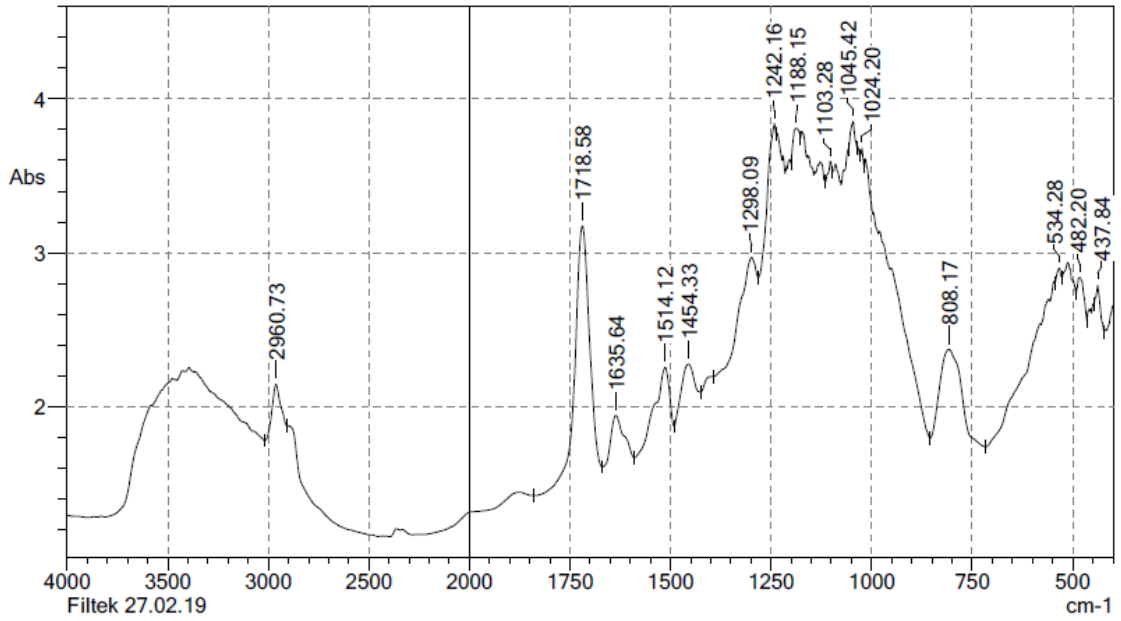


Fig. 3.7 Graphical representation of FTIR for nanofill composite

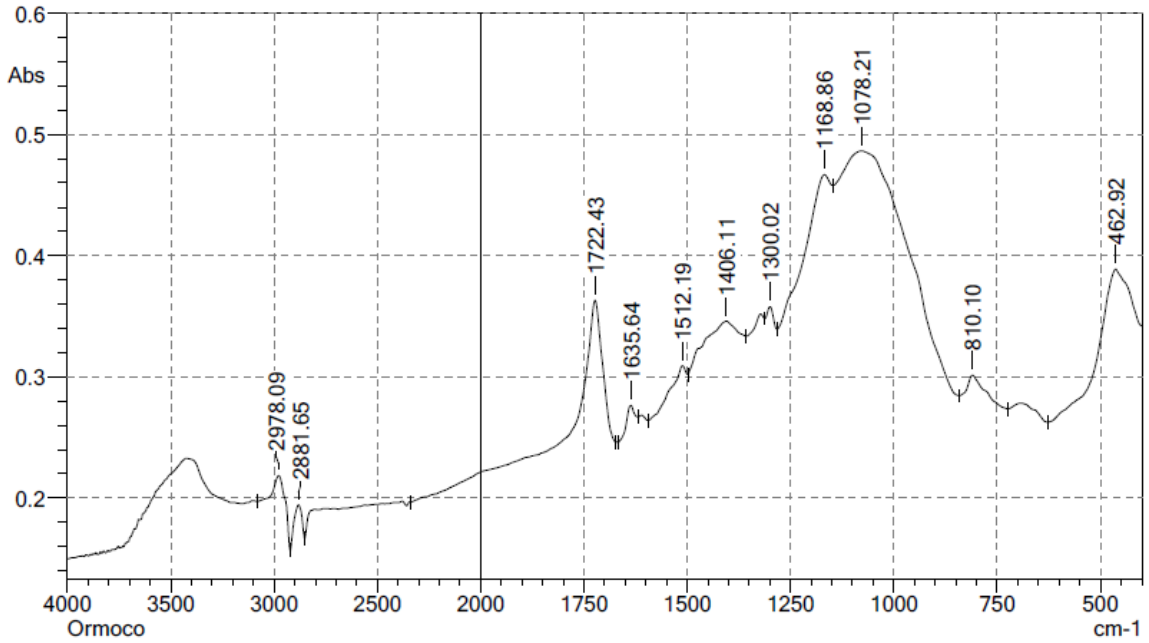


Fig 3.8 Graphical representation of FTIR for Ormocer based composite

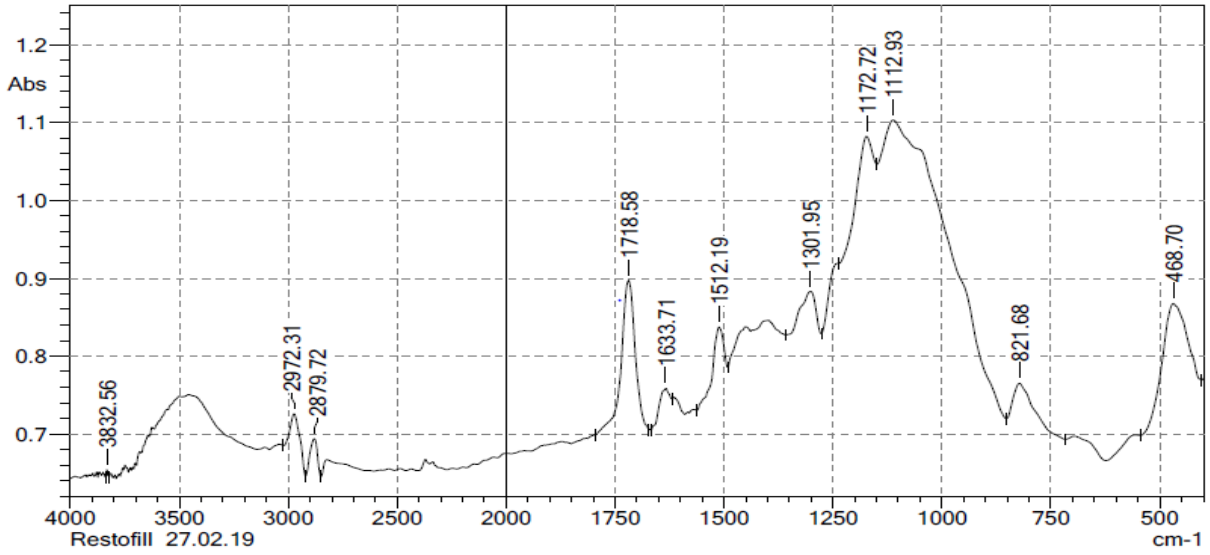


Fig.3.9 Graphical representation of FTIR for microhybrid composite

4.SURFACE HARDNESS ANALYSIS

Fig 4.1 Vicker surface hardness tester

5. SAMPLE PREPARATION FOR MARGINAL ADAPTATION ANALYSIS

Fig 5.1 80 human maxillary premolars

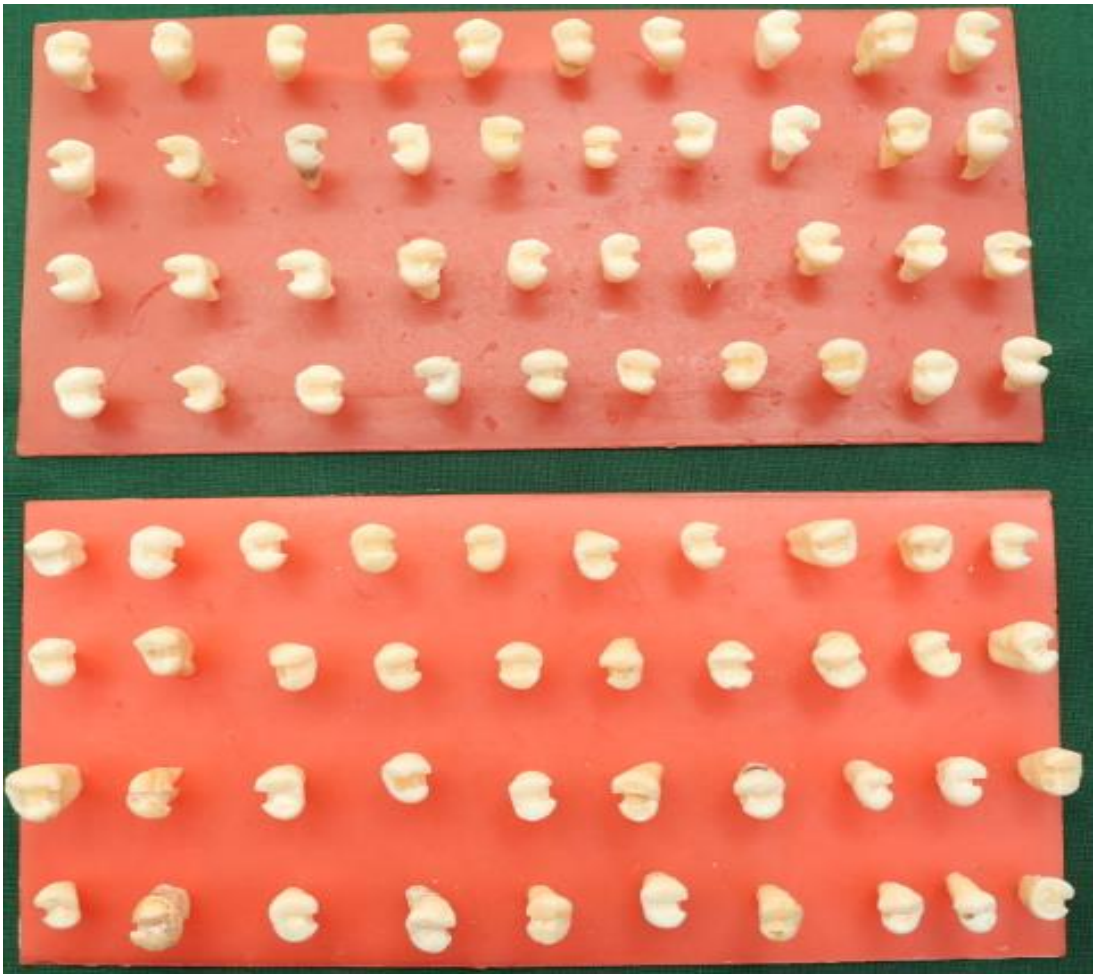


Fig 5.2 Class II cavity preparation in premolars

Fig. 5.3 Palodent sectional matrix application

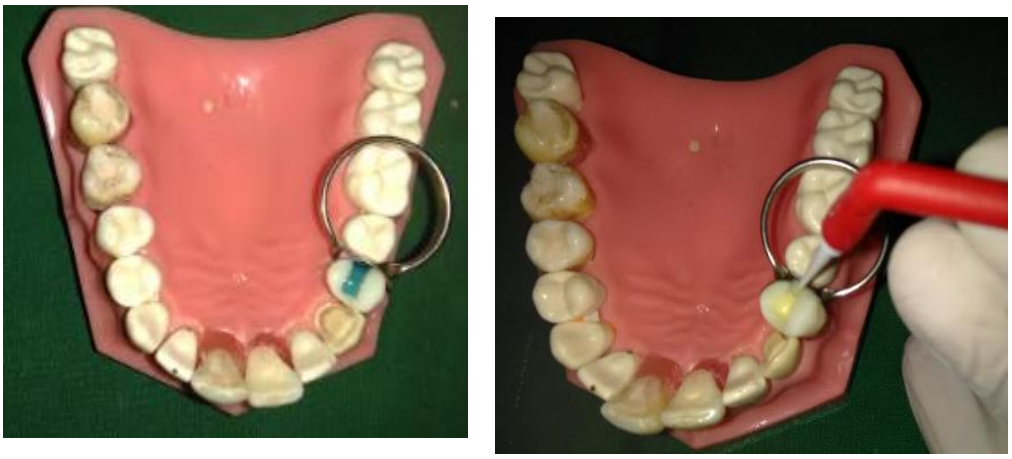


Fig 5.4 Etching with 37% Phosphoric acid gel and bonding agent application

Fig 5.5 Class II composite restorations in 80 premolars based on the respective groups

