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ORIGINAL RESEARCH ARTICLE

Comparative Evaluation of Degree of Conversion of Three Bulk Fill Composite Resins by Fourier Transform Infrared Spectroscopy-Attenuated Transmittance Reflectance (FTIR-ATR)

Namrata Khanna, BDS, MDS[1], Gowrish S, BDS, MDS[2*], Vandana Sadananda BSc, MSc, BDS, MDS[3*]

[1,2,3] Department of Conservative Dentistry and Endodontics, A. B. Shetty Memorial Institute of Dental Sciences, Deralakatte, Mangaluru - 575018, Nitte (Deemed to be University), Karnataka, India

*Corresponding author.Email: drgowrishs@nitte.du.in, drvandanasadananda@nitte.edu.in

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Abstract

Introduction: Since the introduction of resin based composite restorative materials, various improvements have been made to improve their physico-mechanical properties. In an attempt to fasten and simplify the restoration process, a new class of resin based composite materials, called the bulk-fill resin based composites have been introduced, that claim to achieve a depth of cure of 4 mm without affecting the properties of the material. This study evaluated the degree of conversion (DC) of monomer to polymer using Fourier Transform Infrared Spectroscopy-Attenuated Transmittance Reflectance (FTIR-ATR).

Methodology: DC of monomer to polymer of the following were analysed using FTIR-ATR (IRPrestige-21, Shimadzu). Group 1: Bis-GMA based Nano-hybrid bulk-fill material (Tetric N-Ceram Bulk Fill, Ivoclar Vivadent, AG, Liechtenstein), Group 2: UDMA based posterior bulk-fill flowable resin material (Smart Dentin Replacement, SDR™, Dentsply, Konstanz, Germany), Group 3: Nano-hybrid based bulk-fill posterior restorative material (Filtek™ Bulk Fill Posterior Restorative material, 3M™ ESPETM, St. Paul, USA). 10 samples were fabricated for each group. The results obtained were statistically analysed using One-way ANOVA and Scheffe Test.

Results: Group 2, exhibited highest DC (45.16%) followed by Group 3 at 37.86% and Group 1 l at 31.05%.

Conclusion: Highest values of DC may be attributed to the high molecular weight of the monomer and less viscosity. Presence and amalgamation of low viscosity but heavier monomers may be ideal for a bulk-fill resin composite restorative material to provide successful outcome clinically.

Keywords: Degree of conversion, monomer, polymer, bulk-fill composites, FTIR-ATR

Introduction

Resin based composite materials have changed the scenario of restorative dental procedures which can be attributed to their superior aesthetic properties and minimal tooth preparation as compared to the more traditional restorative alternatives. Manufacturing companies strive to achieve properties that may simulate properties of natural enamel and dentine. The structure of composite resin restorative materials has been refined and upgraded ever since their development in order to enhance their physico-mechanical properties(1).

The gold standard for composite resin restorative materials placement has always been the 2mm incremental technique, which is time consuming considering for both the clinician and the patient. To counter such a deficit, a new array of materials, termed the bulk-fill composite resins with claims by the manufacturers of permitting a 4 to 6 mm incremental placement and cure without influencing the properties of the resin structure, have been introduced into the market(2).

The resin structure as we know comprises of three principal constituents, the organic matrix, the organic filler and the coupling agent(3). The properties of the material are dependent on the properties of these basic constituent contents. The organic resin contains a system of functional monomers that could be mono-functional, di-functional or tri-functional and an initiator complex which most commonly is camphoroquinone along with a reducing agent (tertiary aliphatic amine)[4)]. These high molecular weight monomers show high viscosity that is diluted by the addition of lower molecular weight monomers such as those of urethane dimethacrylate (UDMA), bisphenol A-dimethacrylate (Bis-DMA), bisphenol A-glycidyl methacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA) and ethylene glycol dimethacrylate (EGDMA)[5,6)].

