Journal of Macromolecular Science, Part A
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597274

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To cite this Article Tiba, A. and Culbertson, Bill. M.(1999) 'DEVELOPMENT OF VISIBLE LIGHT-CURED MULTI-METHACRYLATES FOR DENTAL RESTORATIVE MATERIALS', Journal of Macromolecular Science, Part A, 36: 4, 489 — 506

To link to this Article: DOI: 10.1081/MA-100101544

URL: http://dx.doi.org/10.1081/MA-100101544

J.M.S.—PURE APPL. CHEM., A36(4), pp. 489–506 (1999)

DEVELOPMENT OF VISIBLE LIGHT-CURED MULTI-METHACRYLATES FOR DENTAL RESTORATIVE MATERIALS

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Key Words: Multi-methacrylates Synthesis, Light Curing, Neat Resins Formulation, Water Sorption

ABSTRACT

This study focused on new visible light-curing (VLC) oligomers exhibiting low shrinkage, low sorption of water, and improved mechanical properties. A family of multi-methacrylates, based on poly(isopropylidenediphenol) resin (BPA), was synthesized, characterized, and evaluated. The commercial BPA resin is prepared from enzymatic polymerization (oligomerization) of bisphenol A. The BPA resin, having an average of eight phenolic hydroxyl groups per molecule, was treated with propylene carbonate and the resultant product, i.e., propoxylated BPA (PBPA) oligomer, was confirmed by Fourier transform infrared spectroscopy (FT-IR) and ^{13}C nuclear magnetic resonance (NMR). The propaxylated BPA (PBPA) was subsequently treated with methacryloyl chloride to produce the multi-methacrylates (EPBPA), identified by FT-IR and NMR. The EPBPA oligomer multi-methacrylate: triethylene glycol dimethacrylate (TEGDMA) (50:50/wt:wt) blends were combined with 0.5 wt% camphorquinone (CQ) and 1.0 wt% N,N-dimethyl-aminoethyl

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methacrylate (DMAEMA). The control was 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl] propane (BisGMA): TEGDMA (50:50/wt:wt) blends having the same levels of CQ/DMAEMA. Differential photocalorimetry (DPC) and differential scanning calorimetry (DSC) showed these multi-methacrylate/TEGDMA (neat resin) blends have polymerization characteristics comparable to the BisGMA/TEGDMA control. These multifunctional oligomers (EPBPA) have lower polymerization shrinkage and lower uptake of water and some common organic solvents. These results suggest that the new type of polyfunctional methacrylate oligomers (EPBPA) have potential application in formulating dental composites as direct esthetic restorative materials with improved properties.

INTRODUCTION

Dental composites consist of a monomer resin, a ceramic filler, a coupling agent which binds the filler phase to the polymer phase, and an initiator or initiating system. While each of these components contributes desirable properties to the curing process or final material behavior, this work attempts to address some of the specific problems associated with the resin itself. Hence, all of the studies will be performed with neat monomer resins and examination of the influence of the resin composition on the polymerization behavior, maximum attainable conversion, and mechanical properties of the cured polymer network.

In terms of the polymer matrix used in dental composites, an ideal system would be extremely strong and wear resistant, unswellable in saliva, adherent to the tooth structure and filler particles, and biocompatible. Current failures in composite restoration can be linked to each of these areas, particularly to problems associated with durability and wear. While the mechanism for the degradation is not completely understood, several factors have been identified as contributing to wear, and one of these factors is induced stresses within the restoration. These stresses arise from differences in thermal expansion between the restoration and tooth structure [1, 2], swelling from moisture uptake [3-5], and volume shrinkage during the polymerization [6-8]. In particular, the swelling behavior and the presence of unreacted monomer, after completion of the cure, contribute to stress and degradation.

In addition to possible detrimental effects on the composite properties (swelling and plasticization), residual, unreacted monomer also presents a concern with respect to the biocompatibility of the restoration. While the polymeric material itself is thought to be noninteracting and biocompatible, the unreacted monomers may possess some degree of toxicity to cell tissues. Perhaps the most adverse effects are related to the potential for residual monomer to be leached from the composite and into the body where it may cause sensitization and allergic reactions in certain patients [9].

During the polymerization of multifunctional monomers for dental restorations, typical final double bond conversions range anywhere from 55-75% [10-12]. These low final conversions are a result of the high crosslink density in the system which limits the mobility of reacting species. Thus, despite the presence of initiating species and unreacted double bonds in the system, the diffusion control of the propagation reaction limits any additional conversion. In addition to the previously discussed biocompatibility-related issues, all unreacted functional groups in the system will act as plasticizers, diminishing the strength of the polymer and increasing the swelling. Finally, the adhesion of resin to the filler particles will be reduced since only 55-75% of the double bonds have reacted that could contribute to binding the filler phase to the polymer phase. Therefore, increasing the double bond conversion of monomers is highly advantageous since it would decrease the extent of unreacted monomer in the system, increase the strength of the polymer (through decreased plasticization and increased crosslink density at higher conversions), and reduce swelling.

