Effect of trifluoroethyl methacrylate comonomer on physical properties of Bis-GMA based dental composites

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The purpose of this study was to investigate the effect of substituting triethyleneglycol dimethacrylate (TEGDMA) with 2,2,2-trifluoroethyl methacrylate (TFEMA) on water sorption, water solubility, degree of conversion, polymerization shrinkage, fluoride release and flexural strength of bisphenol-A-glycerolate dimethacrylate (Bis-GMA)/TEGDMA/urethane dimethacrylate (UDMA) dental composites. Flexural strength, water sorption and water solubility were measured according to ISO 4049 standard. Degree of conversion was determined by FTIR technique. Fluoride release rate in deionized water at 37°C was determined by using photometric method. One-way ANOVA and Tukey’s HSD tests were used for statistical significance analysis (p<0.05). The results demonstrated that TFEMA decreased water solubility and volumetric shrinkage significantly but neither reduced water sorption, enhanced flexural strength nor improved degree of conversion. Partial substitution of TEGDMA with TFEMA in Bis-GMA/TEGDMA/UDMA dental composite had positive effect on its water solubility and volumetric shrinkage quality.

Keywords: Dental composites, Water solubility, Volumetric shrinkage

INTRODUCTION

Secondary caries refers to the recurrence of tooth decay after the initial restoration and is cited as the most frequent and compelling reason for the replacement of existing restorations¹⁻³. Specifically, marginal leakage is thought to be responsible for development of secondary caries⁴⁻⁶. Despite gross micro leakage at the silicate restoration-tooth interface, a lower incidence of secondary caries in the restored teeth, as well as in teeth adjacent to those restorations, has been reported⁷. Such anticariogenic properties of silicate cements were believed to be due to the ability of the material to release fluoride throughout the life of the restoration⁸. Fluoride ions may be incorporated into the tooth structure, making it more resistant to acidic dissolution. The ionic radius of fluoride is similar to that of the hydrogen ion. This has important consequences in that the fluoride ions can replace the hydroxyl ions in the hydroxyapatite lattice and form the more acid-resistant fluoroapatite (Ca₁₀(PO₄)₆F), a fluorinated calcium phosphate⁹⁻¹⁰. Widely used fluoride-releasing restorative materials include glass ionomers, composites, and resin composites¹¹⁻¹⁴. These materials have received much attention due to their significant release of fluoride, the uptake of fluoride in cavity walls and plaque and, thus, the enhanced reprecipitation of calcium and phosphate promoted by the fluoride release¹⁵⁻¹⁶. However, the inferior mechanical properties of glass-ionomers and resin-modified glass ionomer materials have limited their use¹⁰,¹⁷. On the contrary, resin composites have better mechanical properties than glass ionomers due to the higher fillers content. However, high filler content also increases the viscosity of the uncured composite material¹⁸. In this regard, triethyleneglycol dimethacrylate (TEGDMA) is often used to reduce the viscosity of the uncured composite paste¹⁹. However, TEGDMA has been associated with problem of increased volumetric shrinkage upon curing. Moreover, the hydrophilic nature of TEGDMA monomer can increase water sorption and, thus, may have biological side effects when leaching out as unpolymerized monomer¹⁰. Ultimately, these problems reduce a restoration’s lifetime and represent the driving force for research effort toward improving dental composites formulation. Many alternative reactive diluents to TEGDMA have been explored to improve resin shrinkage and water sorption problem. One approach is to use monomethacrylate and acrylate that have been shown to have reduced volumetric shrinkage and increased degree of conversion²⁰.

The purpose of this study was to investigate the effect of replacing some TEGDMA with 2,2,2-trifluoroethyl methacrylate diluent monomer (TFEMA), on the water sorption, water solubility, degree of conversion, volumetric shrinkage, flexural strength and fluoride-release profile of bisphenol-A-glycerolate dimethacrylate (Bis-GMA)/TEGDMA/urethane dimethacrylate (UDMA) experimental dental composites.

MATERIALS AND METHODS

Materials
n-Propylamine (98%), fumed silica (0.007 µm), camphorquinone (99%), 3-methacyryloxypropyl-trimethoxysilane (MPS) (98%), Bis-GMA; TFEMA; TEGDMA; UDMA, ethyl-4-dimethylbenzoate (4EDMAB)
and tetrabutylammonium fluoride (TBAF) were purchased from Sigma-Aldrich, St. Louis, MO, USA. Cyclohexane (Emsure; 98%) was obtained from Merck, Darmstadt, Germany. All chemicals are used as received.