Degree of conversion (DC%) for a composite resin material can be defined as the percentage of carbon-carbon double bonds converted (-C=C-) to single bonds (-C-C-) to form a polymeric resin[2)]. The monomer in the resin does not achieve a 100% conversion while converting into a polymer which leads to the remainder of unsaturated free monomer producing restorations with inferior mechanical properties, as the uncured monomers act as plasticizers. The polymerization of the resin occurs as the monomer bonds link to form a polymer chain of cured resin.

Various methods of testing the DC% of composite resin materials have been utilized that perceive the stretching vibrations of the carbon bonds before and after polymerization has occurred in a resin. Fourier Transform Infrared Spectroscopy-Attenuated Transmittance Reflectance (FTIR-ART) leans on the fact that most molecules absorb light in the infra-red region of the electromagnetic spectrum that correspond specifically to the bonds present in the molecule. FTIR-ART in relation to composite resins, involves the assessment of the peak height ratio obtained by the aliphatic and aromatic carbon double bonds, by translating the data onto a specific graph[7)]. FTIR-ATR not only qualitatively analyses the material but also gives a quantitative measure of how much DC% has occurred after curing.

Turssi et al (2015) and Amirouche et al (2009) studied the effect of filler particle size, geometry and load on the DC% and concluded that DC% progressively decreases with the increasing opacity of the resin and that filler geometry does not have a significant impact on the final conversion[8-10)]. However, other studies did demonstrate that if the filler particle size is similar to that of the wavelength of the curing light, them it confers a negative impact on the DC% as more scattering of light by similar sized molecules takes place.

DC% depends on the internal structure of the just like any other mechanical property. The concentration of the monomer and photo-initiator, their structure and the conditions in which the polymerization occurred all play a significant role in the formation of the polymeric chain. Several studies have been done to assess these properties and their effect on the final DC%.

This research study aimed to evaluate the DC% for three bulk-fill composite resin restorative materials with different predominant constituent resin matrices, all of which claim to exhibit a depth of cure of 4mm by the manufacturing companies. This was tested using Fourier Transform Infrared Spectroscopy-Attenuated Transmittance Reflectance (FTIR-ATR).

Materials and Methods

The bulk-fill composite resin restorative materials were divided into three groups as follows: Group 1: Nano-hybrid bulk-fill material (Tetric N-Ceram Bulk-Fill, Ivoclar Vivadent, AG, Liechtenstein) containing bisphenol A-diglycidyl dimethacrylate (Bis-GMA), ethoxylated bisphenol A dimethacrylate (Bis-EMA) and urethane dimethacrylate (UDMA). The organic matrix accounts for approximately 1% of the mass. Several different types of fillers are incorporated in the material. Barium, aluminium silicate glass with two different mean particle sizes, an Isofiller comprising of cured dimethacrylates, ytterbium fluoride and spherical mixed oxide. It comprises of an overall standard filler content of approximately 75% by weight, 61% volume and 17% polymer fillers or Isofillers. Group 2: Posterior bulk-fill flowable resin material (Smart

Dentin Replacement, SDR^{TM}, Dentsply, Konstanz, Germany) SDR comprising of a complex formulation of patented urethane di-methacrylate resin (molecular weight of 849 g/mol), di-methacrylate resin, di-functional diluents, barium and strontium alumina-fluoro-silicate glasses (68% by weight, 45% by volume), photo initiating system and colourants. Group 3: Bulk-fill posterior restorative material ($Filtek^{TM}$ Bulk-Fill Posterior Restorative material, $3M^{TM}$ $ESPE^{TM}$, St. Paul, USA) containing fillers which comprise of a combination of non-agglomerated/non-aggregated 20 nm silica filler, a non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles) and an ytterbium trifluoride filler consisting of agglomerate 100 nm particles for increased radiopacity. The inorganic filler loading is about 76.5% by weight (58.4% by volume). Filtek bulk-fill contains aromatic dimethacrylate (AUDMA), urethane dimethacrylate (UDMA) and 1,12–dodecanediol dimethacrylate (DDDMA), 10 samples were fabricated for each group. The materials were expressed from their respective containers and packed in the stainless steel mold of height 4mm and diameter 3mm. The material was condensed into the custom made stainless steel mold. Excess material displaced and removed. The samples were covered with a mylar strip and polymerized using a blue light emitting diode light source (Kerr $Demi^{TM}$ Ultra LED Ultracapacitor Curing Light System, KaVo Kerr). The material was irradiated for the time recommended by the specific manufacturers to achieve optimum curing, Group 1: 10 seconds, Group 2: 20 seconds and Group 3: 20 seconds.