In order to make significant improvements in dental composites we need entirely new monomers and polymers to exploit, intermediates which are specifically designed to provide needed improvement. This leads to the proposal for the synthesis, characterization, and demonstration of formulation potential of new multi-methacrylate monomers designed to give improved performance composite materials. The research thrust is in three directions, as follows:

(1) Generation of new intermediates, multi-methacrylate monomers and/or oligomers, to produce improved performance composites.

(2) Evaluate the potential of these new intermediates for formulating improved dental composites.

(3) Evaluate the VLC polymerization characteristics of the new resins using differential photocalorimetry (DPC), differential scanning calorimetry (DSC), and FTIR. The polymerization shrinkage, water sorption will also be examined and compared with the BisGMA-based control.

EXPERIMENTAL

Materials and Methods

The starting material, poly(isopropylidenediphenol) resin (trade name Enzo*BPA 1300), was provided by Enzymol International, Inc., Columbus, OH. It is reported that the Enzo BPA is made from enzymatic oligomerization of bisphenol A. Enzymol claims Enzo*BPA 1300 has a number average molecular weight (Mn) of 1300. Propylene carbonate, from Aldrich Chemical Co., Milwaukee, WI, was used as received. Methacryloyl chloride (Aldrich Chemical) was distilled (bp = 95-96˚C) before use.

Synthesis of Oligomers EPBPA #1-4

The synthesis of EPBPA oligomers involve two steps: propoxylation of BPA and esterification of propoxylated BPA. The BPA resin was propoxylated by using propylene carbonate with $N(n\text{-Butyl})_3$ as a catalyst in a bulk reaction under nitrogen, at temperatures from 160 to 180°C. After reaction for 4-5 hours, the crude product was purified by precipitation using methanol-water solvent, giving excellent yields of slightly yellow-colored product. The hydroxyl groups on the PBPA oligomer were partially esterified by a condensation reaction with methacryloyl chloride (Figure 1). The extent of esterification was adjusted by the addition of controlled amounts of methacryloyl chloride to tetrahydrofuran solution of PBPA and triethylamine under nitrogen, while keeping the temperature at 0-10°C. Four EPBPA oligomers (EPBPA #1-#4), with an increasing level of esterification, were obtained and purified by using column chromatography (silica gel: 200 mesh; eluent: hexane/ethyl acetate). The PBPA and EPBPA oligomers were identified by FT-IR and ^{13}C NMR spectroscopy. The degree of esterification was determined by measuring the percentage of unreacted hydroxyl groups on the EPBPA oligomers, according to the quantitative analyses of organic compounds [13].

IR and NMR Characteristics

The IR spectra were obtained by using a MIDAC FT-IR Spectrophotometer (MIDAC Corp., Costa Mesa, CA). The ^{13}C NMR spectra were collected on a 300 MHz Bruker AM spectrometer, using deuterated chloroform ($CDCl_3$) as a solvent and tetramethylsilane (TMS) as a reference.