Methodology

1. Silanization of silica nanoparticle filler
Silanization was effected using a reported method\textsuperscript{21}. Briefly, silica (12.5 g), MPS (2 mL), cyclohexane (200 mL) and n-propylamine (0.2 g) were stirred at room temperature for 30 min and then heated at 70–80°C for 30 min at atmospheric pressure. The solvent and volatile by-products were distilled off in a rotary evaporator (Buchi R-215, Flawil, Switzerland) at 60°C. The resulting white powder was further heated at 95°C for 1 h in a rotary evaporator and further dried under a vacuum suction overnight.

2. Preparation of dental composite
The experimental as well as the control dental resin matrix contained 0.17 wt% of a photoinitiator system camphorquinone (CQ), 0.69 wt% of accelerator 4EDMAB, 10 wt% TBAF, and 55 w/w% silanized fumed silica (0.007 µm) as filler (Table 1).

All materials were weighed and mixed with pestle and mortar until a transparent paste was obtained. The formed composite paste was kept in a glass vial with its lid sealed using paraffin film, and the vial was wrapped with aluminum foil to prevent exposure to light until further investigation.

3. Water sorption and water solubility
Water sorption and water solubility were measured according to ISO 4049 method\textsuperscript{22}. Disk specimens of 15 mm in diameter and 1 mm thick (n=5) were made in a set of split stainless steel molds. Resins were sandwiched in the molds between two glass slides and photoactivated by a light cure unit (FlashSoft2, Roslev, Denmark) on each side for 20 s each. The light cure unit was calibrated for light output (450 mW/cm\textsuperscript{2}) by means of a radiometer (Demetron, Orange, CA, USA). After removal from the molds, the specimens were kept in glass vials wrapped with aluminum foil and the lids were sealed with paraffin film.

The disks were conditioned by placing them in a desiccator containing calcium sulfate at 37°C until a constant weight had been achieved (m\textsubscript{0}). The disk specimens were then placed in a beaker containing 100 mL of deionized water. The beaker was wrapped in aluminum foil to exclude light and placed in an incubator at 37°C. The specimens were removed at different time points (1, 6, 24 h, and subsequently at two-day intervals), blotted with filter paper, dried and weighed, then returned to water. This was continued until a constant weight (no further weight gain) had been achieved (m\textsubscript{m}). The disks were removed from the water and placed in a desiccator, containing calcium sulfate, at 37°C until a constant weight has been achieved. To speed up the drying process, the desiccator was attached to a vacuum suction to further dry the disks overnight and then reweighed for the last time (m\textsubscript{d}). These steps were carried out to evaluate the water sorption (A) and solubility (S) according to Oysaed and Ruyter formulas\textsuperscript{23}:

Water sorption (µg/mm\textsuperscript{3}), \(A=(m\textsubscript{1}-m\textsubscript{2})/V\)

Water solubility (µg/mm\textsuperscript{3}), \(S=(m\textsubscript{0}-m\textsubscript{2})/V\)

Where m\textsubscript{0} is the sample weight before immersion, m\textsubscript{1} is the sample weight after immersion in water and m\textsubscript{2} is the sample weight after immersion and desiccation.

For the weight measurement, an analytic balance (Mettler Toledo weighing balance) with an accuracy of up to 0.0001 g was used. The means and standard deviations of water sorption and water solubility (n=5) were calculated for each group.

4. Flexural strength
The flexural strength was determined in accordance with ISO 4049:2009\textsuperscript{22} using a universal testing machine (INSTRON, model 1026). Samples (25×2×2 mm (l×h×w), n=12 for each group) were prepared in the same way as mentioned above. The samples were immersed in de-ionized water, stored at 37°C for 24 h, and then tested on the Instron testing machine with a crosshead speed of 0.75 mm/min. The flexural strength, \(\sigma_f\), was calculated from \(\sigma_f=3Fl/2bh^3\), where, F is the maximum load, l is the distance between supports, b is the width of the specimen, and h is the height of the specimen.