Immediately after curing, the specimens were removed from the mold followed by removal of uncured material with the help of a plastic spatula. The samples were stored in an air-tight opaque container. FTIR-ATR (IRPrestige-21, Shimadzu) analysis was done immediately and graphs were obtained.

The graphs obtained were analysed separately for each sample to calculate the degree of conversion using the following formula give below.

Degree of Conversion (DC%)=1- [H2/H1 polymerized/H2/H1 unpolymerized]x100

Where, H2 is the peak height absorbance intensity of carbon double bond peak, aliphatic, H1 is the peak height absorbance intensity of carbon double bond peak, aromatic.

Statistical analysis: The data collected was statistically analysed using Statistical Package for Social Sciences (SPS Inc., version 20.0 for Windows). Using this software mean and standard deviation was calculated for quantitative variables. One-way Analysis for Variance test was applied to verify the existence of statistical significance between group variations, with significance level being set at p-value <0.05. Since statistical significance was found, post-hoc comparison was done using Scheffe Test.

Results

The degree of conversion for each sample was calculated after implementing the above mentioned formula where H2 (aliphatic carbon double bond) and H1 (aromatic carbon double bond) was standardized for each group after calibration of the graphs obtained. (Figures 1-6)

Mean degree of conversion (DC%) values were highest for Group 2 at 45.16±4.18% followed by Group 3 at 37.86±3.37% and lastly Group 1 at 31.05±2.03%. One way Analysis of Variance test showed significant difference between all the three materials. Post hoc comparison was done between all three groups using Scheffe test since the One-way ANOVA test rendered significant results (Table 1).

The DC% between all the three groups against each other also differed rendering significance within the groups all of which were set at p<0.05 (Table 2).

Discussion

The success of a direct posterior composite restoration depends on several factors. Technique sensitivity and the time consumed by the incremental curing is concerning for most clinical practitioners. The polymerization shrinkage is greater in composite resin restorations if they are not placed incrementally, if a bulk increment of more than 2mm thickness is placed, there is reduced polymerization as the light from the curing lamp does not manage to reach the apical most parts of the restoration11,12). With the advent of bulk fill composite resins, the time taken to complete a restoration is far lesser as compared to that taken by traditional composites and so is the polymerization shrinkage stress with the bulk placement of 4mm composite resin due to altered chemistry and properties of the material13,14).

Bulk fill composite resins were more so introduced as flowable composites to act as lining agents in deep cavities without having to worry about the thickness of the increment placed prior to curing and achieving a greater depth of cure of close to 4-5mm. These materials showcased greater translucency, greater flexibility and greater levels of photo-initiator and the presence of more than one such photo-initiator; all of which helped improve the depth of cure and avoided the easy entrapment of air within cavities15,16).

As mentioned, a sufficient degree of conversion is one such necessity to ensure the restoration has a successful outcome and the DC% acts as important factor in determining the physical, chemical, mechanical and biological properties of composite resin restorations. The clinical success and DC% also largely depends on adequate light curing of the composite resin; being the time used to irradiate the resin, irradiation distance, method of light activations used and the light power density and wavelength of the curing lamp required for polymerization17).

