DOI: https://doi.org/10.54393/pbmj.v5i1.246

PAKISTAN BIOMEDICAL JOURNAL
https://www.pakistanbmj.com/journal/index.php/pbmj/index
Volume 5, Issue 1 (January 2022)

Original Article

Conversion, Polymerization Shrinkage, Heat Generation, and Depth of Cure of Novel Dental Composites

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ARTICLE INFO

Keywords:
Dental composites, polymerization shrinkage, degree of conversion, depth of cure

How to Cite:
Liaqat, S., & Jabeen, H. (2022). Conversion, Polymerization Shrinkage, Heat Generation, and Depth of Cure of Novel Dental Composites: Depth of Cure of Novel Dental Composites. Pakistan BioMedical Journal, 5(1), 79–82. https://doi.org/10.54393/pbmj.v5i1.246

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ABSTRACT

Objective: This study aims to determine the conversion, polymerization shrinkage, heat generation, and depth of cure of novel dental composites, and compare with current commercial composites. **Methods:** A total of 24 different formulations were tested for their conversion, shrinkage, heat generation, and depth of cure. Conversion was determined by using FTIR. Polymerization shrinkage and heat generation were determined theoretically using conversion, monomer volume fraction, average molecular weight of monomers present in each formulation, and number of methacrylate groups present in each monomer. Depth of cure was determined using the ISO 4049 scraping test. **Results:** The values obtained in this study for shrinkage, and heat generation were comparable to commercial bulk filled composites Z250, and Gradia. The factors that reduced the shrinkage and heat generation to a greater extent were sample thickness, and CaP addition. The higher shrinkage and heat generation in thin samples can be explained by large number of photons on the top surface. The heat generation is affected by the same variables as that of conversion and shrinkage. The heat generation values for samples using conversions at a thickness of 4 mm were ~ 0.06 kJ/cc, while using 1 mm thickness. values gave ~ 0.08 kJ/cc. The reduction in depth of cure with the addition of CaP was ~ 5 % **Conclusion:** The relatively higher, or comparable degree of conversion for experimental composites than commercial materials, could indicate their greater suitability for clinical applications. Polymerisation shrinkage, and heat generation are directly related to conversion, and conversion is affected by depth (thickness) of composites More detailed analysis is needed to confirm the ultimate depth of cure of these materials.

INTRODUCTION

Dental composites are tooth colored restorative materials. Dental composite usually contains a resin matrix, and inorganic fillers. The fillers can be glass or other reinforcing fillers. The matrix is mainly formed from high molecular weight monomers such as urethane dimethacrylate (UDMA), and bisphenol A-glycidyl methacrylate (Bis-GMA) [1]. Fillers are added to increase strength, reduce polymerization shrinkage and heat generation [2]. A silane coupling agent is used to augment the bond between these two components and to aid filler distribution. An initiator and activator are usually added to begin and later control the polymerization process when external energy (light) is applied. The main problems associated with current composites are the low degree of conversion, depth of cure, and high polymerisation shrinkage. All these properties significantly affect the longevity of a restoration in the oral environment.

One area of composite improvement discussed in this study includes monomer conversion. This is considered very important, as the amount of un-cured monomers can have a potential negative effect on mechanical properties and adverse effects on surrounding tissues [3]. The other area of composite improvement discussed in this study includes polymerisation shrinkage, and heat generation. Shrinkage can be reduced by using high molecular weight monomers and increasing the filler content. High polymerisation shrinkage is a major cause of micro-gaps formation, which can lead to recurrent caries [4].

This study will also discuss the depth of cure of composite formulations. Usually, poor depth of cure means un-cured monomer present in the lower surface of the restoration that may lead to cytotoxicity and reduced mechanical properties. Poor depth of cure and shrinkage effects can be compensated to some extent by placing the composites in small increments [5]. The depth of cure can also be

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improved by better matching of monomer and filler refractive indices.