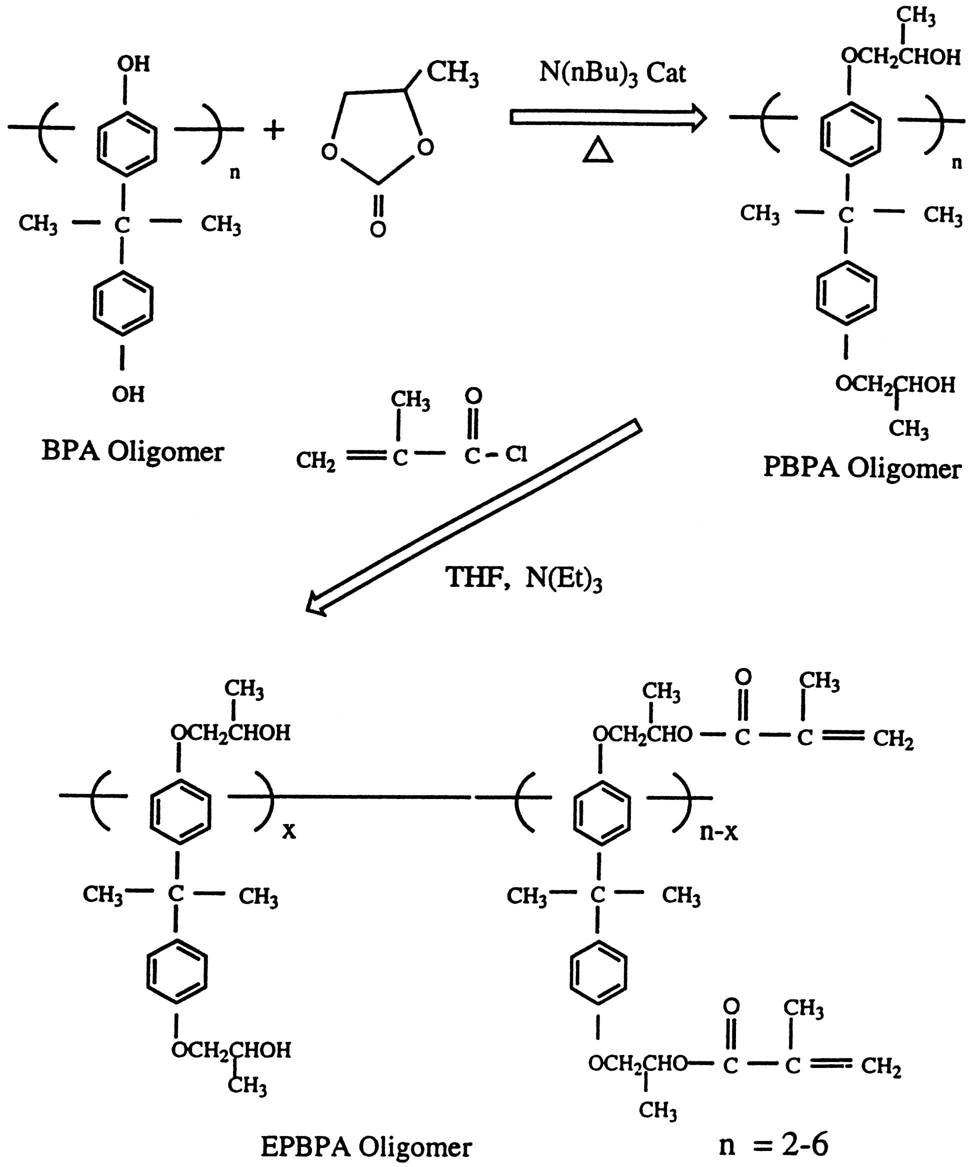


Figure 1. Synthesis route for propoxylated BPA polyols (PBPA) and EPBPA multi-methacrylate oligomers.

Photopolymerization

The visible-light curing (VLC) characteristics of the experimental oligomer blends were evaluated by differential photocalorimetry (DPC). The VLC oligomer systems were formulated as follows: Oligomer (50 g), triethylene glycol dimethacrylate (TEGDMA) (50 g), camphorquinone (CQ, 0.5 g), and

2-N,N-dimethylaminoethyl methacrylate (DMAEMA, 1.0 g). The control was BisGMA/TEGDMA (50/50, wt:wt) blends having the same levels of CQ/DMAEMA. The mixtures (about 5 mg) were weighed into a small aluminum pan and exposed to visible light (argon lamp) for 1 and 3 minutes under nitrogen gas at 25°C, using a TA Instruments 930 DPC unit (TA Instruments, Wilmington, DE). The heat of reaction (ΔH) was calculated by employing the TA data analysis DPC 4.1A program.

In order to examine the postcuring polymerization behavior and remaining double bonds of the VLC resins available for further thermal polymerization, the samples, which had been previously light-cured for three minutes, were further examined by DSC.

Determination of Polymerization Shrinkage

The polymerization shrinkage was obtained by measuring the density differences between uncured and cured resin test specimens. The volumetric shrinkage was calculated using Formulation 1:

$$\%\ Shrinkage = [1 - \frac{d_{uncured}}{d_{cured}}]\ x\ 100\% \tag{1}$$

Monomer density was measured by weighing the liquid injected from a calibrated syringe. The VLC-cured resins were made in a cylindrical shape and polished and their density obtained by the calculation of weight divided by volume.

Preparation of VLC Resin Specimens

The cylindrical resin specimens (3.3 mm in diameter x 6.6 mm in height) were made by putting the VLC oligomer blends into transparent glass molds, followed by photo-curing with an Elipar Light source (wavelength 468 nm, ESPE, Seefeld, Germany) for a total of 5 minutes. After removal from the glass mold, the sample surface was polished by using silicon carbide paper (FEPA P# 400). All specimens were conditioned in distilled water at 37°C for one week prior to property tests.

Water Sorption

For each resin, five VLC cylindrical specimens were conditioned to a constant weight in a desiccator and then immersed in distilled water at 37°C. At different time intervals, the specimens were removed from the water, lightly blotted with a tissue paper, and weighed. After immersion in water for six months, the specimens were removed and reconditioned to a constant weight in a desiccator. The water sorption for each specimen was determined from the difference in weight between the specimen immersed for the predetermined time intervals and the reconditioned specimen. The BisGMA based neat resin served as a control.

A repeated-measured ANOVA was used to determine whether any significant differences in the water sorption between the various resins and the times studied. The REGW multiple range test was used for pairwise comparisons.