Numerous techniques have found their use pertaining to calculating the DC% of composite resins. In spite of FTIR-ATR being the most commonly used technique, other tests that can be employed include – Raman Spectroscopy, Electron Paramagnetic Resonance, and Differential Thermal Analysis18,19). FTIR-ATR and Raman Spectroscopy have also been used to detect water sorption in the past amongst dental composites and are powerful tools to analyse and monitor polymerization and setting reactions for several dental materials20).

Adequate depth of cure and degree of conversion are vital properties of any composite resin to ensure that enough carbon double bonds have broken down to single bonds in forming a polymeric chain even in the most apical regions of the restoration. FTIR-ATR not only identifies the presence of organic groups but also directly detects and measures the quantity of the same in the resin matrix2).

In a study by Jerri et. Al, a comparison between the DC% of incremental composites along with bulk fill composites was done with the bulk fill composites showing a fairly higher degree of conversion rate. It was concluded by other studies by Acquaviva PA et el, Abed et al, Irini et al; and also that bulk fill composite resin constitute of modifiers that attribute special properties to the material allowing for a higher conversion of carbon bonds21,22). The complex structure of these predominantly Bis-GMA based resins lead to successful polymerization of bonds between 45-85%.

The different values of DC% that were obtained could be attributed the variation in chemistry in the matrix of the resin structure since all the conditions under which the study was performed followed a standardized protocol and manufacturer's instructions. According to Siderou et al, the DC% reduced in the following manner for various monomers being: TEGDMA>UDMA>Bis-EMA>Bis-GMA23).

The DC% usually depends on two important factors, the flexibility of the resin structure and the viscosity. A highly viscous and rigid monomeric compound will exhibit a lower conversion rate. Bis-GMA shows high intramolecular hydrogen bonding considering the hydroxyl ion groups and the aromatic ring in its structure. UDMA also consists of an imino group and carbonyl group, both of which show interactions between the hydrogen bonds24). UDMA is not as viscous as Bis-GMA however, and this is due to its higher molecular weight and the presence of a weaker hydrogen bond in the imino group and compared to the hydrogen bond in hydroxyl groups25).

The high viscosity of Bis-GMA reduces its potential for achieving a better DC% and hence, manufacturers have shifted towards the usage of other monomers which are less viscous and more flexible. UDMA has been added in larger amounts in SDR to counteract the disadvantage conferred by Bis-GMA in such cases and thus improving the degree of conversion of the resin. The presence of the imino groups in UDMA increase its reactivity by causing specific chain transfer reactions that aid in polymerization. The radical sites show higher mobility and conversion within the network.

Other monomers with lesser viscosity include TEGMDA and ethoxylated EBPDMA which are less rigid and more hydrophobic. The hydrophilic nature of Bis-GMA attracts more uptake of water that can lead to eventual degradation and discolouration. Bis-GMA based BFCs perform better when these hydrophobic and less viscous monomers are substituted in its place to give a better DC%. The rate of polymerization is improved effectively and the network is more flexible and plasticized26).

In a study done by Monterubbianesi et al the DC% of BFCs (Group 3 - FBF and Group 2 - SDR in this study) was correlated with the curing done using two different curing lights. Elipaar S10 (3M ESPE) and Demi Ultra (KaVo Kerr) were the two curing lamps used. FBF showed a significantly greater DC% immediately after curing (on the top and bottom of the sample) and also after 24 hours when cured with Demi Ultra as opposed to Elipars10. However SDR showed better DC% when cured by Elipars1027).

The structure of Group 3 material has been modified by the addition of a monomer Procrylat, which has a high molecular weight but also less viscosity. Procrylat lacks the less reactive hydroxyl groups which decrease the potential for bonding as seen in the Bis-GMA resin. The absence of the hydroxyl ions in the procrylat helps in better cross linking and chain formation in the polymer. BisMEA and UDMA are also present in the structure and are more reactive as compared to Bis-GMA while also having a high molecular weight28).