5. Degree of conversion
Three different treatment groups for each composite formulation namely: uncured composite paste, freshly cured, one week and one month post-cured composites were analyzed using FTIR spectroscopy technique. The FTIR analysis was conducted at the Chemistry...
The composite powder or paste was placed between two Mylar strips and pressed into a thin film. The FTIR spectrum was recorded at zero time and immediately after exposure to light cure unit for 80 s. The IR spectra were recorded over 600–4,000 cm\(^{-1}\) region and acquired with 32 scans at a resolution of 4 cm\(^{-1}\). For each spectrum, the height of aliphatic C=C absorption peak at 1,638 cm\(^{-1}\) and aromatic C=C absorption peak at 1,608 cm\(^{-1}\) was determined. In order to fit the Beer-Lambert law, base line technique was used. The percentage of carbon-carbon double bond reacted, which shows the percentage of monomer conversion, was determined according to the following equation:

\[
\text{Degree of conversion (\%)} = 100 \left[ 1 - \frac{A_{1638}/A_{1608}}{A_{1638}/A_{1608}}_{\text{uncured}} \right]
\]

where \(A_{1638}/A_{1608}\) is the ratio of absorbance at 1,638 cm\(^{-1}\) over that at 1,608 cm\(^{-1}\) (Fig. 1).

6. Polymerization shrinkage
Volumetric shrinkage was calculated from the difference in density of the uncured and the cured polymers using Archimedes principle. All measurements were made with a density weighing balance (Sartorius AX224) in a temperature-controlled room (T=23°C). Five uncured samples (0.100±0.02 g) were pressed hard with microscope glass slabs into individual squarish matrix and weighed in air and in water. The samples were then compacted into a stainless steel mold [4 mm (w)×4 mm (l)×2 mm (t)] and light cured for 20 s on the top and bottom. The cured samples were weight in air and in water. The sample density and volumetric shrinkage were calculated using the following formulas:

\[
\text{The density in g/cm}^3, \rho = \frac{m_{\text{air}}}{m_{\text{air}} - m_{\text{water}}} (\rho_{\text{water}} - \rho_{\text{air}}) + \rho_{\text{air}}
\]

\[
\text{The percent volumetric shrinkage} = \left( \frac{1}{\rho_{\text{cured}}} - \frac{1}{\rho_{\text{uncured}}} \right) \times 100\%
\]

where \(\rho_{\text{cured}}=\)density of the cured composite and \(\rho_{\text{uncured}}=\)density of the uncured composite.

7. Fluoride release study
A stainless steel mold for a disk specimen with a dimension of 2±0.1 mm in diameter by 5±0.1 mm thickness was used to prepare five test specimens for each of the five experimental composites. The specimens were light-cured, under a glass slide, by irradiating on both the top and bottom sides for 20 s with the light cure unit. After removal from the mold, each specimen was submerged inside a plastic bottle containing 9 mL of deionized water and kept in an incubator at 37°C. After 24 h, each specimen was removed and rinsed with 1 mL deionized water. The combined deionized water was used for fluoride content measurement. The fluoride content determination was continued for 1, 2, 3 and 4 weeks and the next one-month intervals after incubation.
8. Statistical analysis
The data were expressed as mean±standard deviation of at least three replications. Standard errors of the means were presented on all the charts and graphs. Data were analyzed using one-way ANOVA and statistical difference between the means was determined using Tukey’s HSD test, with $p \leq 0.05$ as the level of significance.

RESULTS
All the dental composites could be cured to a transparent solid except Formulation 4. Figure 2 shows their flexural strengths, which ranged from 17.6 to 19.6 MPa. However, the flexural strengths of all the composite formulations were not significantly different from the control, although Formulation 1 had significantly higher flexural strength than that of Formulation 2 ($p < 0.05$).

The water sorption and water solubility of the control and Formulations 1–3 are presented in Figs. 3 and 4, respectively. One-way ANOVA showed that water sorption among the control and Formulations 1–3 was not significantly different ($p > 0.05$), although Formulation 2 showed the lowest water sorption value, after Formulations 3 and 1, with the control group being the highest. On the other hand, the mean water solubility of Formulations 1–3 was significantly lower than that of the control ($p < 0.05$). However, there was no difference between the water solubility of Formulations 2 and 3 ($p > 0.05$), which had almost the same level of solubility. It is noted that the water solubility decreased linearly with increasing content of TFEMA, up to Formulation 2 (in the sequence of C, 1, 2) in which TEGDMA was partially substituted with TFEMA in the formulations. The solubility remained almost unchanged when three quarters of TEGDMA was replaced with TFEMA (from Formulation 2 to Formulation 3).

