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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF DEGREE OF CONVERSION AND VOLUMETRIC SHRINKAGE OF TWO DIFFERENT BULK FILL COMPOSITES, ONE SHORT FIBER REINFORCED COMPOSITE AND A NEW NANOHYBRID COMPOSITE- AN IN VITRO STUDY

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ABSTRACT

Introduction: The physical and mechanical properties of composite resins have been greatly improved over the past couple of years. However, they still have some shortcomings like an insufficient depth of cure, degree of conversion and volumetric shrinkage. **Objective:** The purpose of this study was to comparatively evaluate for percentage degree of conversion (DC%) and volumetric shrinkage(VS%) of two bulk fill composite materials, a SFRC and a new Nanohybrid material. **Method:** A Total of 180 samples were prepared and divided into four groups i.e., Group A (Aura Bulkfill), Group B (Tetric Evocerambulk fill), Group C (Ever X Posterior) and Group D (Harmonize) comprising of 45 samples each. For Group A, samples were further divided into two sub groups. Subgroup A1. For evaluation of DC% (n=30). Subgroup A2. For evaluation of VS(n=15). In Subgroup A1, thirty samples were prepared by cutting teflon tube at two different heights viz; 2mm (n=15) and 4 mm (n=15). These samples were filled with composite in a single increment followed by light curing. Degree of conversion analysis was done with the help of FTIR spectroscope. In Subgroup A2, Fifteen Samples (n=15) were prepared by filling the composite in a stainless-steel mould of 4mm diameter and 4 mm height. These were weighed before and after light curing with the help of an analytical balance. The specific gravity was calculated by using ASTM D-792 method. The volumetric shrinkage was calculated by measuring the difference in specific gravities of uncured and cured samples by mathematical equations. Similar procedures were followed for other Subgroups. However, In Subgroup D1 and D2, 2mm increment of composite material was placed and light cured which was unlike the other tested bulkfills, which were placed in a single increment of 4 mm. **Results**: EP exhibited highest degree of conversion at both 2mm and 4 mm heights, followed by TEC and AB. Harmonize showed almost similar DC% values at both 2mm and 4mm heights. At 4mm height, DC% of Harmonize was found to be better than AB and TEC. VS% was observed in the order: EP< TEC< AB< Harmonize. **Conclusion**: BFCs in the present study performed better in terms of DC % at 2 mm height and VS when compared to the newly introduced nanohybrid composite. However, at 4mm height, DC% of Harmonize was observed to be better than AB and TEC, which can be due to its incremental placement and curing of 2mm each.

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INTRODUCTION

Light-cured resin composite has emerged as the most widely used dental restorative material due to its good aesthetics, biocompatibility, and mechanical properties. However, they still have some shortcomings like an insufficient depth of cure and volumetric shrinkage. The volumetric shrinkage of composite resins occurs following the conversion of monomer molecules to a polymer structure through the replacement of van der Waals spaces with covalent bonds, resulting in closer packing of the molecules leading to bulk contraction. While most composite materials shrink between 1.35% to 7.1%, the average range is between 2-3%. The degree of conversion could be defined as the extent to which monomers react to form polymers or as the degree to which carbon double bonds (C = C) are converted into carbon single bonds (C — C**).** Physical and mechanical properties of dental composites are directly influenced by the degree of conversion achieved during polymerization. Lower degree of conversion results in composites with inferior mechanical properties, greater discoloration and degradation. Aim of this study wasto comparatively evaluate for degree of conversion and volumetric shrinkage of two bulk fill composite materials, a short fibre reinforced composite and a new Nanohybrid material which has a unique feature of Adaptive Response Technology.

MATERIALS AND METHODS

This in-vitro study was conducted in the Department of Conservative Dentistry and Endodontics of Government Dental College and Hospital, Patiala. For the present study, two Bulk fill composites, one fiber reinforced composite material and one nanohybrid material was used for evaluating Degree of conversion and Volumetric shrinkage. Degree of conversion analysis was done at Chemistry Department of Punjabi University, Patiala and volumetric shrinkage analysis was done at Thapar University, Patiala.