6. MARGINAL ADAPTATION EVALUATION USING SEM

Fig. 6.1 Zeiss Scanning Electron Microscope

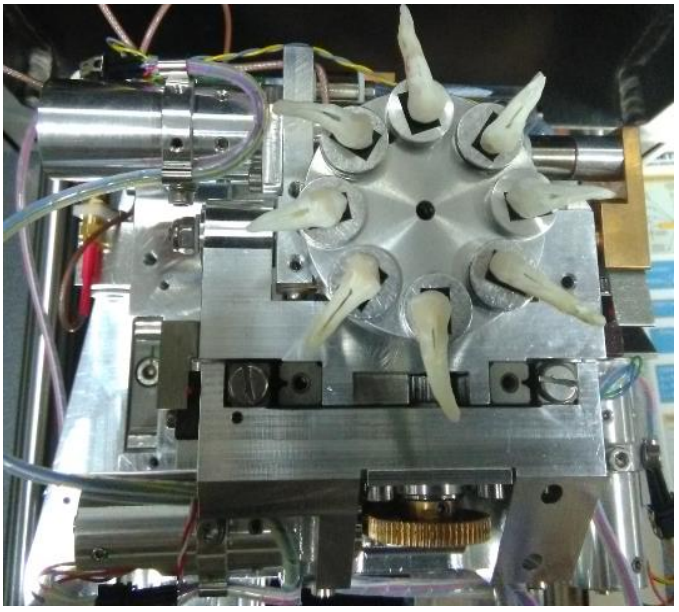


Fig 6.2 Sectioned samples in SEM

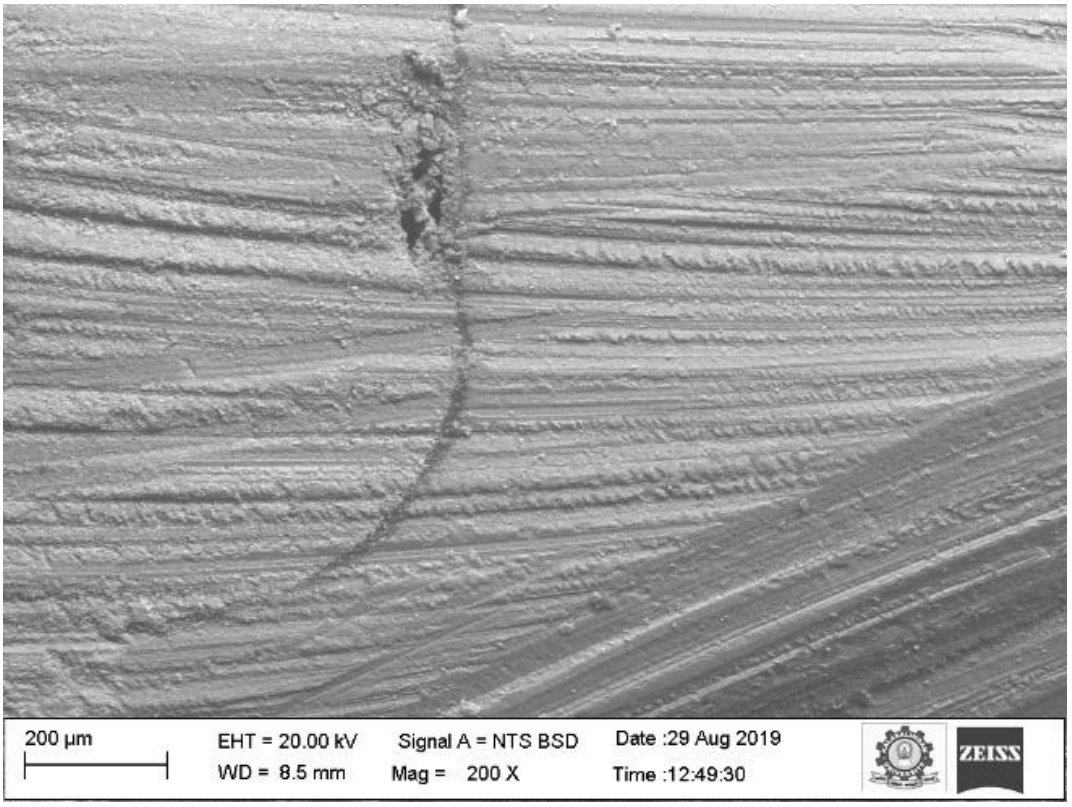


Fig 6.3 SEM image capture at 200x magnification

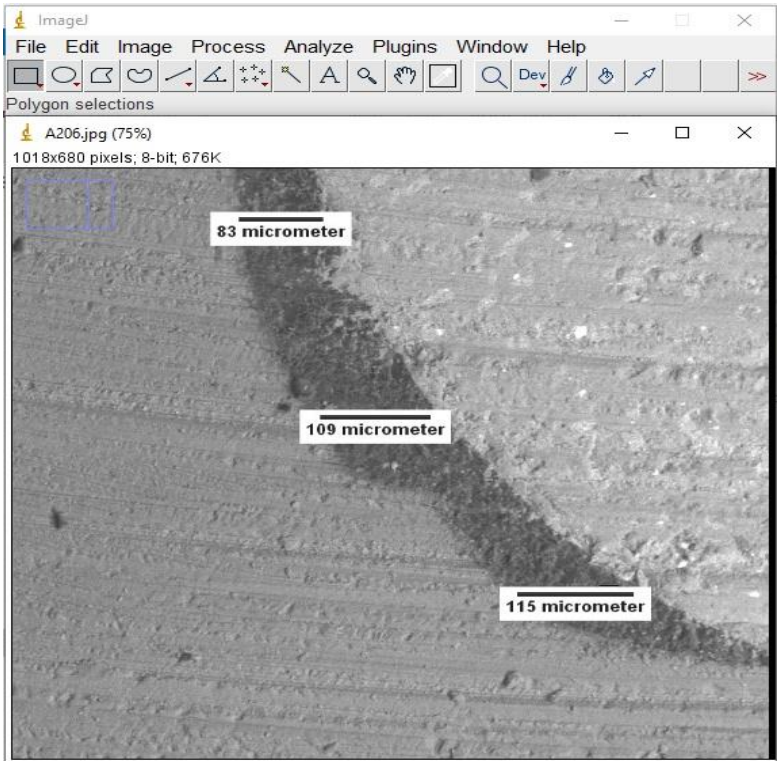


Fig 6.4 Width of the gap analysed using Image J software

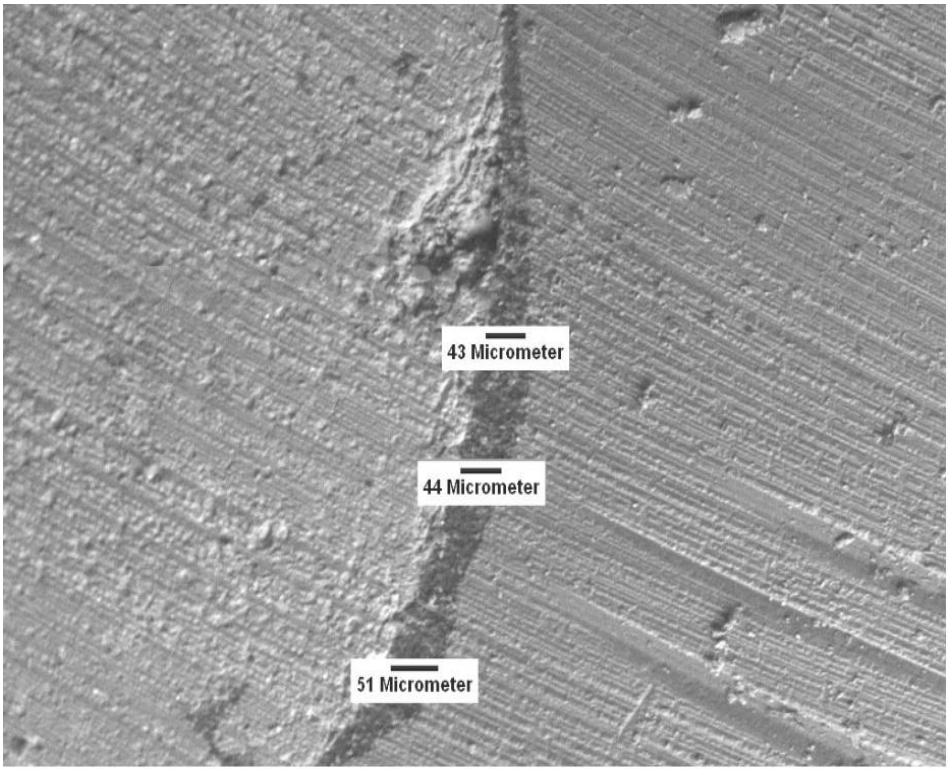


Fig 6.5 SEM image of MQ4 criteria

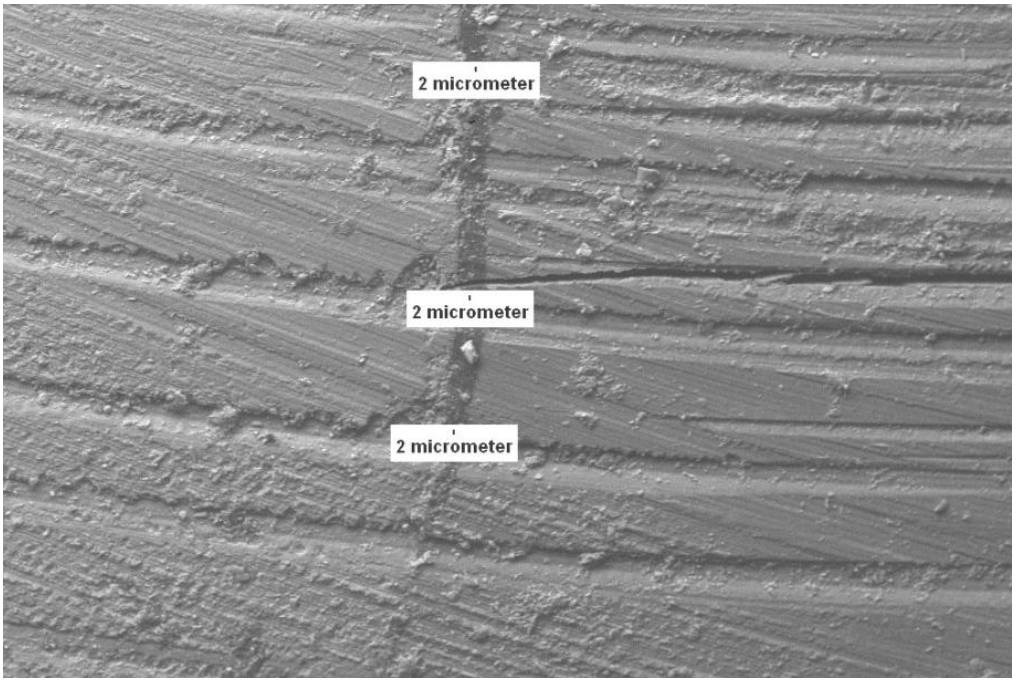


Fig 6.6 SEM image of MQ3 criteria

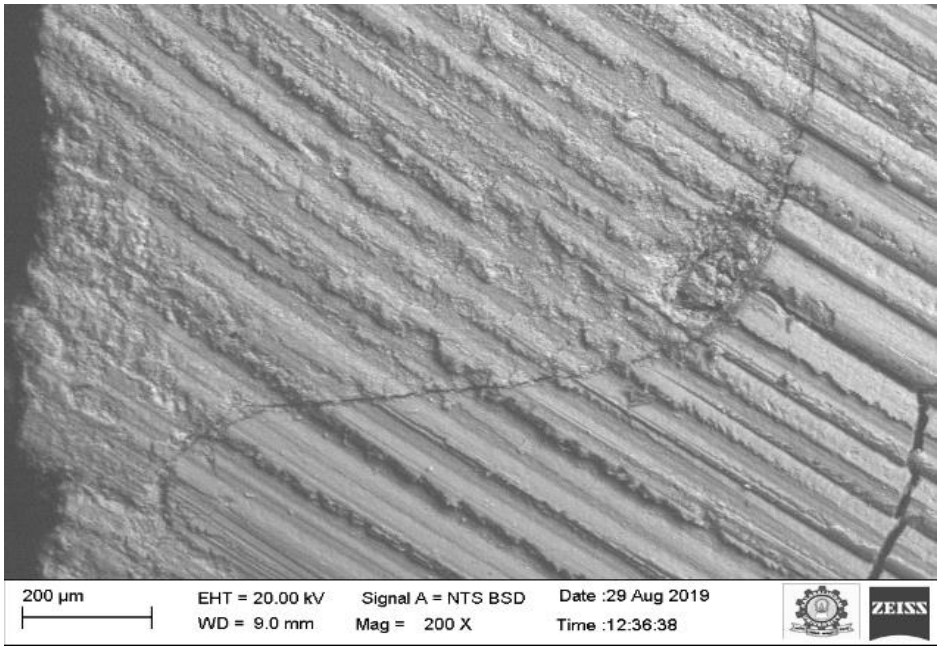


Fig 6.7 SEM image of MQ2 criteria

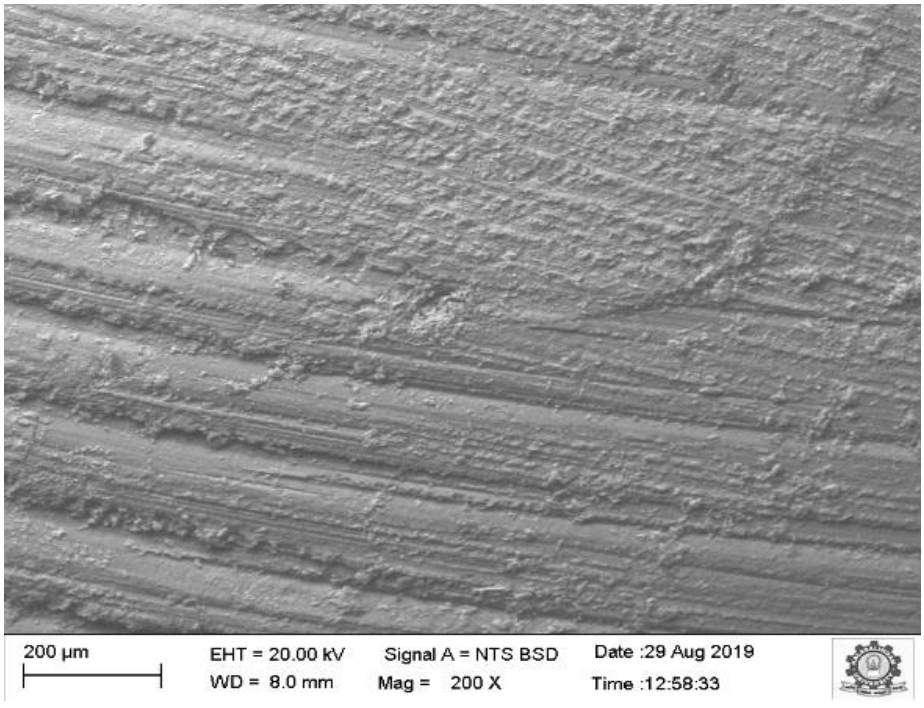


Fig 6.8 SEM image of MQ1 criteria

RESULTS

STATISTICAL ANALYSIS

The data from DC and surface hardness were analyzed by ANOVA. The measures were considered statistically significant at $p < 0.05$. Statistical analysis were performed using Statistical Package for Social Sciences (SPSS) version 21. Descriptive analysis were given in the form of mean ± standard deviation for the DC and surface hardness.

Margin assessment scores in ordinal data were analyzed using non – parametric Kruskal – Wallis test to check if significant differences in frequency of internal adaptation and gap formation existed between the groups.

RESULTS

Degree of conversion

The overall degree of conversion percentage mean values for all the composite samples in both the groups are given in the table 1 (page no: 40).Fourier Transform Infrared Spectrometer was used to evaluate the degree of monomer conversion to polymer**.** Microsoft office excel sheet was used for the data entry of all the samples. The mean value for the degree of conversion was calculated separately for the four composite materials used in two groups. Datas were analysed with Analysis of variance to check out the level of significance among the four different composites in two groups.