Figure 5 shows the degree of conversion of the control as well as the Formulations 1–3, measured at one week and one month respectively after curing. Generally, the degree of conversion one month post curing decreased significantly ($p < 0.05$) in Formulations 1, 2 and 3 as compared to that of the control. Nonetheless, the degree of conversion of Formulation 2 and the control was significantly higher ($p < 0.05$), after storing for longer period (1 month vs. 1 week), which indicates that the curing continues on storage. However, the degree of conversions of TFEMA loaded composites were lower by about 5–10% and 13–23% when compared with that of the control, after storing for one week and one month, respectively.

The polymerization shrinkage, also known as volumetric shrinkage, was determined from the differences in densities of uncured and cured dental composites using Archimedes principle. The result of
Table 2  Polymerization shrinkage of dental composites

| Dental composite | Polymerization shrinkage, % (SD) |
|------------------|----------------------------------|
| Control          | −3.87 (0.34)                     |
| Formulation 1    | −2.35 (0.05)*                    |
| Formulation 2    | −2.26 (0.16)*                    |
| Formulation 3    | −2.12 (0.08)*                    |

*significantly different (p<0.05) from the control
#significantly different (p<0.05) from Formulation 2

polymerization shrinkages measurement is shown in Table 2 below. Replacing TEGDMA with TFEMA in the formulation reduced polymerization shrinkage by around 40%.

Figure 6 shows the mean concentrations of fluoride released from all the dental composites at test intervals up to 12 weeks. The mean fluoride release of the control reached a peak of 12.53 mg/L at 24 h, followed by a sharp decline to 1.39 mg/L on the third week. The Formulation 1 reached a peak of 8.23 mg/L at 24 h and 7.21 mg/L on the second week, followed by a sharp decline with fluoride release at 3.04 mg/L on the third week and gradually declining in the following weeks. On the other hand, the Formulation 2 one reached a peak of 4.66 mg/L at 24 h, followed by a slow fluoride release at 0.85 mg/L on the fourth week, and gradual fluoride release at 2.34 mg/L on the 12th week, whereas the Formulation 3 did not show burst release but reached a peak of 3.80 mg/L towards the end of 12th week.

DISCUSSION

TEGDMA is a highly flexible, low-molecular-weight and low-viscosity monomer that is commonly used in commercial dental composites. These properties of TEGDMA contribute to a high chain mobility during initial stage of polymerization, which favor a monomer to polymer conversion.

Although TEGDMA has been found to be the best diluent for Bis-GMA based dental composite that improved its practicality and application in clinical setting, the problems of high water sorption, increased water solubility, and volumetric shrinkage associated with the use of TEGDMA remained. In particular, high water sorption and high water solubility were responsible for the leakage of unpolymerized TEGDMA from the Bis-GMA dental composite, which is harmful to human body in that it could cause gene mutation, and for a drop in mechanical stability that could result in rapid catastrophic degradation of resin-dentin bonds.

Thus, this study focused on substituting TEGDMA with a monofunctional comonomer TFEMA in composite formulation (Table 1) and its effect on various physical properties of the composites was evaluated. In the preparation of dental composites, Bis-GMA and UDMA composition were fixed while TFEMA and TEGDMA composition were varied with the control formulation containing only fixed components and TEGDMA, a typical clinical formulation, while Formulations 1 to 3 have increasing content of TFEMA (concurrently decreasing TEGDMA content) and the fixed components, and Formulation 4 only contains the fixed component and TFEMA.

The Formulation 4, which does not contain TEGDMA, did not cure under the same radiation condition even with extended exposure to curing light. This indicates that TEGDMA, a highly flexible bifunctional cross-linker monomer with two terminal methacrylate groups, had a pivotal role in the curing of composite material. On the other hand, TFEMA as a monofunctional monomer with only one terminal methacrylate group did not contribute to cross-linking of the fixed component in Formulation 4. Although theoretically the fixed component Bis-GMA/UDMA, both of which are biacrylated, could crosslink, their extremely high viscosity invariably prevented this from happening. This observation suggests that a low viscosity, bis-methacrylated diluent monomer TEGDMA is definitely required for the fixed component to cure. Therefore, TFEMA cannot replace TEGDMA, in totality, in the formulation of Bis-GMA/TFEMA/TEGDMA/UDMA based dental composites. Thus, further investigation on Formulation 4 was discontinued.

