

The effect of the strength and wetting characteristics of Bis-GMA/TEGDMA-based adhesives on the bond strength to dentin

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ABSTRACT

Objectives: This study investigated the effect of the strength and wetting characteristics of adhesives on the bond strength to dentin. The experimental adhesives containing various ratios of hydrophobic, low-viscosity Bis-M-GMA, with Bis-GMA and TEGDMA, were made and evaluated on the mechanical properties and bond strength to dentin.

Materials and Methods: Five experimental adhesives formulated with various Bis-GMA/Bis-M-GMA/TEGDMA ratios were evaluated on their viscosity, degree of conversion (DC), flexural strength (FS), and microtensile bond strength (MTBS). The bonded interfaces were evaluated with SEM and the solubility parameter was calculated to understand the wetting characteristics of the adhesives.

Results: Although there were no significant differences in the DC between the experimental adhesives at 48 hr after curing ($p > 0.05$), the experimental adhesives that did not contain Bis-GMA exhibited a lower FS than did those containing Bis-GMA ($p < 0.05$). The experimental adhesives that had very little to no TEGDMA showed significantly lower MTBS than did those containing a higher content of TEGDMA ($p < 0.05$). The formers exhibited gaps at the interface between the adhesive layer and the hybrid layer. The solubility parameter of TEGDMA approximated those of the components of the primed dentin, rather than Bis-GMA and Bis-M-GMA.

Conclusions: To achieve a good dentin bond, a strong base monomer, such as Bis-GMA, cannot be completely replaced by Bis-M-GMA for maintaining mechanical strength. For compatible copolymerization between the adhesive and the primed dentin as well as dense cross-linking of the adhesive layer, at least 30% fraction of TEGDMA is also needed. [J Kor Acad Cons Dent 2011;36(2):139-148.]

Key words: Bis-GMA derivative; Bond strength; Dentin adhesive; Physical properties; Solubility parameter
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INTRODUCTION

The major role of the adhesive layer in a bonding

complex is to protect the hybrid layer from detrimental stresses during polymerization or masticatory function. A strong, hydrophobic and low-viscosity

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adhesive resin copolymerizes with the primer penetrating onto the decalcified dentinal surface and simultaneously offers bonding receptors for copolymerization with the restorative resin composite.¹ The adhesive resin layer should have strength to resist shrinkage stresses from the polymerizing resin composite and maintain bond strength.² Improvement in the mechanical properties of the adhesive layer is an important factor in increasing dentin bond strength.^{3,4}

There were various efforts to increase the mechanical properties of the adhesive layer: loading fillers, increasing the degree of conversion and improving the monomer system. It was suggested that adding fillers to the adhesives would increase the mechanical strength of the adhesive layers.⁵⁻⁷ To achieve higher bond strength and to improve the stability of the adhesive interface over time, the degree of conversion should be increased optimally by improving photoinitiator system and comonomer-solvent mixture system.^{3,8-11} Most contemporary adhesive resins consist primarily of hydrophobic base resin monomers, such as 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane (Bis-GMA) and urethane dimethacrylate (UDMA), relatively hydrophilic methacrylate monomers, such as triethylene glycol dimethacrylate (TEGDMA) as a viscosity regulator and 2-hydroxyethyl methacrylate (HEMA) as a wetting agent.¹ However, such diluent monomers like TEGDMA have been shown to adversely affect the efficiency and stability by increasing water sorption and curing shrinkage.^{12,13} For this reason, new hydrophobic resin monomer systems including low-viscosity Bis-GMA alternatives and low-shrinkage Silorane have been studied to modify the frequently used Bis-GMA/TEGDMA system and to simultaneously improve the mechanical properties and long-term stability of the dentin adhesives.¹⁴⁻¹⁶

2,2-bis[4-(2-methoxy-3-methacryloyloxy propoxy) phenyl] propane (Bis-M-GMA), a Bis-GMA derivative, is a new base resin monomer obtained by substituting the methoxy groups for hydroxyl groups in the Bis-GMA.¹⁴ The high viscosity of Bis-GMA was dramatically reduced from 574 Pa · s to 3.65 Pa · s by the removal of network junctions, i.e., hydrogen bonds between molecules. Comparing to the resin composite made of Bis-GMA/TEGDMA mixture, the

dental resin composite made of Bis-M-GMA/TEGDMA mixture exhibited low polymerization shrinkage, low water absorption, and increased mechanical properties. By using Bis-M-GMA instead of Bis-GMA, the amount of diluent TEGDMA and its adverse effects on the physical and mechanical properties of dental composites can be minimized.¹⁴

This study investigated the effect of the strength and wetting characteristics of each monomer on the bond strength of the adhesive. For the purpose, this study evaluated whether a novel hydrophobic and low-viscosity Bis-M-GMA monomer with bulky substituent groups could be an alternative to Bis-GMA without sacrificing the degree of conversion and the mechanical properties of the dentin adhesives. Experimental adhesives containing various ratios of Bis-GMA/Bis-M-GMA/TEGDMA were prepared, and the viscosity, degree of conversion and flexural strength of the experimental adhesives and the bond strength of the adhesives to dentin were evaluated. The bonded interfaces were evaluated with scanning electron microscopy (SEM), and the solubility parameter of each monomer was also calculated. It was hypothesized that the experimental adhesive containing Bis-M-GMA instead of Bis-GMA as a base monomer would have comparable physical properties and bond strength to the frequently used Bis-GMA/TEGDMA system.