Statistical analysis showed increased degree of conversion in preheated group (group – 1) with statistically significant results. Ormocer resulted in highest DC followed by nanofill compared to other composites with statistically significant differences between them ($p < 0.001$). Bulk fill nanohybrid composites showed the lowest DC of 44.44% in preheated group. In group 2, nanofill composites showed the highest DC values of about 48.55% followed by Ormocer and nanohybrid composites. Lowest DC values was observed with the microhybrid composites of about 25.77% in composites without heating group.

The graphical representation of the degree of conversion mean values of four composite materials in preheated group (Group -1) using FTIR analysis is shown in Graph 1 (page no: 46).

The graphical representation of the degree of conversion mean values of four composite materials in room temperature group (Group -2) using FTIR analysis is shown in Graph 2 (page no: 46).

Surface hardness

The mean surface hardness values of different composite samples in both the groups are given in the table 2 (page no: 41). Vicker hardness tester was used to check the hardness of the material.The evaluated surface hardness values were entered in Microsoft office excel sheet for the ten samples of each composite materials. The mean value for the degree of conversion was calculated separately for the different composite materials used in two groups.Datas were analysed with Analysis of variance to check out the level of significance among the four different composites in two groups.

Statistical analysis showed increased surface hardness in preheated group than the control group with statistically significant results. Nanofill composites showed the highest surface hardness values of 110.58 VHN followed by Ormocer with 94.73 VHN compared to the other composites and the results are statistically significant ($p < 0.001$). Microhybrid composites showed the least surface hardness values among the preheated groups. In group 2, highest surface hardness values was observed with the nanofill composites of about 79.03 VHN followed by ormocer with the 64.07 VHN and bulk fill nanohybrid composites with the 51.44 VHN. Microhybrid composites showed the lowest surface hardness values of about 44.54 VHN in composites without heating group.

The graphical representation of the mean surface hardness values of four composite materials in preheated group (Group -1) using Vicker hardness tester is shown in Graph 3 (page no: 47). The graphical representation of the mean surface hardness values of four composite materials in room temperature group (Group -2) using Vicker hardness tester is shown in Graph 4 (page no: 47).

Internal marginal adaptation

MQ3 and MQ4 scores were categorized in both the groups based on the gap width measurements using Image J software. The comparison of two groups with the four different composite materials were analyzed using Kruskal Wallis test. The level of significance was kept at $p < 0.05$.

Axial adaptation score result of restorative – dentin interface of bulk fill nanohybrid composite in both the groups - Group 1A & 2Awere given in the table 3 (page no: 42). Higher frequency of MQ4 scores in four samples were observed in preheated group (group 1). None of the samples showed MQ4 score in room temperature nanohybrid composite group. (group 2). Increased MQ1 scores were observed in room temperature nanohybrid composites of about three samples and the results are not statistically significant.

Axial adaptation score result of restorative – dentin interface of nanofill composite in both the groups - Group 1B & 2 Bwere given in the table 4 (page no: 43). MQ4 scores was found in one nanofill sample in both the groups. Higher frequency of MQ4 scores in three samples were observed in preheated group (group 1). Four samples showed MQ4 score in room temperature nanohybrid composite group (group 2). Overall, increased MQ1 and MQ2 scores were observed in six samples out of ten samples in nanofill preheated composites and the results are not statistically significant.

Axial adaptation score result of restorative – dentin interface of Ormocer based composite in both the groups - Group 1C & 2Cwere given in the table 5 (page no: 44). Higher frequency of MQ4 scores in three samples were observed in preheated group (group 1). None of the samples showed MQ4 score in room

temperature Ormocer based composite group (group 2). Overall, increased MQ1 and MQ2 scores were observed in eight samples out of ten samples in Ormocer room temperature composites (group -2) and the results are not statistically significant.

Axial adaptation score result of restorative – dentin interface of microhybrid composite in both the groups - Group 1D & 2Dwere given in the table 6 (page no: 45). Higher frequency of MQ3 scores in five samples were observed in preheated group (group 1). Four samples showed MQ4 score in room temperature based composite group (group 2). Overall, increased frequency of MQ3 and MQ4 scores were observed in seven samples out of ten samples in microhybrid composites in both the groups (group – 1 and 2) and the results are not statistically significant.

Overall, no statistically significant results were found in terms of marginal adaptation and percentage of gap formation in both the groups. But composites without preheating (group -2) showed lesser MQ3 and MQ4 values compared to the preheated group and the results are not statistically significant. Bulk fill nanohybrid and Ormocer showed higher frequency of MQ4 values in preheated group than the control group.The chart displaying the contribution of ten samples in bulk fill nanohybrid composite in group – 1 and group – 2 to their respective axial adaptation scores were shown in pie chart no: 1 (Page no: 48).The chart displaying the contribution of ten samples in nanofill composite in group – 1 and group – 2 to their respective axial adaptation scores were shown in pie chart no: 2 (Page no: 48). The chart displaying the contribution of ten samples in Ormocer composite in group – 1 and group – 2 to their respective axial adaptation scores were shown in pie chart no: 3 (Page no: 49). The chart displaying the contribution of ten samples inmicrohybrid composite in group – 1 and group – 2 to their respective axial adaptation scores were shown in pie chart no: 4 (Page no: 49).

LIST OF TABLES

Table 1: Testing the significance effects of preheating and without preheating on the degree of conversion of four composites

Degree of conversion	Groups	N	Mean	Standard Deviation	Minimum	Maximum	p-value
Tetric	**Group 1A**	10	44.44	2.84	40.29	48.16	< 0.001*
	Group 2A	10	27.58	2.83	22.00	31.02	
Filtek	**Group 1B**	10	66.14	5.46	60.22	75.31	< 0.001*
	Group 2B	10	48.55	8.77	37.49	59.92	
Ormocer	**Group 1C**	10	74.35	6.80	64.36	83.23	< 0.001*
	Group 2C	10	41.15	3.27	37.49	47.32	
Restofill	**Group 1D**	10	49.98	9.39	37.23	60.22	< 0.001*
	Group 2D	10	25.77	2.87	22.38	32.03	

* = Statistically Significant ($p < 0.05$)

p-value based on ANOVA (Analysis of Variance)

Table 2: Testing the significance effects of preheating and without preheating on the surface hardness of four composites

Surface Hardness	Groups	N	Mean	Standard Deviation	Minimum	Maximum	p-value
Tetric	**Group 1A**	10	73.15	2.51	68.89	76.78	< 0.001*
	Group 2A	10	51.44	4.60	43.98	59.00	
Filtek	**Group 1B**	10	110.58	3.35	105.00	116.00	< 0.001*
	Group 2B	10	79.03	2.62	74.89	82.17	
Ormocer	**Group 1C**	10	94.73	3.53	89.01	99.56	< 0.001*
	Group 2C	10	64.07	3.09	58.40	69.00	
Restofill	**Group 1D**	10	58.32	2.95	54.10	63.02	< 0.001*
	Group 2D	10	44.54	2.64	40.62	47.78	

* = Statistically Significant ($p < 0.05$)

p-value based on ANOVA (Analysis of Variance)

Table 3: Testing the significance effects of preheating and without preheating on the marginal adaptation of bulk fill nanohybrid composite				
Internal Marginal Adaptation – Tetric			**Group 1A**	**Group 2A**
Margin Quality	**MQ 1**	**Frequency**	1	3
		Percentage	10%	30%
	MQ 2	**Frequency**	2	2
		Percentage	20%	20%
	MQ 3	**Frequency**	3	5
		Percentage	30%	50%
	MQ 4	**Frequency**	4	0
		Percentage	40%	0%
Mean Rank			12.70	8.30
p-value			0.082	
* = Statistically Significant ($p < 0.05$) p-value based on Kruskal-Wallis Test				

Table 4: Testing the significance effects of preheating and without preheating on the marginal adaptation of nanofill composite

Internal Marginal Adaptation – Filtek			**Group 1B**	**Group 2B**
Margin Quality	**MQ 1**	**Frequency**	3	3
		Percentage	30%	30%
	MQ 2	**Frequency**	3	2
		Percentage	30%	20%
	MQ 3	**Frequency**	3	4
		Percentage	30%	40%
	MQ 4	**Frequency**	1	1
		Percentage	10%	10%
Mean Rank			10.20	10.80
p-value			0.813	

* = Statistically Significant ($p < 0.05$)

p-value based on Kruskal-Wallis Test

Table 5: Testing the significance effects of preheating and without preheating on the marginal adaptation of Ormocer based composite

Internal Marginal Adaptation – Ormocer			**Group 1C**	**Group 2C**
Margin Quality	**MQ 1**	**Frequency**	3	5
		Percentage	30%	50%
	MQ 2	**Frequency**	2	3
		Percentage	20%	30%
	MQ 3	**Frequency**	2	2
		Percentage	20%	20%
	MQ 4	**Frequency**	3	0
		Percentage	30%	0%
Mean Rank			12.35	8.65
p-value			0.143	