Materials used

- **Aura Bulkfill** (SDI, Melbourne, Victoria, Australia)
- **Tetric Evoceram Bulkfill** (IvoclarVivadent, Schaan, Liechtenstein)
- **Ever X Posterior** (GC, Tokyo, Japan)
- **4. Harmonize** (Kerr dental, USA)

A Total of 180 samples were prepared and divided into four groups: Group A (Aura Bulkfill), Group B (Tetric Evocerambulkfill), Group C (Ever X Posterior) and Group D (Harmonize) comprising of 45 samples each. For Group A (Aura Bulkfill) samples were divided into two sub groups

Subgroup A1. For evaluation of Degree of conversion (n=30)
Subgroup A2. For evaluation of Volumetric shrinkage (n=15)
For Subgroup A1, Thirty samples were prepared by cutting teflon tube at two different heights viz; 2mm (n=15) and 4 mm (n=15). These samples were filled with composite followed by light curingof sample with LED light curing unit (Mini-LED, Acteon Satelec, Merignac, France) with continuous monitoring of light intensity by radiometer. After light curing, the specimens were stored in opaque light proof containers at room temperature for 24 hours.

Degree of conversion analysis was done with the help of FTIR spectroscope (Perkin Elmer, Spectrum Two, FT-IR, CT, USA). For Subgroup A2, Fifteen Samples (n=15) were prepared by filling the composite in a stainless-steel mould of 4mm diameter and 4 mm height. These samples were weighed before and after light curing with the help of a Commercial density determination kit of the analytical balance(XS105, Mettler Toledo, Greifensee, Switzerland). The specific gravity was calculated by using ASTM D-792 method. The volumetric shrinkage was calculated by measuring the difference in specific gravities of uncured and cured samples by mathematical equations. Similar procedure was followed for Group B (Tetric Evoceram), Group C (Ever X Posterior) and Group D (Harmonize). However, In Subgroup D1 and Subgroup D2, 2mm increment of composite material was placed and light cured which was unlike the other tested bulkfills, which were placed in a single increment of 4 mm.

RESULTS

For pairwise comparison between different subgroups, Tukey's post hoc test was performed.

Table 1. Shows Comparison of mean percentage degree of conversion (DC%) at height of 2mm of all the groups

Subgroups	Mean	Standard Deviation	P value	Significance
Subgroup A1 (Aura Bulkfill)	56.0313	2.62902	<0.001	HS
Subgroup B1 (Tetricevoceram)	59.7882	2.10713		
Subgroup C1 (Ever X posterior)	63.9271	2.84896		
Subgroup D1 (Harmonize)	57.3363	2.94149		

Table 2. Shows Comparison of mean percentage degree of conversion (DC%) at height of 4mm of all the groups

Groups	Mean	Standard Deviation	P value	Significance
Subgroup A1 (Aura Bulkfill)	48.5392	3.13246	<0.001	HS
Subgroup B1 (Tetricevoceram)	51.7973	2.85393		
Subgroup C1 (Ever X posterior)	59.3555	3.03337		
Subgroup D1 (Harmonize)	55.0258	3.14114		

Table 3 Shows Comparison of mean Volumetric shrinkage of all the groups

Groups	Mean	Standard Deviation	P value	Significance
Subgroup A2 (Aura Bulkfill)	2.7193	.32139	<0.001	HS
Subgroup B2 (Tetricevoceram)	2.5947	.39993		
Subgroup C2 (Ever X posterior)	2.2180	.36297		
Subgroup D2 (Harmonize)	2.9127	.45082		

DISCUSSION

Resin-based composites have been successfully used in dentistry for many years and have widely replaced amalgam as a posterior restoration.