The initial DC% for bulk-fill composite resins is usually lesser and improves considerably in the first hour and by the end of 24 hours. It is affected by various factors such as the chemical moiety of the monomers present, the filler particle load within the resin, filler size and geometry, the other compounds used to modify the resin and the type of photo-initiator used29).

The addition of polymerization inhibitors is necessary to stall the reaction from occurring far too quickly causing higher levels of polymerization shrinkage stress. The intensity of the light from the curing lamp also contributes to the rate at which polymerization can occur. Bulk-fill composite resins show less DC% initially but lesser polymerization shrinkage stress overall in comparison with conventional composite resins which may be the effect of the alterations done to the chemical structures of the former1).

Group 1 showed the least DC% in this study and that could be attributed to the fact that it utilizes a higher concentration and ratio of Bis-GMA to other monomers. Group 1 resin contains Ivocerin, a photo-initiator (patented by Ivoclar Vivadent) and requires 10 seconds of curing by a high power output curing lamp. Ivocerin is a sensitive photo-initiator that makes the resin more likely to commence (methoxybenzoyl) diethylgermanium) is a combination photo-initiator that unites champhorquinone and acyl phosphine oxide. It is a dibenzoylgermanium derivative that when irradiated by visible light, disintegrates and produces benzoyl and germyl free radicals that initiate the polymerization reaction. Hence, Ivocerin initiates the reaction by itself and is not depended on the presence of another co-initiator molecule30).

A study done by Alshali et al, led them to conclude that all bulk-fill composite resins (except FBF) showed an increase in the DC% at 24 hour after curing was done when stored at 37 degree Celsius as compared to the values obtained immediately after irradiation1). Studies done also confirm that while the polymerization reaction in a resin is occurring, there is a race between chain formation and cyclization and eventually chain formation becomes more dominant and even a slight increase in the DC% eventually reflects positively on the overall mechanical properties and clinical performance of the restoration.

Generally, bulk-fill composite resins are more translucent as compared to conventional composite resins since a greater depth of cure and curing efficiency is obtained at a higher incremental depth of 4mm. The higher the opacity of the filler particles, the lesser the translucency and lesser the polymerization at the apical end of the restoration. Light transmission is better in bulk-fill composite resins with less filler content ratio. However, the compromise on the filler content will reduce the strength and elastic modulus of the restoration even though a high filler content is an obstruction in the way of sufficient polymerization31).

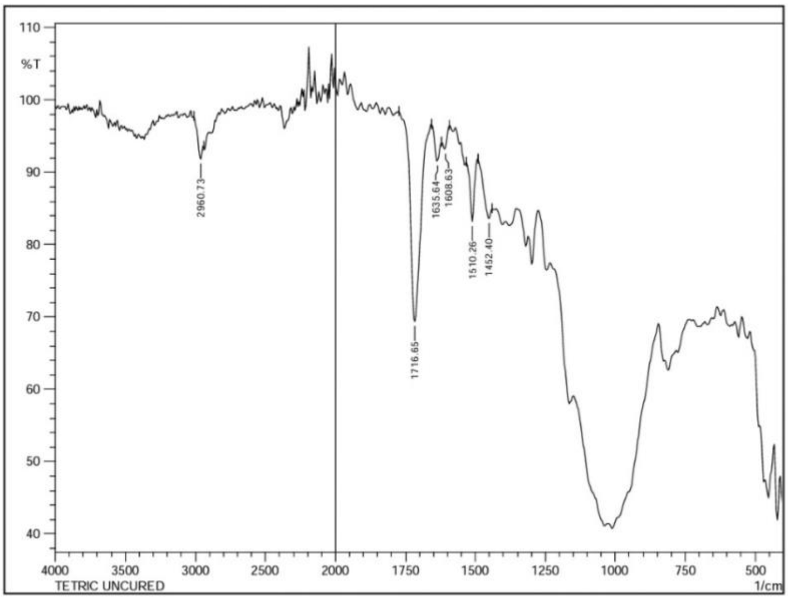
The co-polymerization of different monomers of high molecular weight help in heavier cross linking and chain formation with denser and stiffer polymer networks like with the combination of Bis-GMA with that of UDMA and TEGDMA. In spite of the presence of Bis-GMA in Group 3, it still showed reasonable DC% due to the dilution of the resin matrix with other monomers like Bis-EMA, UDMA and Procrylat. Group 1 on the other hand, consists of 75-77% filler percentage and is the least translucent amongst the three materials with less DC% along with its higher concentration of the Bis-GMA – a molecule with decreased mobility and viscosity. Group 2 material has a filler loading of about 68% and Group 3 that of, 65% which could also be the reason of better DC% in the two materials.