METHODS:

This section outlines all the materials used throughout the study.

Abbreviation	Name	Supplier	Product code	MW
UDMA	Urethane dimethacrylate	DMG Dental	100112	470
TEGDMA	Tri ethylene glycol dimethacrylate	DMG Dental	100102	228
HEMA	Hydroxyethyl methacrylate	DMG Dental	100220	130
4-META	4-Methacryloxyethyl trimellitate anhydride	Polysciences	17285	286

Table 1: Details of Monomers that were used throughout this study. Molecular weight information from manufacturers

Abbreviation	Name	Manufacturer	Product Code	Size (ìm)	Silanated
Glass (Contains Particles and Fibres)	Barium -boro-alumino -silicate glass particles (GP)	DMG Dental	680326	~ 7	Yes
	Silane coated borosilicate glass fibre (GF)	MO -SCI	0322201 -S	~ 15 * 300	Yes
MCPM	Mono Calcium Phosphate Monohydrate	Himed	MCP-B26	~ 53	No
TCP	â – Tri Calcium Phosphate	Plasma biotal	SSB210907	~ 53	No
CHX	Chlorhexidine diacetate salt hydrate	Sigma - Aldrich	1001447866	~ 44	No
PLS	å - Polylysine	Handary SA	28211-04-3	~ 74	No

Table 2: Details of filler materials used throughout this study. Information from manufacturer

Abbreviation	Name	Manufacturer	Product Code	MW	Function
CQ	Camphorquinone	Alfa Aesar	10120023	166.22	Initiator
DMPT	N,N-Dimethyl-p-Toluidine	Sigma Aldrich	15205BH	135.21	Activator

Table 3: Details of initiator, activator and inhibitor chemicals used throughout this study

A total of 24 different formulations were tested for their conversion, shrinkage, heat generation, and depth of cure. They were divided into two groups. The basic formulation consists of UDMA: TEGDMA 3:1 containing CQ and DMPT (both 1wt %) added to a powder containing 5 wt % fibres and glass particles.

The variables investigated in studies of composite monomer conversion, shrinkage, and heat generation included: sample thickness (4 or 1 mm), adhesive monomer 4-META or HEMA, polylysine level (5 or 0.5 wt %), chlorhexidine level (5 or 0 wt %), and CaP level (20, 10, or 0 wt %). All the samples were cured for 40 s. With depth of cure studies, the same variables were tested, except the first variable was curing time (40 or 20 s) instead of sample thickness. The sample size was 3 for all techniques. To analyse all data three variable (thickness or cure time, 4-META vs HEMA or PLS level), two level factorial analysis was initially undertaken for each wt % of CaP and CHX. With this factorial analysis it was possible to determine the level of effect of the first three variables, and any interaction effects.

RESULTS:

Monomer Conversion and Polymerisation Shrinkage

Figure 1 shows representative FTIR spectra for formulations with reactive fillers before and after light cure of 40 s. All the changes observed upon light exposure were characteristic of methacrylate monomer polymerisation. The 1320 cm^{-1} peak heights before and after cure was measured to obtain conversion. This corresponds to C-O bond stretching in the polymerising methacrylate group. The spectra also shows monomer/polymer peaks at 1710 cm^{-1} (C=O stretch), 1640 cm^{-1} (C=C stretch), 1528 cm^{-1} (N-H deformation), 1455 cm^{-1} (C-H bend), and 1160 cm^{-1} (C-O-C stretch). The spectra also showed the presence of TCP, and MCPM at 1005/940 cm^{-1} and 1040 cm^{-1} respectively due to phosphate (P-O) stretching.