Table 4. Shows Pairwise Comparison of mean percentage degree of conversion (DC%) at height of 2mm among the subgroups

Subgroups	Mean	Standard Deviation	P value	Significance
Subgroup A1 &	56.0313	2.62902	0.002	HS*
Subgroup B1	59.7882	2.10713		
Subgroup A1 &	56.0313	2.62902	<0.001	HS
Subgroup C1	63.9271	2.84896		
Subgroup A1 &	56.0313	2.62902	0.537	NS*
Subgroup D1	57.3363	2.94149		
Subgroup B1 &	59.7882	2.10713	<0.001	HS
Subgroup C1	63.9271	2.84896		
Subgroup B1 &	59.7882	2.10713	0.066	NS
Subgroup D1	57.3363	2.94149		
Subgroup C1 &	63.9271	2.84896	<0.001	HS
Subgroup D1	57.3363	2.94149		

Table 5. Shows Pairwise Comparison of mean percentage degree of conversion (DC%) at height of 4mm among the subgroups

Subgroups	Mean	Standard Deviation	P value	Significance
Subgroup A1 &	48.5392	3.13246	0.024	S*
Subgroup B1	51.7973	2.85393		
Subgroup A1 &	48.5392	3.13246	<0.001	HS*
Subgroup C1	59.3555	3.03337		
Subgroup A1 &	48.5392	3.13246	<0.001	HS
Subgroup D1	55.0258	3.14114		
Subgroup B1 & Subgroup C1	51.7973	2.85393	<0.001	HS
	59.3555	3.03337		
Subgroup B1 &	51.7973	2.85393	0.026	S
Subgroup D1	55.0258	3.14114		
Subgroup C1 &	59.3555	3.03337	0.001	HS
Subgroup D1	55.0258	3.14114		

Table 6. Shows Pairwise Comparison of mean Volumetric shrinkage among the subgroups

Subgroups	Mean	Standard Deviation	P value	Significance
Subgroup A2 &	2.7193	.32139	0.814	NS*
Subgroup B2	2.5947	.39993		
Subgroup A2 &	2.7193	.32139	0.004	HS*
Subgroup C2	2.2180	.36297		
Subgroup A2 &	2.7193	.32139	0.524	NS
Subgroup D2	2.9127	.45082		
Subgroup B2 &	2.5947	.39993	0.048	S*
Subgroup C2	2.2180	.36297		
Subgroup B2 &	2.5947	.39993	0.122	NS
Subgroup D2	2.9127	.45082		
Subgroup C2 &	2.2180	.36297	<0.001	HS
Subgroup D2	2.9127	.45082		

*HS - Highly Significant, *NS - Non significant, *S – Significant

Dental composites are expected to have mechanical properties comparable to those of tooth enamel and dentin and provide a long life of service. Abed YA, Sabry HA &Alrobiegy NA (2015). Currently, there is a growing trend among practitioners to use bulk-fill resin based composite materials because of their more simplified procedures. Bulk fill composites may be adequately polymerized at greater thicknesses, but the differences, both in the chemical composition of the resin matrix and in the type of filler particle significantly affect the DC and the mechanical properties of the materials (Kubo et al 2018). The degree of conversion is an important tool to estimate the physical, mechanical and biological properties of composite resin restorations. Higher degree of polymerization is an essential factor for obtaining superior physical and mechanical properties. Some authors found similar or higher DC and lower shrinkage of bulk-fill materials at 4 mm thickness. On the other hand, others revealed a significant decrease in degree ofconversion in BFCs at 4 mm thickness or higher volumetric shrinkage than that of conventional composites. Thus, the clinical implications of the use of these new materials seem unclear. Current study included two Bulkfill composites and one SFRC which, like other bulkfill composites, can be placed and light cured in a single 4mm increment, as recommended by the manufacturer. Conventional RBCs were not tested in a layer thickness of 4 mm, rather we tested nanohybrid composite with 2 increments of 2 mm each (as recommended by the manufacturer) because it provided more data to estimate the effective size of the investigated variables. FTIR was chosen in this study to analyse DC% during this study because it is an efficient and commonly used technique. It provides a quantitative measure of the number of carbon double bonds that are converted to single bonds which reflect the DC% and effect of photopolymerization (Balagopal S et al, 2021). Despite continued advancement in adhesive dentistry, volumetric shrinkage of resin composites still remains a major challenge.