* = Statistically Significant ($p < 0.05$)

p-value based on Kruskal-Wallis Test

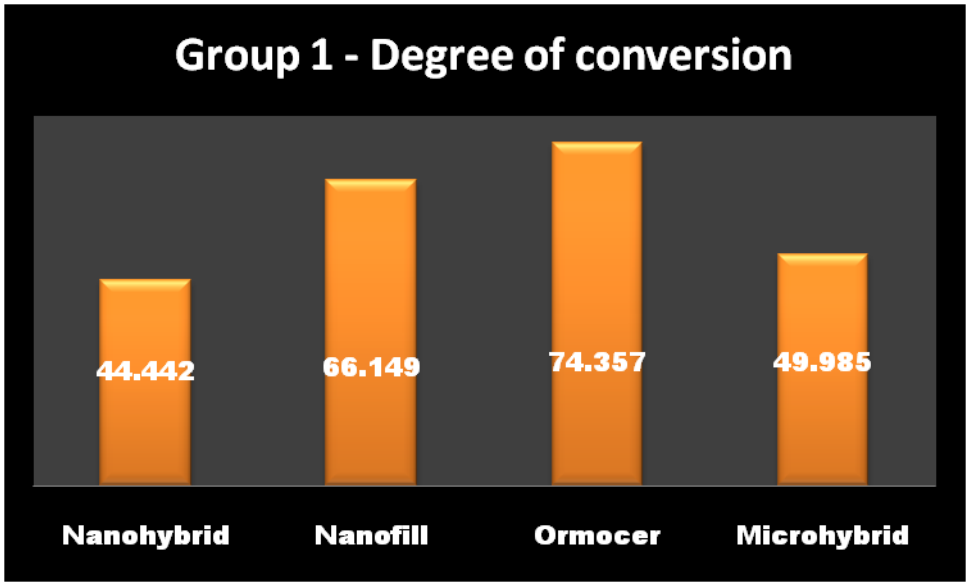
Table 6: Testing the significance effects of preheating and without preheating on the marginal adaptation of microhybrid composite

Internal Marginal Adaptation – Restofill			**Group 1D**	**Group 2D**
Margin Quality	**MQ 1**	**Frequency**	2	3
		Percentage	20%	30%
	MQ 2	**Frequency**	1	0
		Percentage	10%	0%
	MQ 3	**Frequency**	5	4
		Percentage	50%	40%
	MQ 4	**Frequency**	2	3
		Percentage	20%	30%
Mean Rank			10.30	10.70
p-value			0.872	

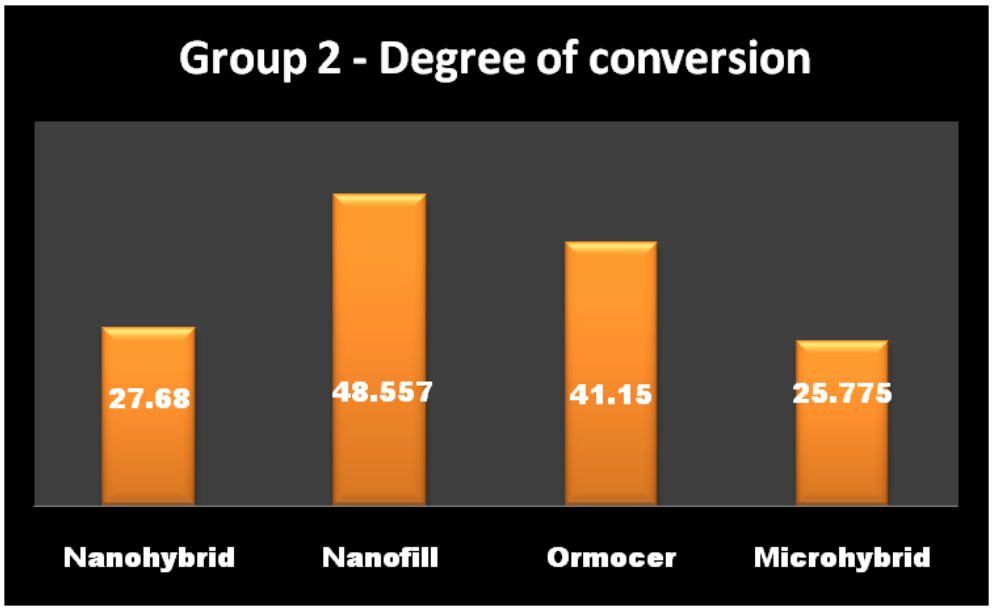
* = Statistically Significant ($p < 0.05$)

p-value based on Kruskal-Wallis Test

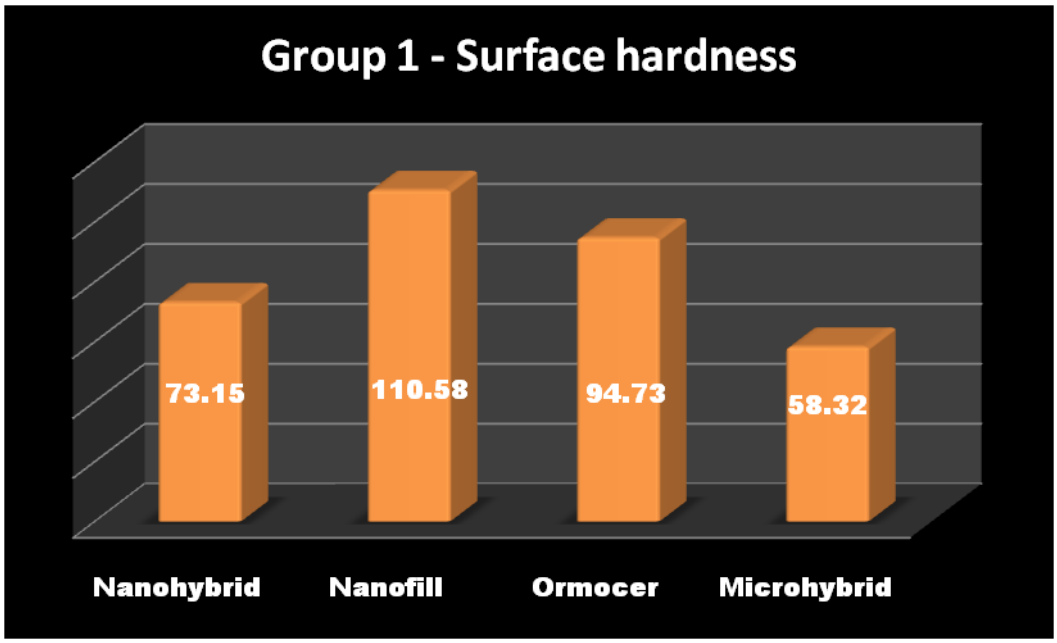
List of graphs



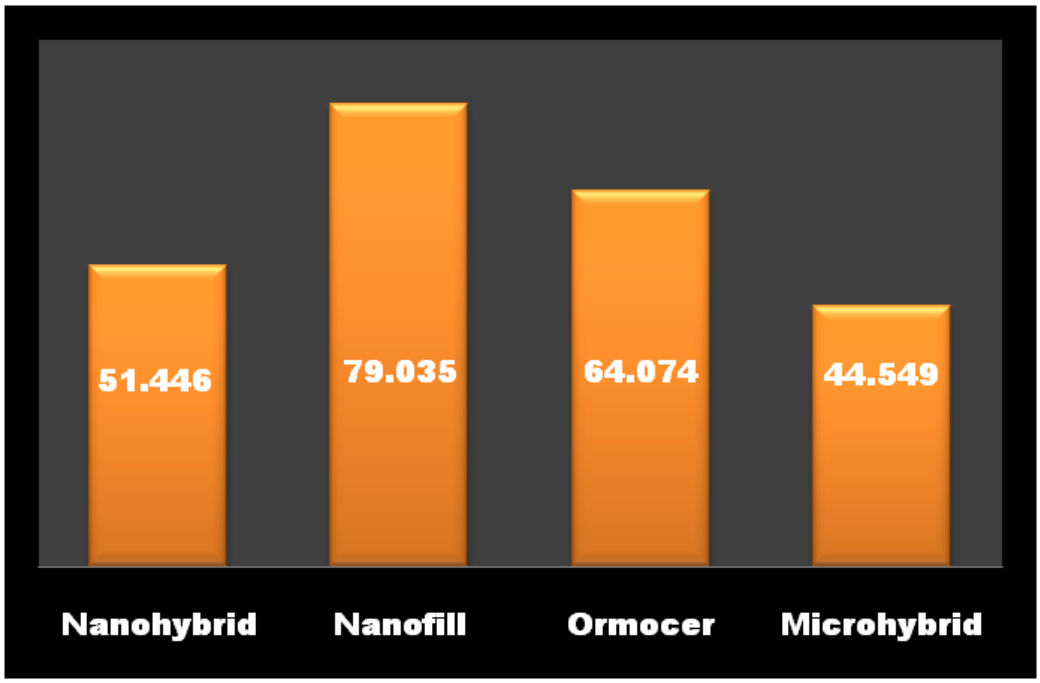
Graph 1: Comparison of mean values of four composites in preheated group



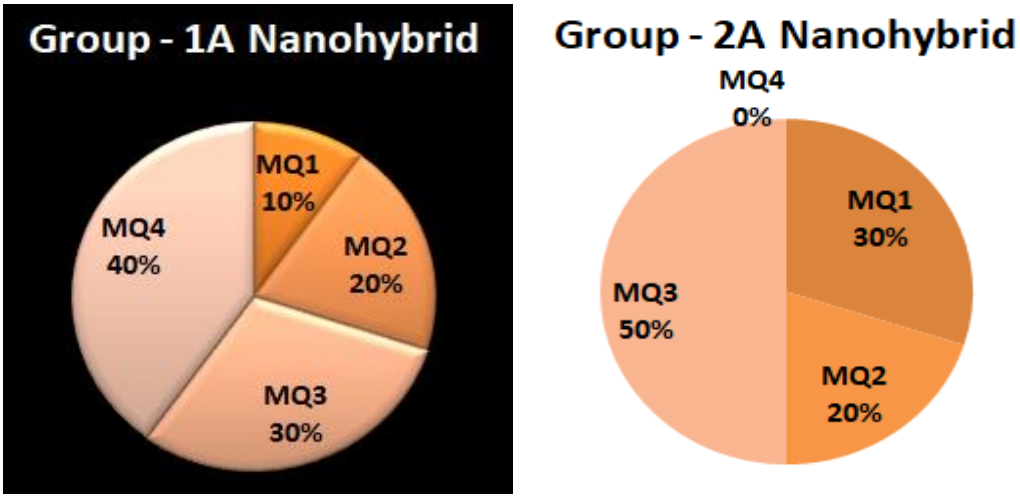
Graph 2: Comparison of mean values of four composites in group without preheating