Conclusion

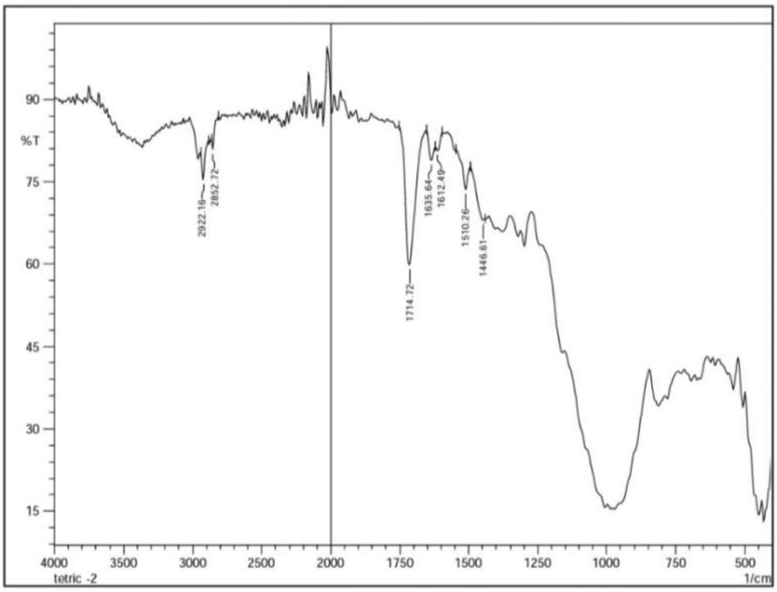
Statistically significant differences were obtained in this study in the DC% values of the three groups. It was concluded that the predominant monomer conversion was highest for the UDMA based resin > nanohybrid resin > Bis-GMA based resin.

The UDMA based resin showed the highest values of DC% and that could be attributed to the high molecular weight of the monomer but comparatively, lesser viscosity. A more flexible monomeric molecule will polymerize much faster than a rigid one like Bis-GMA. The presence and amalgamation of such low viscosity but heavier monomers is more ideal for a bulk fill composite and will provide a better and successful outcome clinically. The light intensity and time taken to irradiate a sample is also sensitive to the overall degree of conversion. Bulk-fill composite resins are certainly a boon in the clinical scenario. Care has to be taken to provide an adequate irradiation time and avoiding over increasing the increment thickness.