Dental composite resins primarily consist of a dimethacrylate resin filled with organic or inorganic filler particles and upon cure the dimethacrylate matrix undergoes a volumetric shrinkage which in turn causes mechanical failure at the composite tooth interface or the formation of marginal gaps permitting the ingress of bacteria into the cavity. Thus, low volumetric shrinkage is generally seen as a key performance indicator for this material genre Rao, Hegde and Shetty (2017). Bulk-fill resin composites are inhomogeneous and differ in terms of filler type, size and content, viscosity, and chemical

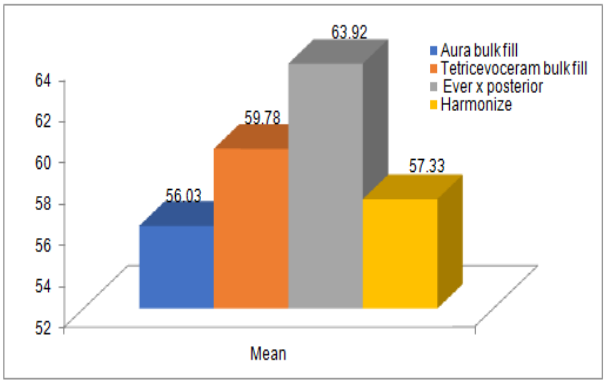

Figure 1. Shows Comparison of mean percentage degree of conversion (DC%) at height of 2mm of all the groups

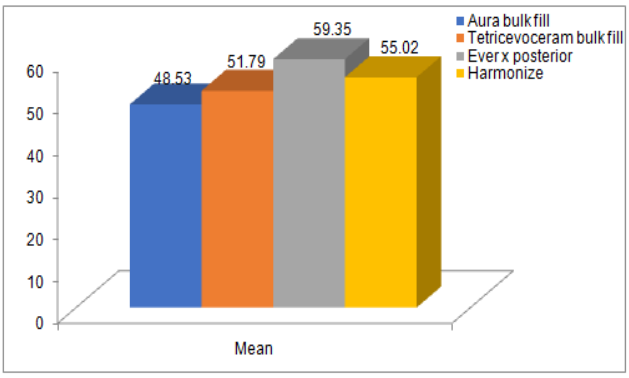

Figure 2. Shows Comparison of mean percentage degree of conversion (DC%) at height of 4mm of all the groups

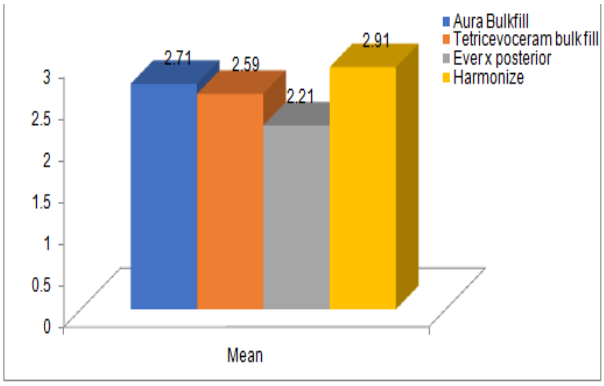

Figure 3. Shows Comparison of Volumetric shrinkage % of all the groups

components. Previous studies reported a typical volumetric shrinkage of 2–5% for conventional and 1.5–3% for bulk-fill resin composites, which are consistent with the values obtained in our study. The present study illustrates a simplified procedure to evaluate volumetric shrinkage by using a modified version of ASTM method D792 "Specific Gravity and Density of Plastics by Displacement" as suggested by Puckett and Smith in 1992. It provides a precise measure of volumetric shrinkage without the need of advanced equipment. The equipment required is an analytical balance capable of measuring to the nearest 0.1 mg (Tomar H and Choudhary E, 2018). In the present study, TEC showed significantlly better DC% when compared with Aura Bulkfill at both heights.