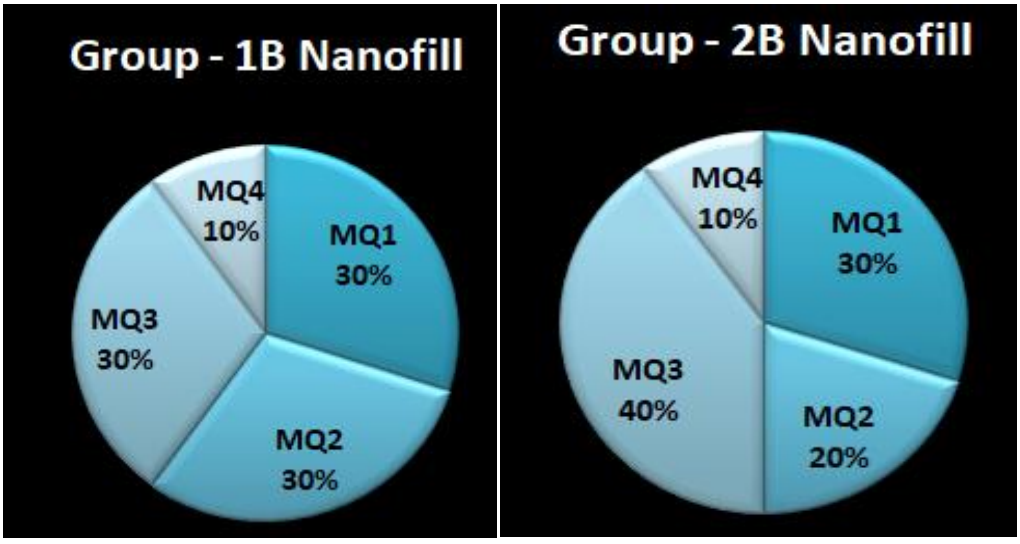
Graph 3: Comparison of mean values of four composites in preheated group



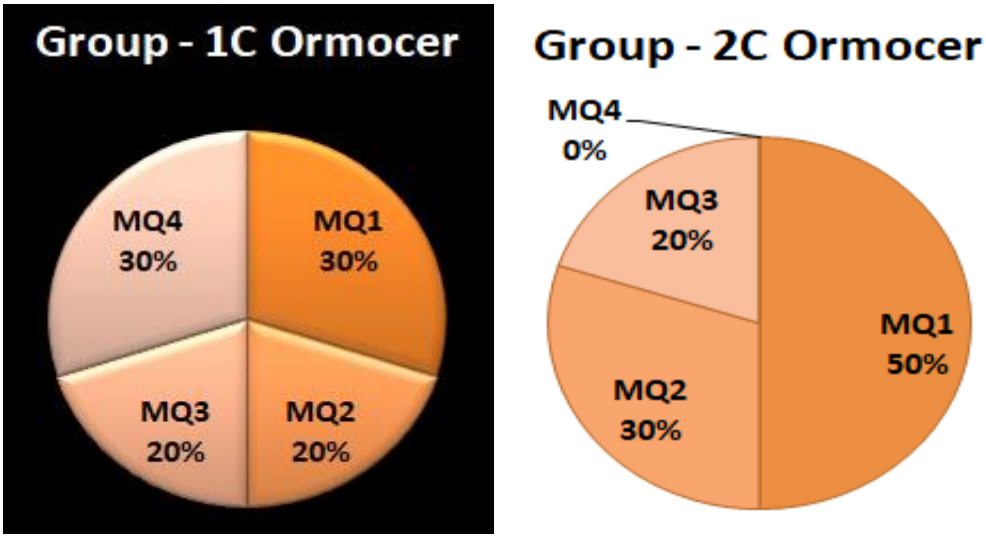
Graph 4: Comparison of mean values of four composites in group without preheating



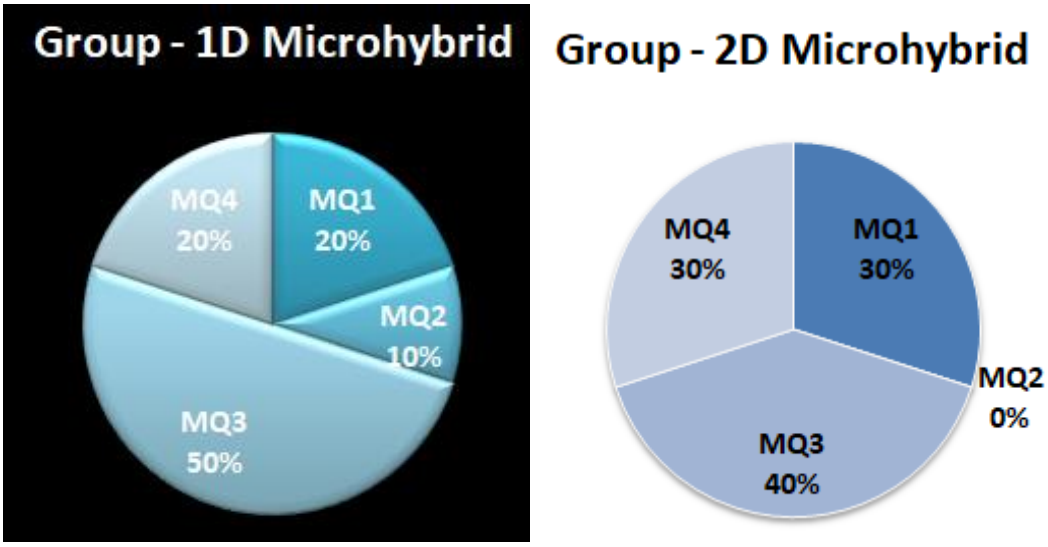
Pie chart 1: Comparison of percentage distribution of axial adaptation scores of bulk fill nanohybrid composite in group 1 and group 2



Pie chart 2: Comparison of percentage distribution of axial adaptation scores of Nanofill composite in group 1 and group 2



Pie chart 3: Comparison of percentage distribution of axial adaptation scores of Ormocer based composite in group 1 and group 2



Pie chart 4: Comparison of percentage distribution of axial adaptation scores of Microhybrid composite in group 1 and group 2

DISCUSSION

In this study, a split mold of length 5 mm × width 5 mm × height 3 mm of clinically relevant dimensions was filled with the four different composite resins before and after heating the composites. Different types of composite resins were included in this study: 1) Bulk fill nanohybrid Tetric N ceram composite, 2) Filtek Z350 universal nanofill composite, 3) Admira ormocer based composite, 4) Restofill microhybrid composite. Since the polymerization reaction plays an important role on the mechanical properties of resin composites. Polymerization reaction and the degree of conversion depends on the type of resin monomer used, filler content of the composite, opacity of the composite resin, elastic modulus of the material, intensity of light used, cavity configuration factor and the curing characteristics of the composite resin [30] (Table no: 7, page no: 57).

According to the results of present study, preheating the composites would cause increase in degree of conversion and surface hardness of the samples, and the null hypotheses was accepted. This study showed that monomer conversion and surface hardness were significantly improved by preheating the composites. Decrease in paste viscosity was clinically examined in the study due to heating. Although the composite was preheated at 61ºC for 20 minutes in this study, a slightly lower composite temperature was observed below the preset temperature. It was in accordance with the previous study, stated that there was a noteworthy drop in temperature around 40% in 40 seconds after compule was removed from the heating device [31]. So, by the time when the composite is light cured, it may reach its room temperature but transient viscosity reduction achieved after preheating in this study helped to improve the workability inside the cavity preparation. It is important to note that thinner viscosity of flowable composites in syringes cannot be expected

in the preheated composites at 61ºC which provide only moderate transient viscosity reduction.

The effect of composite preheating on its viscosity depends on the composite type and brand used. In this study, bulk fill nanohybrid composites showed more viscosity reduction due to heating and Ormocer showed lesser viscosity reduction since it was a hybrid polymer. Preheated composites can act as a thermal insulator because of the presence of organic resin matrix and inorganic filler particles present. It was observed in a study conducted by da Costa J *et al* which stated that heating composites had differences in the flow of certain brands of resin composites [32].

In this study, 4 different composite materials were used including nanohybrid, nanofilled, microhybrid and organically modified ceramic composites. Also, variations in placement techniques included one subgroup of bulk fill nanohybrid composite and other subgroups with incrementally layering technique was used. Since, final degree of conversion of a material depend on the differences in composition of composites, the thickness of the material used in posterior restorations and the light intensity used. The variability in DC of different composites may be due to changes in composition of resin matrices and initial viscosity of monomers used.

In this study, the mean DC values obtained for each composite materials in preheated group were between 44.44 % and 74.35%. Ormocer showed highest monomer conversion values in preheated group followed by nanofill 66.14% and microhybrid composites 49.98%. Ormocer which is an organically modified ceramic based composites showed a range of DC values about 74.35% in preheated group and 41.15% in group without preheating. These material was commercialized in

dentistry after 1998, consist of hybrid polymer based material containing pure inorganic organic polymers that are highly viscous. Inorganic 3D network is formed by polycondensation of the alkoxysilanes, producing siloxane bonds (Si O Si). This results in matrix of inorganic backbone of silica chains with polymerizable organic groups as lateral chains [33]. The reason why ormocer showed highest DC values is due to the hybrid polymers with the use of diluting monomers when compared to the conventional monomers. Heating further enhanced the monomer conversion in preheated group that resulted in highest DC in Ormocer.

