POLYMERIZATION SHRINKAGE, DEGREE OF CONVERSION, DEPTH OF CURE, AND HEAT GENERATION OF NOVEL EXPERIMENTAL AND COMMERCIAL COMPOSITE

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ABSTRACT

Objective: *To compare and characterise commercial and control composites. By characterising these composites, target properties for new experimental dental composites can be outlined.*

Materials and Methods: *Three commercial, and two experimental composites were compared and investigated. The composition of commercial materials were provided from published literature and manufacturer's information. The changes in intensity of spectral peaks that were involved in free radical addition polymerization were used to determine the degree of conversion using FTIR (n=6). The samples were prepared in 1 mm, and 4 mm thickness and cured for 40 s from top. Depth of cure, Polymerization shrinkage and heat generation were determined using conversion data, and equations.*

Results: *The conversion for Gradia, and Z250 were significantly lower than the flowable Vertise flow, and experimental composites C-HEMA, and C-4META in both 1 mm, and 4 mm depth. The shrinkage, and heat generation values suggests opposite trends to conversion. With the highest shrinkage, and heat generation associated with Vertise flow. Depths of cure of all materials were in the range of 2.28 to 2.49 mm at 20 s, and 40 s cure.*

Conclusion: *The experimental composite with added monomers had the potential to compete with commercially available composites, without compromising the properties.*

Keywords: *Commercial and control composites, polymerization shrinkage, depth of cure.*

INTRODUCTION

Dental caries, commonly known as tooth decay, is one of the most common diseases affecting people of all ages. Dental caries causes destruction of dental hard tissues due to the production of acidic by-products by bacterial fermentation of carbohydrates.[1] Dental caries, if left un-treated, can cause damage that can be biological, and physical. In order to stop caries advancement and restore structure and function of the tooth, the damaged dental structures are repaired using different restorative materials.

Dental composites are tooth coloured 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-glicidyl methacrylate (Bis-GMA).[2] Fillers are added to increase strength, reduce polymerisation shrinkage and heat generation.[3] 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 polymerisation process when external energy (light) is applied.

The experimental control composite studied in this project consists of two bulk filled composites, one with hydrophilic monomer HEMA (5 wt %), and

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other with adhesive, and de-mineralizing monomer 4-META (5 wt %). The filler phase consists of 100 % glass particles. The purpose of these control composites was to make comparisons between commercial composites and provide benchmark properties for the subsequent development of new composites.

MATERIALS AND METHODS

Three commercial, and two experimental composites were compared and investigated.

The details are given in table 1.

The control composites were prepared with the following compositions (see table 2).

The composition of commercial materials were provided from published literature and manufacturer's information.

The changes in intensity of spectral peaks that were involved in free radical addition polymerisation were used to determine the degree of conversion using FTIR (n=6). The samples were prepared in 1 mm, and 4 mm thickness and cured for 40 s from top. Polymerisation shrinkage and heat generation were determined using conversion data. Depth of cure was performed according to ISO 4049 scraping technique (n=6) after 20 s and 40 s cure.

RESULTS

Monomer Conversion:

Monomer conversion at the bottom of the composite discs 1 mm, and 4 mm in depth after curing for 40 s are given in figure 1. The commercial bulk filling composites showed lower conversion than the experimental bulk composites and commercial flowable composite. The conversions at 4 mm depth were on average 10 % less than that at 1 mm. Experimental composite C-HEMA, and C-4-META showed higher conversion of ~ 80 % and ~ 70 % at 1 mm and 4 mm depth, followed by Vertise flow ~ 65 % and ~ 55 % at 1 mm and 4 mm depth. The lowest conversions were noted with Gradia and Z250 (~ 45 % and ~ 40 % at 1 mm and 4 mm depth).

Polymerization Shrinkage and Heat Generation:

Polymerization shrinkage and heat generation were measured theoretically using conversion data. The exact amounts of the monomers in commercial materials were not disclosed by the manufacturers, the shrinkage results were calculated assuming equal quantities of each monomer. Monomer conversion is directly proportional to polymerization shrinkage and heat generation. So, for example if monomer conversion is low the shrinkage and heat generation associated with it will be less, and vice versa.

The shrinkage at the bottom of 1 mm and 4 mm deep specimens of commercial bulk filling composites were ~ 2.5 and 2 % respectively. The experimental composite showed a shrinkage of ~ 4.2 % and 3.5 % at the bottom of 1 mm and 4 mm thick samples. The highest shrinkage was associated with flowable composite Vertise flow with a shrinkage of ~ 5 and 4.2 % at the bottom of 1 mm and 4 mm deep samples (see figure 2).

The same trends can be seen in case of heat generation. Bulk filled commercial composites showed less heat generation at the bottom of 1 mm, and 4 mm thick samples than experimental bulk filled composites, and the commercial flowable composite (figure 3).