As reported, water sorption is a diffusion-controlled process that occurs in the organic resin matrices. Specifically, the amount of water that composite resins can absorb depends on the hydrophilicity of the polymeric matrices and proportion, and the filler type. The water sorption values obtained in this study ranged from 83.3 to 92.87 µm/mm³ and are higher than a maximum value of 40 µm/mm³ set by the ISO 4049 standard. The relatively high water sorption values are most likely due to somehow lower filler content, 55% (w/w), used in this study, as it was reported that high filler content is correlated with low water sorption. Thus, there is still a potential for further improvement.
by loading higher amount of filler in these formulations. However, several other factors, such as the polymeric matrix composition, filler particle type, and the degree of curing reached after the polymerization reaction can also influence the solubility and sorption behavior of the dental composites. Nonetheless, some reduction in water sorption attributed to TFEMA was noted, although not significantly different from that of the control. This is possible because the TFEMA has been reported to demonstrate water and oil repelling, low surface tension, and non-adhesion propeties. The observed trend in the water solubility of the experimental composites, which is significantly lower (p<0.05) than that of the control, explained the benefits of TFEMA have to offer.

The degree of conversion (Fig. 5), from FTIR analyzes, decreased with TFEMA content in the experimental composites as compared to the control one. The tendency to undergo cyclization of TEGDMA, which does not contribute to network formation, also led to higher conversion, but this cyclization did not happen in the case of TFEMA. Nonetheless, the degree of conversion of the control as well as the experimental composites continued to increase on its own after light-source assisted curing. This is beneficial as further curing consolidates the stability and strength of the composite structure.

The polymerization shrinkage of dental composite resin can affect the integrity of composite-dentin bond and, in the worst case, an onset of microcrack that could lead to secondary caries. It was reported that increasing TEGDMA concentration resulted in higher shrinkages due to higher degree of conversion. In this study, the volumetric shrinkage was significantly reduced by nearly 40% (Formulations 1–3 as compared to the control) and this is most probably attributed to a decrease in the TEGDMA content in the composite which, in turn, due to lack of ether linkage flexibility of TEGDMA and, thus, less free volume reduction upon curing. This volumetric shrinkage reduction may be counteracted by the more rigid TFEMA, as further reducing TEGDMA content (Formulations 2 and 3) did not effect more significant shrinkages.

Despite of gross micro leakage happening at the silicate restoration-tooth interface, a lower incidence of secondary caries in the restored teeth, as well as in teeth adjacent to these restorations has been reported. The preventive effects of fluoride released from silicate and conventional glass polyalkenoate cements have encouraged dental materials manufacturers to incorporate fluoride in a number of other restorative materials.

The effect of TFEMA on fluoride release of the composites was evaluated. All the formulations and the control were incorporated with same amount of tetrabutylammonium fluoride, the source of water soluble fluoride. It is believed that the fluoride ions were released from the composites by a dissolution and diffusion mechanism:

1) Dissolution of the material, releasing all its component parts including fluoride ions.
2) Diffusion, which can be either the release of fluoride in conjunction with an appropriate counter ion, typically sodium, or fluoride release via exchange with hydroxyl groups of the surrounding aqueous environment.

The control formulation has the highest fluoride release during the first two weeks period, followed by Formulation 1 and Formulation 2, with Formulation 3 having the slowest of fluoride release, which showed that fluoride release decreased with increasing content of TFEMA. It seems that these results were somehow correlated with the water solubility degree of the composites, whereby composites with higher water solubility gave higher fluoride release. The fluoride ions, being water soluble, would be carried through by diffusion by virtue of its solubility. This may suggest that the fluoride ions are accumulating in the vicinity of the more water soluble domain in the formulation and rapid release of the ions from the control was due to higher solubility associated with TEGDMA bulk. In the case of Formulation 3, the result showed that fluoride ions were released steadily, and the fluoride concentration doubled by the end of 12th week. This shows that the Formulation 3 was capable of releasing fluoride in a more sustained manner.

CONCLUSION

TFEMA had significant effect in attenuating water solubility and volumetric shrinkage of the cured Bis-GMA/TFEMA/TEGDMA/UDMA based dental composites but effected lower degree of conversion and slower and steady release of fluoride ions. Replacing TEGDMA with TFEMA in the dental composite formulation material may reduce silane hydrolysis and microcrack formation by virtue of lower water solubility, lower polymerization shrinkage, and the release of fluoride ions in a sustained manner.

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