This may be attributed to the presence of both, a camphorquinone-amine initiator and Ivocerin, an intensifier capable of polymerizing the material at greater depths by absorbing light in a higher wave length range, in TEC. The 'Ivocerin' based initiator system has an absorption spectrum very close to Camphor quinone (CQ). It has been reported that photopolymerized activation is higher than CQ due to the absorption of visible radiation (Kubo CS et al 2018). Lesser DC of Aura Bulk Fill in comparison with EP and TEC at both the heights may indicate lower polymerization depth and might be related to the composition of the resin matrix. It has been observed from a study conducted by Chaves et al. (2019) that Aura Bulkfill has Bis-EMA ((2,2-bis[4-(2-methacryloxy) ethoxy) phenyl] propane) present in its resin matrix which tends to restrict the mobility of UDMA (diurethanedimethacrylate) monomers and decrease their reactivity and conversion value. Current study revealed that EP showed lesser VS% than Aura bulkfill which is in concurrence with results obtained by Rao, Hegde and Shetty (2017) who stated that the fibre length plays an important role in a restorative composite resin. According to Cheng TH et al the critical fibre length with Bis-GMA polymer matrix varies between 0.5 and 1.6mm.

In order for the fibre to act as effective reinforcement for polymers, stress transfer from the polymer matrix to the fibres is essential. This is achieved by having a fibre length equal to or greater than the critical fibre length. EP has a fibre length of 1mm to 2mm thus exceeding the critical fibre length. This contributes to substantial improvements in its physical properties. During placement into the cavity, the fibres orientate into a horizontal plane within the cavity. Due to strong adhesion between resin and silanated fibres in EP the direction of the fibres minimizes volumetric shrinkage. Results of this study showed that EP has better DC% when compared to TEC at both heights which are in agreement with Rezaei S et al (2019) who found that the use of short fibers as fillers in EP does not seem to negatively affect the curing depth. Its resin matrix composition includes Bis-GMA, TEGDMA, and PMMA, which comprise a semi-interpenetrating polymer network that provides optimal bonding properties. Also, E-glass fibers enhance the toughness of the polymer matrix.

In fact, the random orientation of fibers and semi-interpenetrating polymer network structure of their polymer matrix probably play an important role in the improvement of the mechanical properties of EP. The differences in the organic matrix structure of the composite resins have an effect on their degree of conversion. While the TetricEvoCeram composite did not contain TEGDMA, the presence of this monomer in the Ever x Posterior may have caused higher DC. According to a study conducted by Demir K and Bayraktar Y (2020), TEGDMA increases DC and crosslinking when mixed with Bis-GMA**.** Our study has demonstrated that short glass fiber reinforced composite resin EP showed lower volumetric shrinkage which can be attributed to the strong adhesion between resin and silanated e-glass fibers. Additionally, the direction of the fibres minimizes shrinkage in the horizontal plane after placement. This could derive better performance and durability in posterior restorations Rao, Hegde and Shetty (2017). However, this result is not in agreement with a study conducted by Abbasi M et al. (2018) which states that triethylene glycol dimethacrylate (TEGDMA), which is present in the composition of EP, has a high reactivity and results in a higher conversion of double bonds and