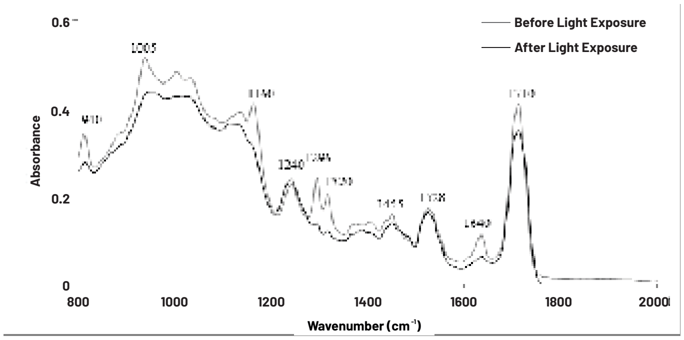

Figure 1: Representative FTIR spectra of an experimental composite before and after 40 s light curing. The specific formulation has PLR 4:1, glass powder 55 wt %, glass fibre 5 wt %, MCPM 5 wt %, TCP 5 wt %, CHX 5 wt %, and PLS 5 wt %

The conversions at 5 minutes from the start of cure of all formulations are given in figure 2. This was between 50-80 % with highest conversion noted with 1 mm thickness, and least conversion noted with 4 mm samples.

Assuming small changes in the filler density with varying composition has negligible effect on the volume fraction of monomer, the polymerisation shrinkage and heat generation will be directly proportional to conversion. The estimated polymerisation shrinkage calculated theoretically for all formulations using polymerisation levels at the 2 different depths are given in figure 2. The shrinkage values estimated using polymerisation levels at 4 mm were ~ 2.5 %, while those using 1 mm depth conversions were ~ 3.5 %.

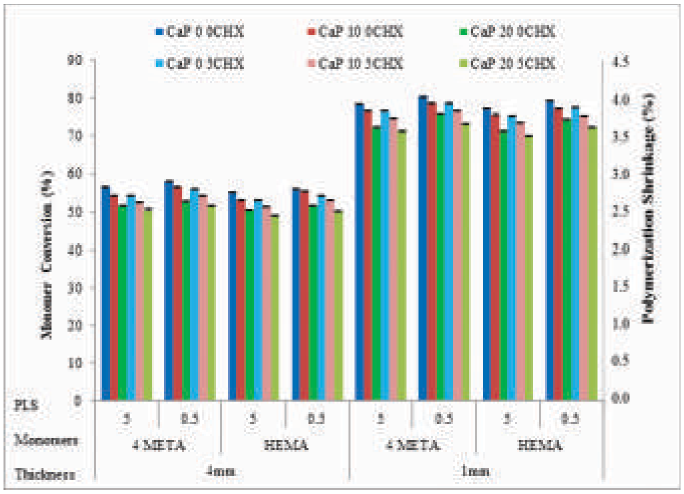

Figure 2: Monomer conversion and Polymerisation shrinkage with PLS (5 or 0.5 wt %), adhesive monomers (4-META or HEMA), and sample thickness (4 mm or 1 mm) for all formulations with varying levels of calcium phosphate (20, 10, or 0 wt %), and chlorhexidine (5 or 0 wt %). Error bars represent 95 % CI, (n=6)

Heat generation

The heat generation was calculated theoretically from conversion and polymerisation shrinkage. The heat generation is 0.0175 times the calculated shrinkage values. The heat generation is affected by the same variables as that of conversion and shrinkage. The heat generation values for samples using conversions at a thickness of 4 mm were ~ 0.06 kJ/cc, while using 1 mm thickness values gave ~ 0.08 kJ/cc.

Depth of cure

Depth of cure of experimental composite formulations obtained using ISO 4049 are given in figure 3. The values obtained for depth of cure of experimental formulations passed the minimum ISO requirement of 1.5 mm minimum thickness. The values obtained were between 1.55-2.5 mm with greater depths being achieved with 40 s cure and lower CaP level.