RESULTS AND DISCUSSION

Structural Characterization of Oligomers

The synthesis scheme for the EPBPA multi-methacrylates (oligomer) is shown in Figure 1. The structures of BisGMA and hypothetical EPBPA, with 50% and 100% esterification, are illustrated in Figure 2. The ^{13}C-NMR chemical shifts of BPA, PBPA and EPBPA-#4 are shown in Table 1. The typical absorption bands in the IR spectra of the same materials, 3500-2800 and 1800-1500 cm^{-1} range, are plotted in Figure 3.

For BPA, there is a strong, wide absorption peak for phenolic-OH at 3387 cm^{-1}. After propoxylation, this peak shifts to 3413 cm^{-1}, representing an aliphatic -OH absorption, and strong bands also appear at 2877 and 2990 cm^{-1}, indicating addition of propylene type residues. With increasing levels of esterification, the -OH absorption peak gradually decreased and shifted to higher wavenumbers. As esterification progresses, the 1720 (c=o) and 1640 (c=o) cm^{-1} peaks, not present in PBPA, become more intense. The FT-IR and NMR data were consistent with the structures shown in Figure 1. The degree of esterification, and hydroxyl group content are shown in Table 2. The experimental multi-methacrylates EPBPA have significantly higher molecular weights and more rigid molecular chains than the BisGMA, due to a lack of flexible connecting units between the covalently coupled aromatic rings. The viscosities of the EPBPA oligomers decreased with an increase in the extent of esterification.

Photopolymerization

The enthalpy values (ΔH) of the DPC results for the multi-methacrylate resins, subjected to visible light-curing for a total of 1 and 3 minutes, are shown

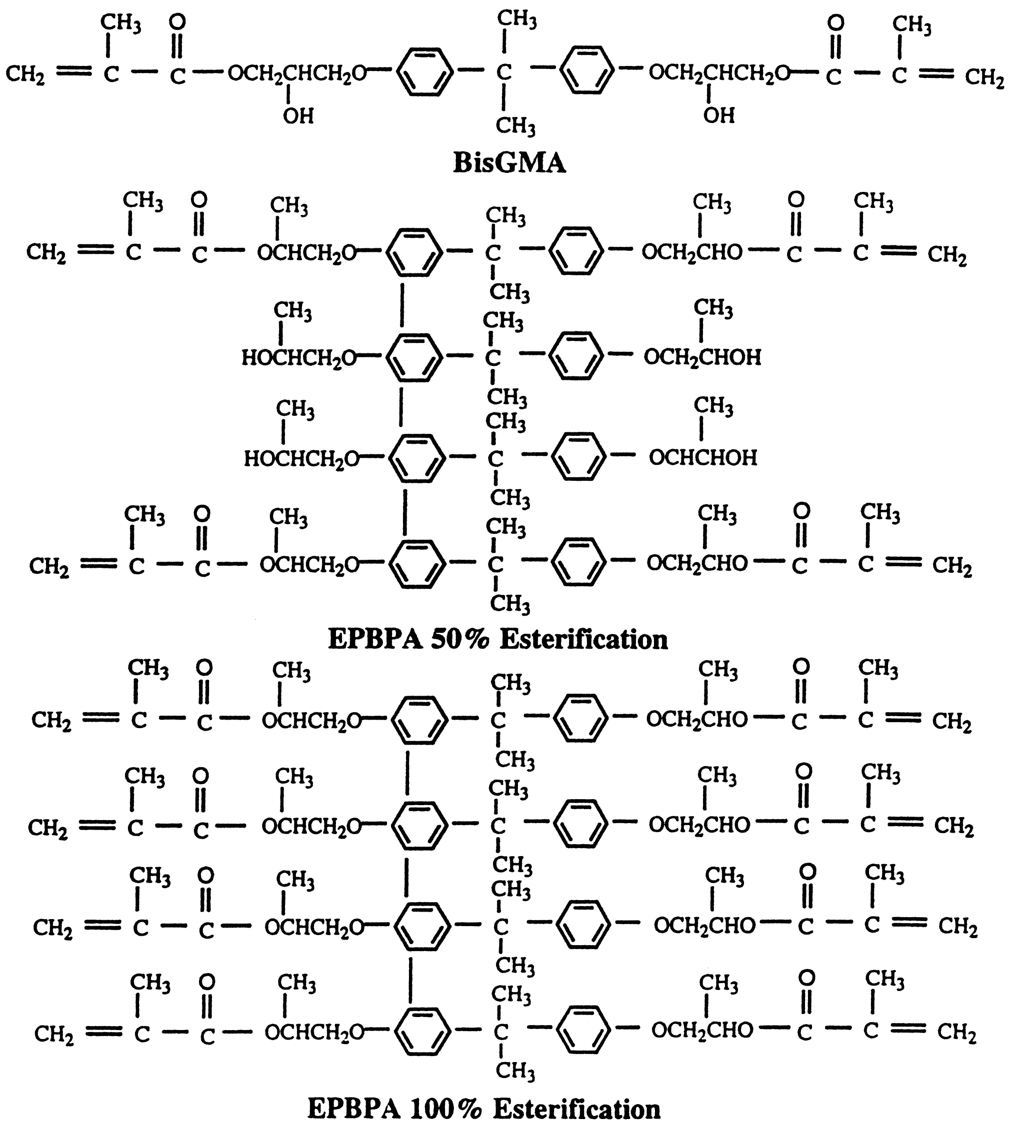
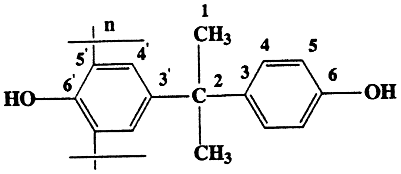
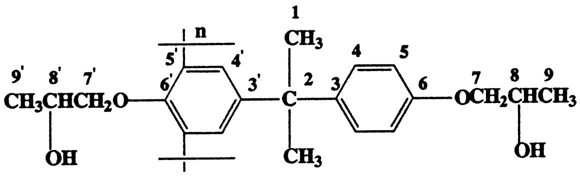
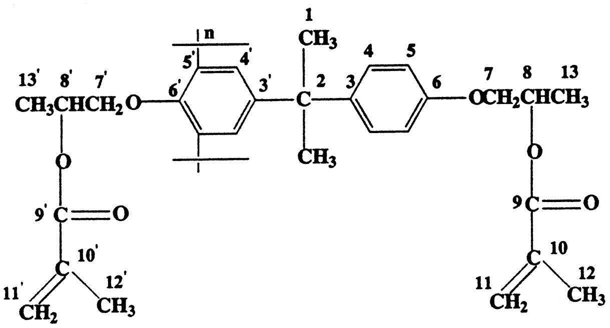