Depth of Cure:

Depth of cure after 20 or 40 s light exposure were measured using ISO 4049 method outlined in section 2.2.6.2 (see figure 4). All composites showed a depth of cure between 2.28-2.36 mm after 20 s cure. The cure depth was increased further after 40 s cure to 2.45-2.49 mm. The maximum depth of cure was seen in case of Z250, while the rest of the composites

Table 1: Commercial and control materials to be investigated with manufacturers and type explanation.

Filtek™ Z250	3M ESPE, USA	Bulk Filling Dental Composite
GC Gradia Direct posterior	GC Corporation, Europe	Bulk Filling Dental Composite
Vertise™ Flow	Kerr Corporation, Italy	Self-Adhering Flowable Composite
C-HEMA	Experimental Composite	Bulk Filling Dental Composite
C-4META	Experimental Composite	Self-Adhering Bulk Composite

Table 2: Control materials monomer and filler phase composition.

C-HEMA	Powder to liquid ratio 4:1	Monomer Phase: UDMA 69.75 wt %, TEGDMA 23.25 wt %, HEMA 5 wt %, CQ 1 wt %, DMPT 1 wt %. Filler Phase: Silane treated glass particles 100 wt %.
C-4META	Powder to liquid ratio 4:1	Monomer Phase: UDMA 69.75 wt %, TEGDMA 23.25 wt %, 4-META 5 wt %, CQ 1 wt %, DMPT 1 wt %. Filler Phase: Silane treated glass particles 100 wt %.

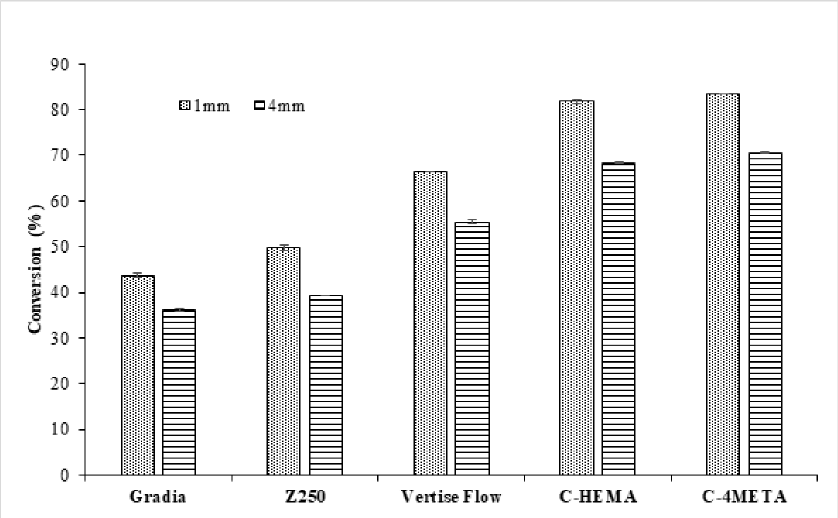

Figure 1: Conversion in percentage of commercial and experimental composites at depth of 1 mm and 4 mm (Error bars are 95 % confidence interval, n=6).

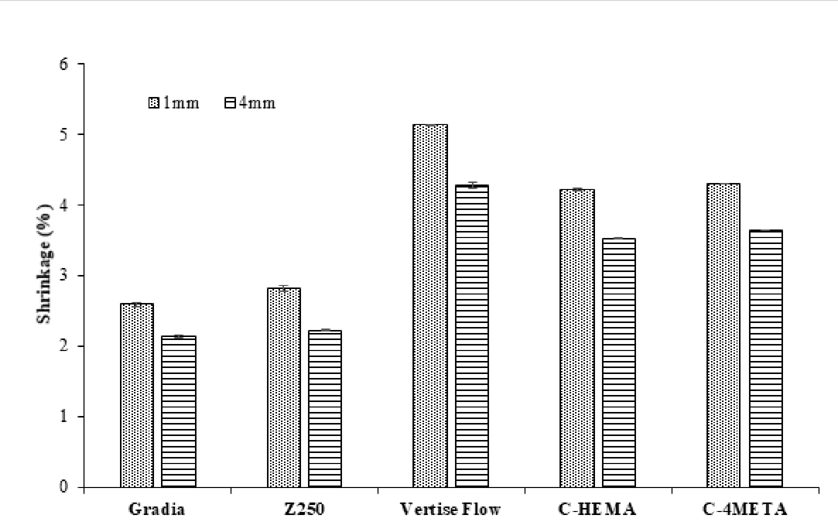

Figure 2: Volumetric shrinkage in percentage of commercial and experimental composites at depth of 1 mm and 4 mm (Error bars are 95 % confidence interval, n=6).

showed no significant difference in depth of cure.

DISCUSSION

The above study has compared three commercials (2 bulk filled, and 1 flowable composite), and two experimental bulk filled composites, with C-4META composite having self-adhesive properties.