The results of the present study showed that the DC values of nanofill and nanohybrid composites were significantly lower than the Ormocer based composites. The results are in agreement with the other studies who stated that silica nanoparticles and nanoclusters in nanofill and nanohybrid composites have a light scattering effect. Due to its higher filler loading, its light intensity might be reduced by the effect of dispersing the light [34, 35]. Another reason for lower DC in tetric bulk fill composites might be due to its lower light transmission inside the bulk of the composite. Since 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.

In the present study, the mean surface hardness values for the composites in preheated group was between 110.58 VHN and 58.32 VHN. Among them, nanofill composites showed the highest surface hardness values followed by Ormocer and it was statistically significant. Bulk fill material also shown better hardness values. Degree of conversion usually correlate with the surface hardness. But the results of the present study showed that bulk fill nanohybrid composites have lowest DC and

comparatively higher surface hardness than the microhybrid composite. It might be due to the light intensity used during curing was greater at the surface and reduced as it penetrate deeper into the material, thus it could affect the degree of monomer conversion [10].

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 60°C. The efficiency of these warmers was studied by Daronch and co-workers [31]. 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 our study, Composite warmer (Delta product, Chennai, India) was used to heat the composite materials to the required temperature constantly. The clinical technique with the delta heating device include after the unit is turned on and the green LED indicator flashes that indicate the functioning of the unit. It was available with five different sizes of composite holders to heat the composite compule [36].

One of the benefit provided by Calset warmer was that it stored the preheated compules at the preset temperature until its use, that cannot be maintained with the Delta warmer [37]. So here the question of repeated preheating was necessary for the preparation of all composite samples. So the alternative repeated preheating and cooling of composite resins might have its influence on the material's properties. A study conducted by D' Amario M *et al* stated that repeated preheating cycles of 39 ºc did not affect the mechanical properties of composite materials tested [38]. It was in contrast to another study which stated that more than 10 cycles of repeated preheating had negative influence on the flexural strength of resin composites tested [39].

In previous studies, two temperature settings at 54°C and 60°C were alternatively used. Maximum syringe temperature achieved was 49°C when calset unit was preset to 54°C and 55.1°C when preset to 60°C. The lower film thickness provided by the preheated composite at 60°C was similar to that of preheated at 54°C [40].

The major concern in class II direct composite restorations is to achieve proper adaptation to dentinal wall in axial and gingival margins. Usually, flowable composites was used as stress absorbing layer to minimize contraction shrinkage. In a study conducted by Labella *et al* described the use of flowable composites in two different methods as an intermediate layer light cured either before or simultaneously with the overlying composite [41]. Light curing both the flowable liner and the high viscosity composites simultaneously can provide maximum stress relief than the flowable composites that was polymerized beforehand [42]. But use of flowable composites were associated with lower elastic modulus of elasticity, thus it may result in inferior mechanical properties.

Another way to use low viscosity composites can be achieved with the preheating method. Previous studies stated that the advantage of using preheated composites instead of flowable composites was that resin composite with increased filler loading was used with no restorative compromise [43].

In our study, different preheated composites were used to assess their effect on internal marginal adaptation compared to the use of room temperature composites. The results of the present study showed that there was differences in terms of marginal gap formation in preheated composites than the room temperature composites. Preheated composites showed higher frequency of internal gap formation with no statistically significant results.

The criteria given by Blunck and Zaslansky where MQ1 and MQ2 was associated with absence of gap. The differences was in terms of marginal irregularities. While MQ3 and MQ4 scores was associated with the presence of gap and the difference was in terms of gap width. The mean average of gap width in three areas were measured using Image J analysis software. Among them, higher frequency of MQ4 values was observed in bulk fill nanohybrid and Ormocer based preheated composites. Microhybrid composites showed higher MQ3 values in preheated group.

The results of our present study was in accordance with the study conducted by C Sabatini *et al* regarding gingival marginal gap formation of preheated composites . The reason behind the increased frequency of gap formation in preheated group can be explained in relation to the cooling curve of the resin composite. Light curing the resin at its higher temperature was associated with the increased degree of monomer conversion into polymer. Higher conversion rates was related to the production of higher shrinkage stresses. Thermal contraction in relation to time also potentially increase the contraction stress that resulted in combined shrinkage stress that adversely hamper the marginal adaptation of hybrid resin [44].

There are certain limitations in this in- vitro study as there are some in-vivo variables that have role in marginal gap formation in posterior composite restorations that cannot be duplicated in – vitro. The room temperature composite temperature was around 23ºC. The temperature of oral cavity was around 35º C, so the preheated composites can be expected to retain the temperature for more time than the room temperature composites. In the present study, cavity margins were

placed at the level of CEJ in dentin since dentin possess greater adhesive challenge than enamel.

In accordance to the results of present study, future studies should be carried out simulating the in – vivo conditions, since the clinician's technique had an influence on the observed results.

Table 7: Characteristics of resin composites investigated in this study

Composite product type	**Composite brand (Manufacturer)**	**Composition (Resin matrix)**	**Composition (Filler type)**
Nanohybrid packable bulk fill composite	Tetric N-Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein, Western Europe)	Dimethacrylates 21.0% (Bis-GMA, Bis-EMA,UDMA)	Prepolymer Filler 17.0% (Glass filler, Ytterbium trifluoride), Ba – Al – Si glass, Mixed oxide 61.0%, Additive, Initiators, Stabilisers, Pigments, 1.0%
Nanofilled conventional composite	Filtek ™ Z350 XT (3M ESPE AG, Brazil)	Bis GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA	20nm nanosilica fillers, 5-20nm agglomerated zirconia/silica particles,0.6-1.4um clusters particle size, 78 wt%
Organically modified composite	Admira (VOCO GmBH, Cuxhaven, Germany)	Organically modified ceramic nano-particles	A special blend of micro and nano fillers of average particle size of 20 nm Glass filler size (mean) µm 1.2 – 1.6
Microhybrid conventional composite	Restofill (Anabond Stedman Pharmaceuticals, India)	Bis-GMA, bis-EMA, TEGDMA	Barium aluminio borosilicate (<1m). Barium fluoro aluminio borosilicate (<1m) Highly dispersed silicon dioxide (10–20nm)

CONCLUSION

CONCLUSION:

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. Ormocer and nanofill preheated composites displayed the highest degree of conversion and surface hardness.

3. Bulk fill nanohybrid preheated composites showed the lowest degree of conversion and microhybrid showed the lowest surface hardness.

4. Preheated composites exhibited poor internal marginal adaptation than the room temperature composites.

5. Among them, bulk fill nanohybrid and Ormocer composites showed higher frequency of gap formation with more MQ4 scores.

SUMMARY

This *in vitro* study was done to evaluate the degree of conversion, marginal adaptation and surface hardness of four different composite resins namely bulk fill nanohybrid, nanofill , ormocer and microhybrid compositesbefore and after preheating the composites.

160 composite blocks were prepared for this study using split mold. Among them, 80 composite blocks were taken for degree of conversion analysis with FTIR and another 80 blocks were taken for surface hardness analysis with Vicker hardness tester. The samples were broadly divided into two groups (Preheated composites and room temperature composites) and preheating was carried out at 61ºc using delta composite warmer. Degree of conversion was determined by powdering the composite blocks and mixed with potassium bromide to create a thin disc which was placed inside the cell holder of FTIR spectrometer. The changes in the peak height ratio of aliphatic C= C bonds at 1637cm $^{-1}$ was compared against an internal standard of aromatic C = C bond at 1610 cm $^{-1}$before and after polymerization.

Surface hardness was assessed on the top surfaces of each sample using a microhardness tester with a vicker pyramidal indenter under a 200g load for 30 seconds. Three indentations were carried out on the surface of each sample, 1mm away from each other. The average of 3 values was calculated as VHN value for each sample. Likewise, surface hardness was assessed for 80 composite blocks.

Eighty human maxillary premolars were selected for the marginal adaptation analysis. Class II cavities were prepared on the proximal surface of all the teeth and using typhodont jaw model, sectional matrix was placed followed by acid etching, bonding agent was applied and resored with the respective composites of 20 each. Composite resin were heated in the warmer and placed in the cavity in group 1. The

teeth were then sectioned in mesiodistal direction and axial adaptation was assessed with SEM at 200X magnification. Qualitative analysis was performed with the Blunck and Zaslansky scoring criteria. Width of the gap was measured with Image J analysis software.

Data were analyzed statistically using SPSS version 21 software for Windows. Data were expressed in its mean and standard deviation and were analyzed using ANOVA and Kruskal Wallis test.

The results of the study showed that there was a significant increase in degree of conversion and surface hardness for all the preheated composites tested when compared with the room temperature composites (p value < 0.05). Ormocer (74.35%) displayed the highest conversion mean values and bulk fill nanohybrid (44.44%) the lowest. Nanofill composite (110.58 VHN) displayed the highest hardness mean values and microhybrid (58.32 VHN) the lowest. But marginal adaptation was poor in preheated composites with the high frequency of MQ4 scores.

Within the limitations of the present study, it could be concluded that all the four composites used in the study exhibited significant increase in degree of conversion and surface hardness after preheating. Among them, Ormocer and Nanofill composites exhibited better results. Preheating results in more gap formation with the bulk fill nanohybrid and Ormocer showed higher MQ4 scores than the room temperature composites.