Figure 2. Structures of BisGMA and hypothetical EPBPA oligomers, having 50 and 100% esterification.

in Table 3. The ΔH for the BisGMA control was significantly higher than that of each experimental oligomer system. The ΔH values of the experimental oligomers, with different levels of esterification, are not significantly different after light-curing for 1 minute. However, after light-curing for 3 minutes, the experimental EPBPA oligomers with higher levels of methacryloyl esterification show somewhat higher ΔH values.

TABLE 1. ^{13}C NMR Chemical Shifts of BPA, PBPA Polyol, and EPBPA Multimethacrylate

Compound / Structure	^{13}C NMR Chemical Shift (ppm)	
BPA 	C_1 (32.1); C_2 (42.2); C_4 (128.2); C_4 (119.8); C_6 (156.2); C_6 (153.9)	C_3 (147.2); C_3 (143.0); C_5(115.7); C_5 (126.2)
PBPA 	C_1 (31.3); C_2 (42.1); C_4 (128.1); C_4 (125.9); C_6 (157.1); C_6 (140.9); C_8 (62.0); C_9 (36.2)	C_3 (143.7); C_3 (140.1); C_5(114.2); C_5 (127.8); C_7(70.1); C_7 (70.4) C_8(60.9); C_9 (38.1)
EPBPA 	C_1 (30.9); C_2 (41.6); C_4 (125.9); C_4 (115.2); C_6 (156.4); C_6 (156.1); C_8 (63.4); C_8 (63.0) C_{10} (143.1); C_{10} (136.1); C_{12} (24.1); C_{13} (41.7)	C_3 (142.9); C_3 (136.9); C_5(114.1); C_5 (127.5); C_7(65.6); C_7 (66.9); C_9 (173.1); C_9 (166.9); C_{11} (114.9); C_{11} (124.8); C_{12} (18.1); C_{13} (41.3)

The photo-polymerization properties can be further characterized by DSC and FT-IR to analyze the postcuring behavior and the amount of remaining double bond after photo-curing. The samples, which had been previously light-cured for three minutes, were further examined by DSC. The results are given in Table 4. From the ΔH values for the post-polymerization, all the experimental oligomers and BisGMA mixtures underwent some extent of further polymer-