Experimental composites (C-HEMA, C-4META) and flowable composite (Vertise flow) final conversion were higher than that of the bulk filled

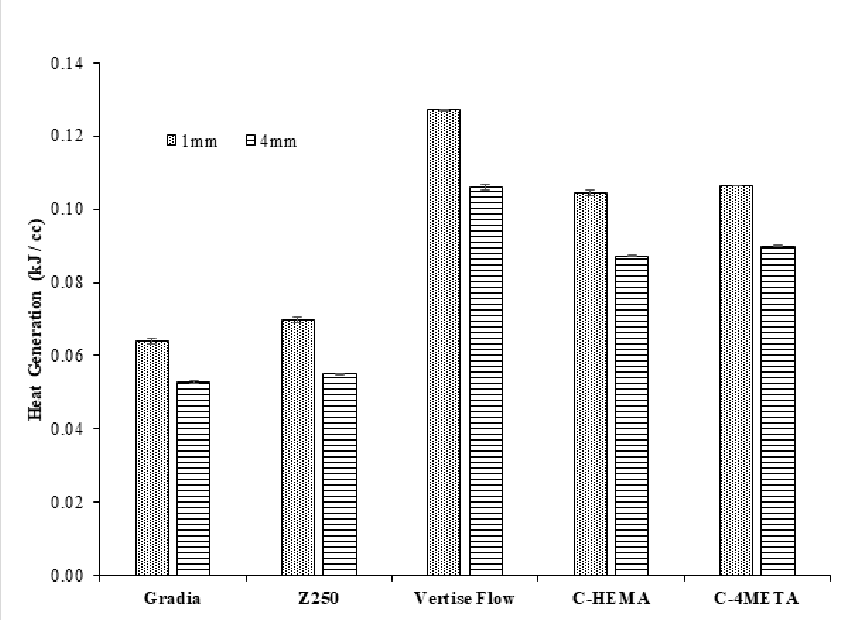

Figure 3: Heat generation of commercial and experimental composites at depth of 1 mm and 4 mm (Error bars are 95 % confidence interval, n=6).

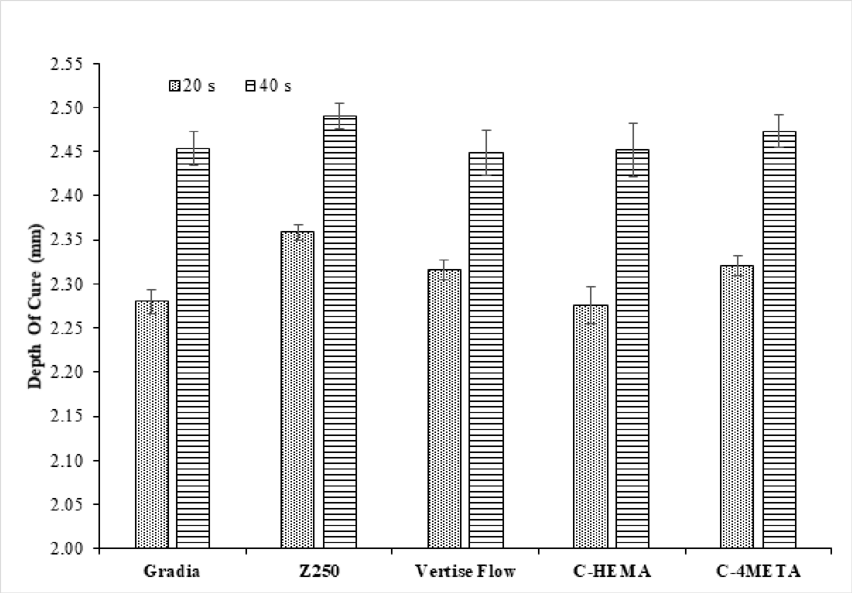

Figure 4: Depth of cure using ISO scraping technique of composites after curing of 20 s and 40 s (Error bars are 95 % confidence interval, n=6).

commercial composites. Incomplete conversion is usually associated with un-reacted monomers in the polymer matrix, which has the potential to dissolve in the wet oral environment.[4,5] This can reduce the longevity of the composite filling. The above conversion data for commercial composites Z250, Gradia, and Vertise flow were comparable to that noted in literature using FTIR.[6-9]

In the literature most of the resin composites have shown water sorption in aqueous solution to some extent. In conventional composites the water sorption was mainly associated with the resin phase. On absorption of water, various chemical, biological, and mechanical changes could be seen. The release of un-reacted monomers from the composites upon water sorption is considered a serious problem. These un-reacted monomers can leach into the oral environment, and can have cytotoxic effects.[10] Additionally, these un-reacted monomers cause plasticisation of the polymer matrix, and encourage water sorption catalysed hydrolytic degradation, that results in reduced mechanical properties.[11,12]

CONCLUSIONS

The experimental composite with added monomers had the potential to compete with commercially available composites, without compromising the properties.

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