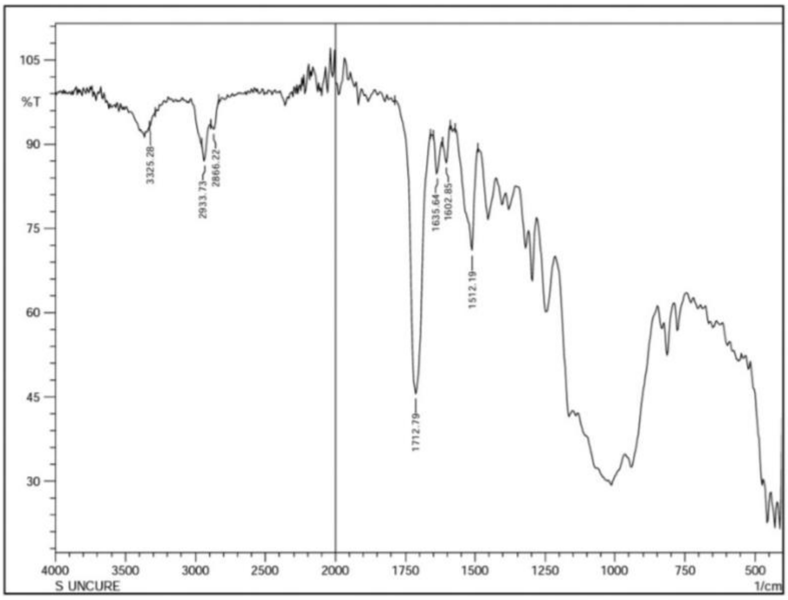
FIGURES AND TABLES



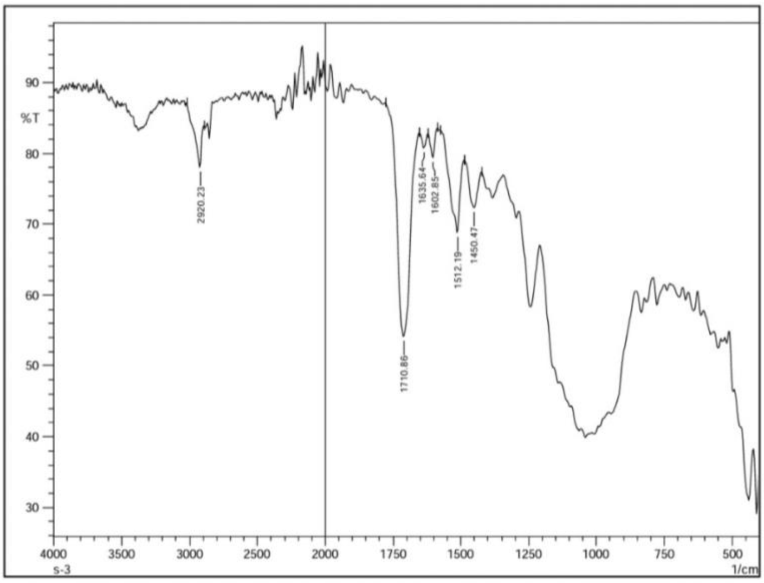
Graph 1: Group 1 prior to polymerization with H2 (aliphatic carbon at 1635 cm^{-1} and H1 (aromatic carbon) at 1716 cm^{-1}.



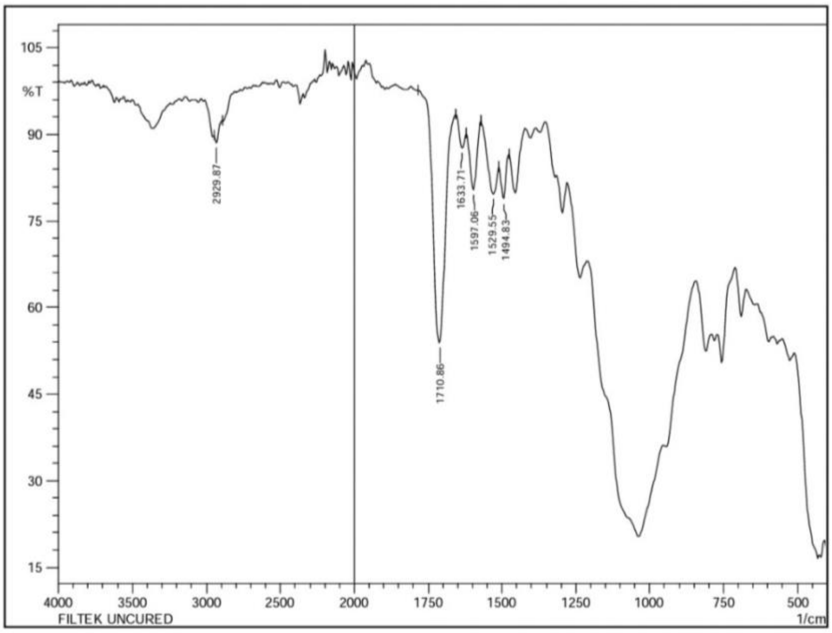
Graph 2: Group A subsequent to polymerization with H2 (aliphatic carbon) at 1635 cm^{-1} and H1 (aromatic carbon) at 1714 cm-