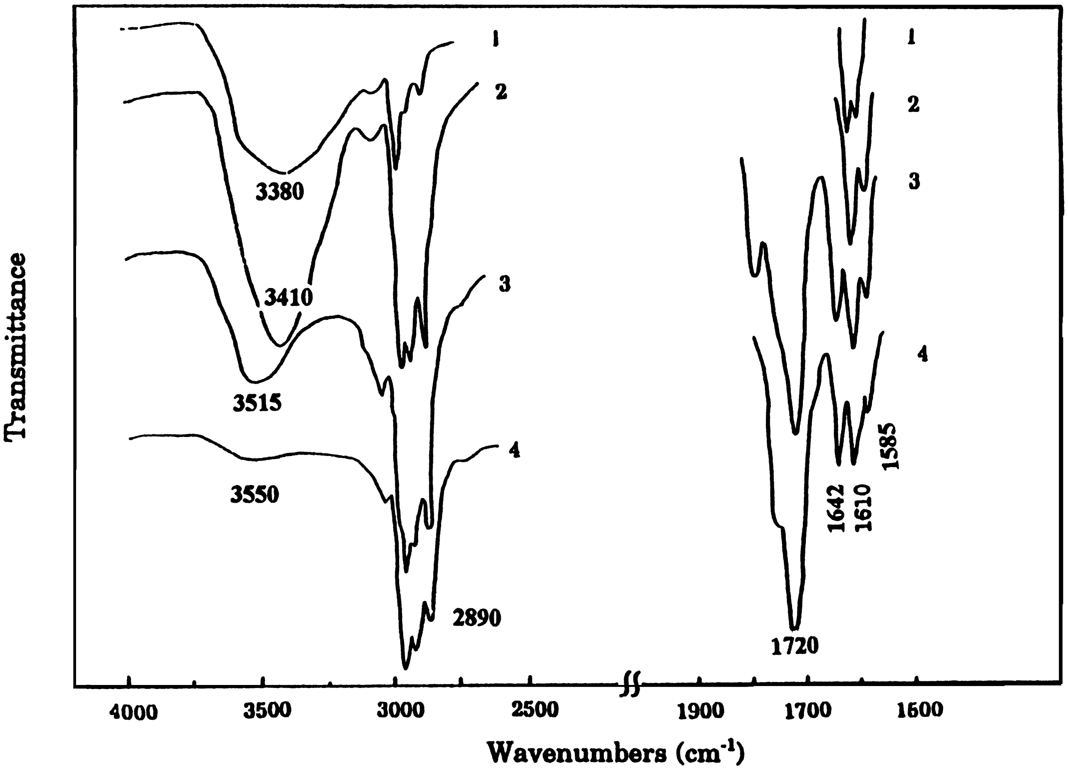


Figure 3. Partial IR spectra of 1: BPA; 2: PBPA; 3: EPBPA #2; 4: EPBPA #4.

ization after being cured by visible light for 3 minutes. The magnitude of postcure for BisGMA and oligomer/TEGDMA mixtures is in the range of 9.2-15.8%.

The percentage remaining double bonds (RDB %) available for further heat polymerization in the VLC BisGMA is significantly lower than that in each experimental oligomer, statistically. The possible clinical relevance is that after composites are put into the tooth cavities and cured by visible light, they may continue curing in the oral cavity @ 37°C. The latter curing may be referred to as “dark cure”.

The RDB % of dental composite resins can also be quantitatively estimated by using FT-IR [13]. In this method, the transmission IR spectra of initial monomer and cured resin were recorded and the absorbance area of the methacrylate C=C band at 1640 cm^{-1} were determined for both monomer (Am_{1640}, Am_{1610}) and resulting cured resin (Ar_{1640}, Ar_{1610}). The ratio between Ar_{1640}/Ar_{1610} and Am_{1640}/Am_{1610} was then taken as the fraction of unreacted dou-

TABLE 2. TEGDMA and BisGMA Monomers and EPBPA Oligomers Characteristics

Oligomers	Wt. % of -OH Group	Number of -OH	Number of C=C	P (%)*
TEGDMA			2.0	
BisGMA	6.67	2.0	2.0	
EPBPA #1	5.01	4.5	3.5	43
EPBPA #2	3.62	3.5	4.5	58
EPBPA #3	1.90	2.0	6.0	75
EPBPA #4	0.67	0.7	7.3	92

* P: Degree of Esterification

TABLE 3. Heat of Reaction (ΔH) of Multi-methacrylates Visible Light-Cured 1 and 3 Minutes

	Light-cured 1 min**		Light-cured 3 min**	
	ΔH (J/g) (SD)	Degree of Cure (%)	ΔH (J/g) (SD)	Degree of Cure (%)
BisGMA	147.2 (5.2) A	50.1 (1.7) A	250.4 (17.2) A	84.1 (5.1) A
EEBPA #1	47.8 (7.3) B	20.1 (3.7) B	166.7 (9.7) B	69.1 (5.9) B
EEBPA #2	56.35 (11.2) B	21.4 (3.4) B	155.8 (21.4) B	60.7 (8.1) B
EEBPA #3	59.3 (10.1) B	19.1 (3.1) B	191.4 (13.1) BC	68.2 (4.1) B
EEBPA #4	61.2 (9.6) B	18.5 (3.0) B	210.2 (18.1) C	67.2 (5.3) B

* Oligomer/TEGDMA 50/50 (wt/wt) with 0.5 wt. % initiator (CQ) and 1.0 wt. % co-initiator (DMAEMA).

** The ΔH values, as determined by DPC, using TA Instruments 930 DPC fitted with an argon lamp for VLC, employed a sample size range of 5-6 mg. Each entry is the mean value (standard deviation) for a group of three specimens (N = 3). Means with the same letter, i.e., A, B, and C, for ΔH and degree of cure are not significantly different at the $\alpha = 0.05$ level ($P > 0.05$).