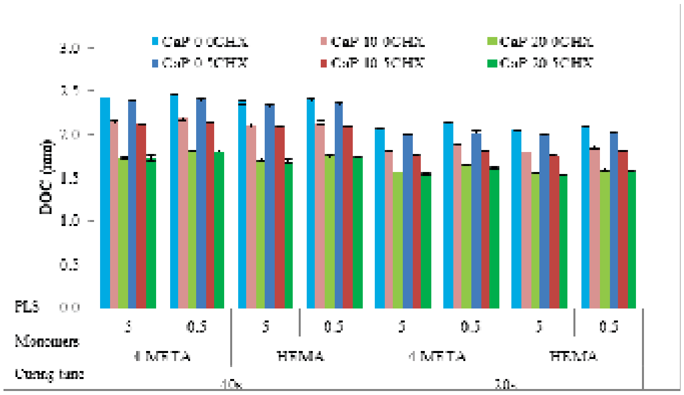

Figure 3: Depth of cure with PLS (5 or 0.5 wt %), adhesive monomer (4-META or HEMA), and sample curing (40 s or 20 s) for all formulations with varying levels of calcium phosphate (20, 10, or 0 wt %), and chlorhexidine (5 or 0 wt %). Error bars represent 95 % CI, (n=6)

Addition of 4-META instead of HEMA and reducing level of PLS also slightly increased depth of cure. Interaction effects between these three variables (curing time, adhesive monomer or PLS level) were negligible in comparison with the level of the effect of all the variables.

DISCUSSION:

Degree of Conversion

The degree of conversion is an important property that affects the performance of dental composites. The mechanical, and biological properties of resin-based composites generally improve with increase in monomer conversion. The improved mechanical properties will reduce the chances of material failure under masticatory loads [6]. Additionally, high conversion can substantially reduce the cytotoxic effects associated with the release of un-reacted monomers [7]. Generally, the monomer conversion of methacrylate based dental materials are lower (36-69 %) [8,9]. The high degree of conversion in the above experimental composites in this study could be attributed to the incorporation of diluent co-monomers (TEGDMA, HEMA, and 4-META) in addition to the bulk monomer UDMA [10]. In this study higher conversion was seen with samples thickness of 1 mm, as compared to 4 mm. The low conversion associated with thicker samples, suggests the possibility of un-cured monomers at critical areas in a restoration such as composite-dentine interface. With thicker samples the amount of un-cured material at the bottom is much higher.

Polymerisation Shrinkage and Heat Generation

The factors that reduced the shrinkage and heat generation to a greater extent were sample thickness, and CaP addition. In case of thicker samples less conversion is seen. For thick layer placement of composite, this might be a mechanism of reduced shrinkage and heat generation in lower sections due to less monomer conversion while maintaining a higher conversion at the top surface [11]. Previous studies have also found reduced polymerisation shrinkage with increase in thickness of the samples [12,13]. This suggests that there may be more contraction in the top composite surface than the lower surface. The higher shrinkage and heat generation in thin samples can be explained by large number of photons on the top surface [14]. All these factors explain the decrease in shrinkage and heat generation in thick samples (4 mm).

Depth of Cure

The main factors that affected the depth of cure of experimental materials were the use of reactive fillers CaP and curing time (20 s vs 40 s). The reduction in depth of cure with the addition of CaP was ~ 5 %. This reduction with high levels of CaP can be attributed to the mismatch in refractive indices of filler, and liquid phase, which results in decreased depth of cure [15]. It was shown in the literature that the filler addition, especially the CaP, increases the translucency of the material [16], making it difficult for the light to penetrate into thick samples [17]. This low translucency with CaP can also explain the decrease in depth of cure. The obtained values in this study were on average less than the commercial bulk composites which can also be related to the addition of various fillers to the experimental composites. The depth of cure is influenced by the duration of light cure [18-21]. More detailed analysis is needed to confirm the ultimate depth of cure of these materials.

ACKNOWLEDGEMENT:

The author would like to acknowledge Higher Education Commission Pakistan for funding.

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