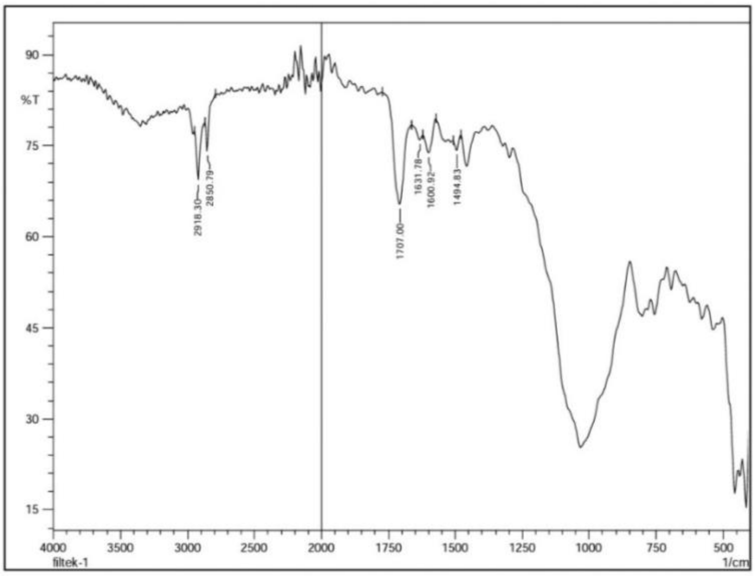
Graph 3: Group 2 before polymerization with H2 (aliphatic carbon) at 1635 cm^{-1} and H1 (aromatic carbon) at 1712 cm^{-1}



Graph 4: Group B after polymerization with H2 (aliphatic carbon) at 1635 cm-1 and H1 (aromatic carbon) at 1710 cm^{-1}



Graph 5: Group 3 with H2 (aliphatic carbon) at 1633 cm^{-1} and H1 (aromatic carbon) at 1710 cm^{-1} before polymerization



Graph 6: Group 3 with H2 (aliphatic carbon) at 1631 cm^{-1} and H1 (aromatic carbon) at 1707 cm^{-1} after polymerization

Significant results were obtained overall and within the groups. (Table 1 and Table 2.)

Table 1: Mean and standard deviation of degree of conversion (DC%) values for all three groups.

Material	N	Mean	Standard Deviation	Minimum	Maximum
Group 1	10	31.054	2.038	27.61	34.19
Group 2	10	45.168	4.183	37.14	52.38
Group 3	10	37.868	3.375	32.57	43.28

1)Mean degree of conversion (DC%) values were highest for Group 2 at 45.16±4.18% followed by Group 3 at 37.86±3.37% and lastly Group 1 at 31.05±2.03%. One way Analysis of Variance test showed significant difference between all the three materials. Post hoc comparison was done between all three groups using Scheffe test since the One-way ANOVA test rendered significant results.

Table 2: Multiple Intergroup Comparisons for Degree of Conversion (DC%) using Scheffe Test wherein the dependent variable was the DC%

(I) Group	(J) Group	Mean Difference (I-J)	p-value	95% Confidence Interval	
				Lower Bound	Upper Bound
Group 1	Group 2	-14.144	<0.05	-17.95	-10.26
	Group 3	-6.814		-10.65	-2.969
Group 2	Group 3	-7.30		-11.14	-3.455

2)The DC% between all the three groups against each other also differed rendering significance within the groups all of which were set at $p<0.05$

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