TABLE 4. Polymerization Characteristics of BisGMA and EPBPA Oligomers/TEGDMA Blends

Oligomer/TEGDMA Mixtures*	Degree of postcure (%) (DSC) (SD)**	Percentage of RDB (%) (FT-IR) (SD)**	Shrinkage (%) (SD)**
BisGMA	9.2 (2.0) A	18.2 (2.1) A	11.2 (0.5) A
EPBPA #1	13.9 (1.2) B	25.2 (3.7) B	10.1 (0.5) AB
EPBPA #2	15.8 (2.0) B	23.1 (1.7) AB	9.6 (0.6) B
EPBPA #3	15.9 (0.7) B	21.2 (3.2) AB	9.0 (0.5) B
EPBPA #4	14.6 (1.3) B	20.8 (3.9) AB	9.1 (0.4) B

* Oligomer/TEGDMA 50/50 (wt/wt) with 0.5 wt. % initiator (CQ) and 1.0 wt. % co-initiator (DMAEMA).

** Each entry is the mean value (standard deviation) for degree of postcure ($N = 3$); percentage of RDB ($N = 5$); polymerization shrinkage ($N = 6$). Means with the same letter, i.e., A, B, and C, for each characteristic, are not significantly different at the $\alpha = 0.05$ level ($P > 0.05$).

ble bonds in the cured resin. The data for remaining double bonds of experimental oligomers and BisGMA are also listed in Table 4. Except for EPBPA #1, the remaining double bonds of the experimental oligomers are not statistically significant from that of the BisGMA system.

Water Sorption

Water sorption has been identified as the principal factor in the physical and chemical degradation of resin composite materials. Water sorption has been directly related to degradation of the filler-matrix interface, plasticization of the matrix, and reduction of the tensile strength and wear resistance, as well as other properties of the material (Kalachandra and Wilson 1992) [14].

Figure 4 shows the water sorption values of visible light-cured experimental oligomers and the BisGMA control. For a period of six-month immersion in 37°C water, the water sorption for the BisGMA control was significantly higher than each of the experimental resins. Although the water sorption decreases for the higher esterification degree of EPBPA oligomers, the differences are not statistically significant. The percent weight increases for the neat resins after 30 day-immersion in acetone, ethanol, and water are shown in Table 5. As expected, the VLC neat resins tend to absorb more solvents, such as acetone and ethanol, than water. Again, the percent weight increase of BisGMA control is significantly higher than that of either EPBPA #2 or EPBPA #3 in the above three liquids. The liquid absorption in the neat resins is a diffusion-controlled process determined by the chemical composition and structure of each resin, the affinity of the polymer matrix with the liquid medium, the extent of cure, and the sample size and shape. Here, for each resin, the composition, the sample size and shape, and the extent of cure were almost kept the same. Since the neat resins had the largest weight increases in acetone, as shown in Table 5, it seems that acetone has the best affinity with BisGMA and its analogous resins, followed by ethanol and water.

Aliphatic hydroxyl groups are usually considered to increase the water sorption of the cured resin due to enhanced hydrophilicity. This partially accounts for the high water sorption of BisGMA neat resins. In addition, Kalachandra [14] found that the water sorption is in direct proportion to the total weight percent of oxygen content in the cured resins. In fact, our experimental oligomers EPBPA #1-3 also contain hydroxyl groups, but the weight percent ratios of hydroxyl group and the total weight percent of oxygen content in the molecules are significantly lower than the BisGMA control. Moreover, the