consequently a higher shrinkage. Present study revealed EP having better DC% at both heights when compared to Harmonize. This can be due to the higher filler content in Harmonize (81wt%) when compared to EP (74.2 wt%). According to a study conducted by Salem HN, Hefnawy SM and Nagi SM (2019), both the intensity of the light source, attenuating power of the material plus the filler loading influence the DC. Higher filler loading of Harmonize might strongly influence the intensity of the incident light, limiting the depth of cure. This is also in agreement with Majidinia S et al (2020), who stated that resin composites with more filler content would show less DC. In this current study, the DC decreased upon increasing the filler content. EP, with lowest filler content of 74% wt among groups, presented with highest degree of conversion values and Harmonize with highest filler content (81wt%) showed lesser values of DC % at 2 mm height. Among all the tested composites, highest volumetric shrinkage was observed with Harmonize. It can be due to the presence of TEGDMA in its matrix. Higher TEGDMA/BisGMA ratios in experimental composites resulted in higher contraction stress values due to increased volumetric shrinkage, as a result of enhanced conversion. Diluent monomers have lower molecular weight than the host monomers and they increase the density of polymerizable carbon double bonds, which may lead to more shrinkage. Langalia A et al (2015). However, EP also has TEGDMA in its matrix but still it showed least VS which can be due to the semi-IPN resin matrix that results in a plasticized matrix and short e-glass fibers in different directions, which does not allow the material to shrink easily in one dimension; these characteristics resulted in less shrinkage Jafrania S (2021).

Our study showed that Harmonize has better DC% at 4 mm height when compared to Aura bulkfill and TEC which is contrary to the majority of observations which claim that DC% of most of the bulkfills is better than the conventional composites. It can be related to a study conducted by Rezaei S et al (2019) who stated that the manufacturers of bulk-fill composites claim that they are superior to conventional composites in many aspects; however, some controversies still exist in this respect. For instance, the DC of >55% is clinically acceptable for bulk-fill composites but this value is still lower than the DC of conventional composites. In the current study, Ever X Posterior (EP) exhibited highest degree of conversion at both 2mm and 4 mm heights which is in agreement with results of a study conducted by Goncalvez F et al (2018) which states that Ever x posterior material has E-glass reinforcing fibers which did not reduce the passage of light through the material. Consequently, high conversion values at greater depths have been reported for this material. EP showed significantly lower volumetric shrinkage than the other tested resin composites. This observation was in line with Tsujimato A et al (2016) which stated that using short E-glass fibers with a semi-IPN-resin matrix may be one of the reasons why the volumetric shrinkage of SFRC is reduced.

CONCLUSION

1. Better DC% of EP is due to the presence of short fibers as fillers along with TEGDMA in its resin matrix. Lower volumetric shrinkage of EP is due to semi-IPN resin matrix that results in a plasticized matrix and short e- glass fibers in different directions, which does not allow the material to shrink easily in one dimension.
2. Significantly higher DC levels were measured in the samples of AB, TEC, EP at 2 mm height as compared to the 4 mm height which is due to the reason that the energy of light radiated by the light-curing unit is gradually attenuated as it passes through the composite mass. As a result, a gradual reduction in DC% of composite resin monomer occurs as the distance from the irradiated surface increases.

Harmonize showed almost similar values at both the heights. This may be due to the reason that it was incrementally placed and cured in thickness of 2 mm (according to manufacturer's instructions) when compared to the 4 mm single increment of BFCs.

3. BFCs and SFRC in the present study performed better in terms of DC % at 2 mm height and VS when compared to the newly introduced nanohybrid composite. However, at 4mm height, DC% of Harmonize was observed to be better than AB and TEC, which can be due to its incremental placement and curing of 2mm each.
4. In the present study, no direct correlation was found between degree of conversion (in %) and volumetric shrinkage (in %) of the RBCs.

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LIST OF ABBREVIATIONS USED

%	Percentage
AB	Aura Bulkfill
ANOVA	Analysis of Variance
BFC	Bulk Fill Composites
DC	Degree of conversion
EP	Ever X Posterior
FTIR	Fourier Transform Infrared Spectroscope
HS	Highly Significant
LED	Light Emitting Diode
min.	Minutes
mm	Millimetres
NS	Non significant
RBCs	Resin Based Composites
S	Significant
SD	Standard Deviation
SFRC	Short Fiber-reinforced Resin Composite
TEC	TetricEvoceram
VS	Volumetric Shrinkage

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