BIBLIOGRAPHY

1. Walls AW, McCabe JF, Murray JJ. The polymerization contraction of visible-light activated composite resins. *J Dent 1988;16(4):177-81.*

2. Davidson CL, de Gee AJ. Relaxation of polymerization contraction stresses by flow in dental composites. *J Dent Res 1984;63:146-8.*

3. Meredith N, Setchell DJ. In vitro measurement of cuspal strain and displacement in composite restored teeth. *J Dent 1997;25:331-7.*

4. Davidson CL, de Gee AJ, Feilzer A. The competition between the composite-dentin bond strength and the polymerization contraction stress. *J Dent Res 1984;63:1396.*

5. Ferracane JL, Greener EH. The effect of resin formulation on the degree of conversion andmechanical properties of dental restorative resins. *J Biomed Mater Res 1986;20:121-31.*

6. Braem M, Davidson CL, Vanherle G, Van Doren V, Lambrechts P. The relationship between testy methodology and elastic behavior of composites. *J Dent Res 1987;66:1036-9.*

7. Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. *J Dent Res 1987;66:1636-9.*

8. Giachetti L, Scaminaci Russo D, Bambi C, Grandini R. A review of polymerization shrinkage stress: Current techniques for posterior direct resin restorations. *J Contemp Dent Pract 2006;4:079 – 088.*

9. Braga RR, Hilton TJ, Ferracane JL. Contraction stress of flowable composite materials and their efficacy as stress-relieving layers. *J Am Dent Assoc 2003;134:721-8.*

10. Nívea Regina Fróes-Salgadoa, Luciana Maria Silvaa, Yoshio Kawanob, Carlos Franccia,Alessandra Reisc, Alessandro D. Loguercio. Composite pre-heating: Effects on marginal adaptation, degree of conversion and mechanical properties. *Dent mater 2010;26:908–14.*

11. de Almeida, Martins Mendes et al. Influence of preheating and post-curing on a novel fiber-reinforced compositepost material. *Braz. Oral Res. 2018;32:e97*

12. Yang Joshua Ng Chor. "Effects of Preheated Composite on Micro Leakage-An in-Vitro Study". *JCDR 10.6 (2016): ZC36-ZC38.*

13. M Par, O Gamulin , D Marovic, E Klaric , Z Tarle. Raman Spectroscopic Assessment of Degree of Conversion of Bulk-Fill Resin Composites – Changes at 24 Hours Post Cure. *Oper Dent 2015; 40(3): 92-101.*

14. RE dos Santos, AF Lima, GP Soares, GM Marchi, JR Lovadino. "Effect of Preheating Resin Composite and Light-Curing Units on the Microleakage of Class II Restorations Submitted to Thermocycling". *Oper Dent 2011;36(1): 60-65.*

15. Lucey S, Lynch CD, Ray NJ, Burke FM, Hannigan A. Effect of pre-heating on the viscosity and microhardness of a resin composite. *J Oral Rehabil. 2010 Apr;37(4):278-82.*

16. WC Wagner, MN Asku, AL Neme, JB Linger, FE Pink, S Walker. Effect of Pre-heating Resin Composite on Restoration Microleakage. *Oper Dent 2008; 33(1): 72-78.*

17. John S. Blalock, Robert G. Holmes, Frederick A. Rueggeberg. Effect of temperature on unpolymerized composite resin film thickness. *J Prosthet Dent 2006;96(6):424- 32.*

18. Knight JS, Fraughn R, Norrington D. Effect of temperature on the flow properties of resin composite. *Gen Dent. 2006 Jan-Feb;54(1):14-6.*

19. M. Daronch, F.A. Rueggeberg and M.F. De Goes. Monomer Conversion of Pre-heated Composite. *J Dent Res 2005; 84(7):663-7.*

20. Jeffrey W. Stansbury, Marianela Trujillo-Lemon, Hui Lu, Xingzhe Ding. Conversion-dependent shrinkage stress and strain in dental resins and composites. *Dent Mater 2005; 21: 56–67.*

21. Peutzfeldt A, Asmussen E., Investigations on polymer structure of dental resinous materials. *Trans Acad Dent Mater 2004; 18:81-104.*

22. Lovell LG, *et al.*, The effect of cure rate on the mechanical properties of dental resin. *Dent Mater 2001;17:504-11.*

23. Lovell LG, *et al.*, Understanding the kinetics and network formation of Dimethacrylate dental resins. *Polym. Adv. Technol .2001;12: 335-45.*

24. Zrinka Tarle, Andrej Meniga, Jozo Šutalo. Correlation Between Degree of Conversion and Light Transmission Through Resin Composite Samples. *Acta Stomatologica. Croatica 1995; 29: 9— 14.*

25. Ferracane JL., Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dent Mater 1985;1: 11-14.*

26. Wayne D. Cook, Derrick R. Beech, Martin J. Tyas. Resin-based restorative materials—a review. *ADJ 1984;24(5): 291-5.*

27. Bausch JR, de Lange C, Davidson CL. The influence of temperature on some physical properties of dental composites. *J Oral Rehabil. 1981 Jun;8(4):309-17*

28. Tobias T. Tauböck, Franziska Jäger, Thomas Attin. Polymerization shrinkage and shrinkage force kinetics of high- and low-viscosity dimethacrylate- and ormocer-based bulk-fill resin composites. *Odontology. 2019 Jan;107(1):103-10.*

29. Gayatri C, Rambabu T, Sajjan G, Battina P, Priyadarshini MS, Sowjanya BL. Evaluation of marginal adaptation of a self-adhering flowable composite resin liner: A scanning electron microscopic study. *Contemp Clin Dent 2018;9:S240-5.*

30. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G.Progress in dimethacrylate-based dental compositetechnology and curing efficiency. *Dent Mater 2013;29:139–56.*

31. Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent. 2006;18(6):340-50.*

32. da Costa J, McPharlin R, Hilton T, Ferracane J. Effect of heat on the flow of commercial composites. *Am J Dent. 2009 Apr;22(2):92-6.*

33. Sandro Aurélio de Souza Ventera, Silvia Luciana Fávarob, Eduardo Radovanovicb, Emerson Marcelo Girotto. Hardness and Degree of Conversion of Dental Restorative Composites Based on an Organic-inorganic Hybrid. *Materials*

Research. 2013; 16(4): 898-902.

34. Tahereh-Sadat Jafarzadeh, Mohammad Erfan, Marjan Behroozibakhsh, Mostafa Fatemi, Reza Masaeli, Yashar Rezaei, Hossein Bagheri, Yasaman Erfan. Evaluation of Polymerization Efficacy in Composite Resins via FT-IR Spectroscopy and Vickers Microhardness Test. *J Dent Res Dent Clin Dent Prospect 2015; 9(4):226-32.*

35. Silva EMd, Almeida GS, Poskus LT, Guimarães JGA. Relationship between the degree of conversion, solubility and salivary sorption of a hybrid and a nanofilled resin composite. *J Appl Oral Sci. 2008;16(2):161-66.*

36. Vipin Arora, Pooja Arora, Ammar Al Shammrani, Mohammed Khalil Fahmi. Devices & Methods for pre-heating/prewarming Dental Resin Composites: A Critical Appraisal. *Int J Oral Health Med Res 2017;4(2):52-5.*

37. Friedman J. Heating assembly for preheating dental materials. US patent 11.6236020. 2001

38. M D'Amario, F De Angelis, M Vadini, N Marchili, S Mummolo, C D'Arcangelo. Influence of a Repeated Preheating Procedure on Mechanical Properties of Three Resin Composites. *Oper Dent 2014; 39(6): 181-89.*

39. D'Amario M, Pacioni S, Capogreco M, Gatto R, & Baldi M. Effect of repeated preheating cycles on flexural strength of resin composites. *Oper Dent 2013; 38(1): 33-38.*

40. Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized composite film thickness. *J Prosthet Dent 2006;96: 424-32.*

41. Labella R, Lambrechts P, Van Meerbeek B & Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. *Dent Mater Journal 1999; 15(2): 128-37.*

42. Kemp-Scholte CM & Davidson CL. Complete marginal seal of Class V resin composite restorations effected by increased flexibility. *J Dent Res 1990; 69(6):1240-43.*

43. TrujilloM, Newman SM& Stansbury JW. Use of near- IR tomonitor the influence of external heating on dental composite photopolymerization. *Dent Mater Journal 2004; 20(8):766-77.*

44. C Sabatini, U Blunck, G Denehy, C Munoz. Effects of preheated composites and flowable liners on class II gingival margin gap formation. *Oper Dent 2010; 35(6):663-71.*

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TAMIL NADU, INDIA – 627105

RDCEC No: 14/2017

ETHICAL CLEARANCE

The Study titled "**The Comparative Evaluation of the Degree of Conversion, Marginal Adaptation and Surface Hardness of four Different Composite Resins Before and After Preheating – An Invitro Study**" by **Dr. Mohanapriya R**, Department of Conservative Dentistry and Endodontics, Rajas Dental College & Hospital, on scrutiny by the Rajas Dental College Ethics Committee (RDCEC) has been given Ethical Clearance to conduct the study.

Recommended for a period of 3 years

Date of Review: 12/12/2017

Dr. Shyam Mohan A. M.D.S., D.N.B.
MEMBER SECRETARY
INSTITUTIONAL ETHICS COMMITTEE
RAJAS DENTAL COLLEGE
KAVALKINARU, TIRUNELVELI DIST

Note:

- Inform RDCEC immediately in case of any adverse events and serious adverse outcomes.
- Inform RDCEC in case of any change of study procedure, site and investigator.
- The permission is only for the period mentioned above.
- Annual report has to be submitted to RDCEC.
- Members of the IEC have the right to monitor the trial with prior intimation.

Address for Correspondence:
Dr. SHYAM MOHAN A, Member Secretary, Rajas Detal College Ethics Committee, Rajas Dental College & Hospital, Kavalkinaru, Tirunelveli District, Tamil Nadu, India – 627105
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CERTIFICATE – II

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