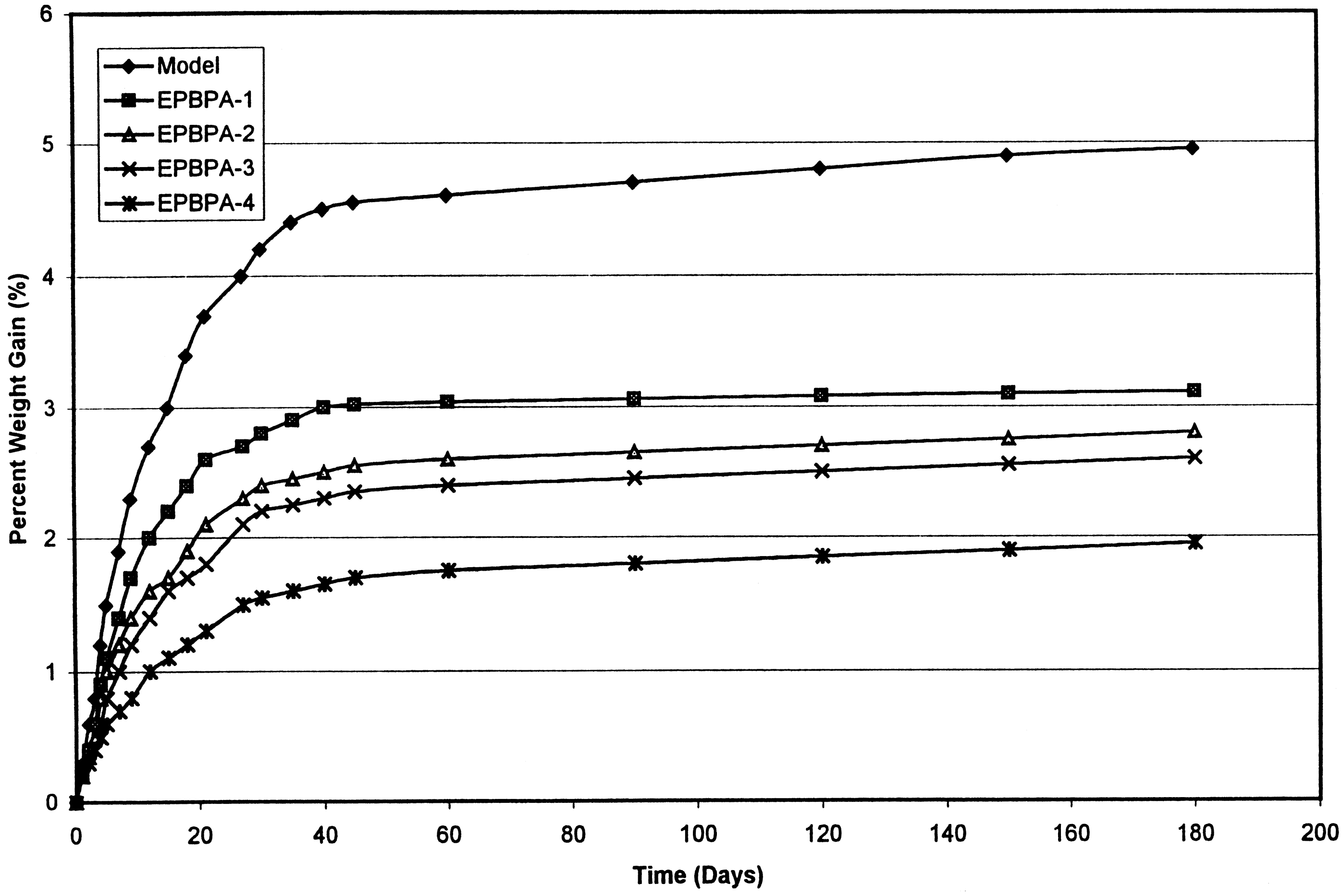


Figure 4. Sorption of water for the VLC experimental and BisGMA neat resins at 37°C, using distilled water.

TABLE 5. The Percent Weight Increase of VLC Neat Resins after 30-day Immersion in Several Liquids at 37°C

Oligomer/TEGDMA Mixtures*	Weight Gain (%) (SD)**		
	Water	Acetone	75 % Ethanol***
BisGMA	4.3 (0.2) A	14.1 (0.3) A	11.3 (0.2) A
EPBPA #2	2.3 (0.4) B	11.4 (0.2) B	7.2 (0.8) B
EPBPA #3	2.2 (0.4) B	11.0 (0.3) C	6.4 (0.5) C

*** Oligomer/TEDGMA 50/50 (wt/wt) with 0.5 wt. % initiator (CQ) and 1.0 wt. % co-inititiator (DMAEMA).**
****Each entry is the mean value (standard deviation) for a group of five specimens (N = 5); means with the same letter, i.e., A, B, and C, for weight gain, are not significantly different at the $\alpha = 0.05$ level ($P > 0.05$).**
*****Ethyl alcohol: water (75:25, vol:vol).**

experimental resins contain more double bonds in a single molecule. Thus, these multi-methacrylate oligomers form highly dense, crosslinked structures when cured, along with formation of rigid molecular chains. Presumably, the hydroxyl group may be restrained in such a network. All these factors contribute to the lower water sorption for the experimental resins.

CONCLUSION

This effort is part of a continuing study to obtain a better understanding of the structure-property relations needed to obtain improved composites and to develop new and improved resin matrix systems for dental composite applications.

A family of multi-methacrylates, based on enzyme oligomerized bisphenol A resins, was synthesized, characterized by IR and NMR, and evaluated.

These multi-methacrylates are miscible with TEGDMA, a common diluent used in dental composites, and able to form photo-polymerizable monomer mixtures.

Neat resins prepared from these polyfunctional oligomers possess comparable visible light-curing characteristics to the BisGMA control, along with having lower polymerization shrinkage and lower sorption of water and other liquids.

This new type of polyfunctional methacrylate oligomers has potential application in formulating dental composites with improved properties, as well as composites for a variety of other applications, including such materials as bone cements.

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Received October 15, 1998
Revised date December 10, 1998

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