MATERIALS AND METHODS

Preparation of experimental adhesives

The new Bis-GMA derivative, i.e., Bis-M-GMA, was prepared by substituting a methoxy group for the hydroxyl group in Bis-GMA, according to the earlier described method (Figure 1).¹⁴ Bis-GMA and TEGDMA were obtained from Aldrich Chemical Co. and were used as received. Five experimental adhesives formulated with various Bis-GMA/Bis-M-GMA/TEGDMA ratios were prepared as shown in Table 1, and 1.0 wt% of camphorquinone (CQ) and 1.0 wt% of ethyl 4-dimethylaminobenzoate (EDMAB) were added to each mixture. The adhesive of the Scotchbond Multi-Purpose adhesive system (SBMP, 3M ESPE Dental Products, St. Paul, MN, USA) was used as a reference.

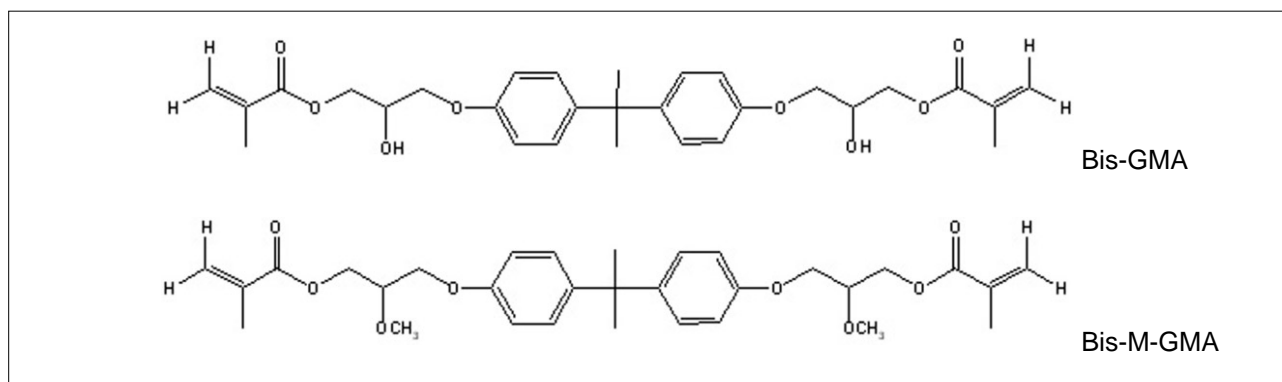


Figure 1. The chemical structures of Bis-GMA and Bis-M-GMA. Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane; Bis-M-GMA, 2,2-bis[4-(2-methoxy-3-methacryloyloxy propoxy) phenyl] propane

Measurement of viscosity (η)

The viscosities (η) of the experimental adhesives were measured by means of a rotational rheometer (AR 2000, TA instrument, New Castle, DE, USA). The geometry used for the steady shear test was a cone and plate. The measurements were carried out in a dark room at 25°C at a shear rate range of 0 to 50 s⁻¹.

Measurement of degree of conversion (DC)

The degree of conversion (DC, %) was measured using Fourier Transform Infrared Spectroscopy (FT-IR, Spectrum One, Perkin-Elmer Instruments, Shelton, CT, USA) under constant nitrogen flow. Press-molded potassium bromide (KBr) disks were used as window materials. The spectra were obtained in transmission mode with 128 scans at a resolution of 4 cm⁻¹. After collecting the background spectrum with two blank KBr disks, the respective adhesive was dispersed onto a KBr disk with a disposable brush, and the spectrum of the uncured adhesive between the two KBr disks was collected. The sample was then cured for 20 seconds using a light-curing unit (Hilux Ultra Plus, Benlioglu Dental Inc., Ankara, Turkey; Light intensity: 600 mW/cm²), and the spectrum of the cured adhesive was collected immediately after curing. The cured samples were stored for 48 hours in a dark container, and the spectrum of the cured adhesive at 48 hours after curing was also obtained.

Using a baseline technique in absorbance mode, the

DC was calculated from the ratio of the corrected absorbance peak height of aliphatic double bonds at 1637 cm⁻¹ before and after polymerization. The correction was performed with an aromatic absorbance peak height of 1608 cm⁻¹ as an internal reference.¹⁷

$$\text{DC (\%)} = 100 - \frac{[\text{abs (aliphatic C=C) / abs (aromatic C=C)}]_{\text{polymer}}}{[\text{abs (aliphatic C=C) / abs (aromatic C=C)}]_{\text{monomer}}} \times 100$$

Measurement of flexural strength (FS)

The experimental adhesive was poured into a rectangular stainless steel mold with a dimension of 1 × 1 × 12 mm, attached to a slide glass with double-sided Teflon adhesive tape. The specimen was cured for 20 seconds using the same light-curing unit with a wide light-guide tip (13 mm in diameter). The specimen was stored in a dark container at room temperature with 100% relative humidity. After 24 hours, the flexural strength (FS) was measured with three-point loading using a universal testing machine (UTM, LF plus, LLOYD Instruments LTD, Fareham Hampshire, UK). The load was applied to the center of the specimens placed on the support with a 7.7 mm span at a crosshead speed of 1.0 mm/min. The flexural strength of each specimen was determined by

$$\text{FS} = \frac{3Fl}{(2bh^2)}$$

where F, l, b, and h are the maximum load, the distance between the supports, the specimen width, and the specimen height, respectively.

Measurement of microtensile bond strength (MTBS)

Twenty-six extracted human molars without caries or restoration were cleaned and immersed in a 0.5% chloramine-T solution for one week and were then stored in deionized water at 4°C. The apical third of the root was imbedded in a resin block with self-curing acrylic resin. In order to expose the dentin, the crown was cut perpendicular to the long axis of the tooth using a low speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) with a coolant. The exposed dentin surface was polished with 500-grit silicon carbide paper using an automatic polishing machine (Rotopol-V, Struers Ltd, Glasgow, UK) under running water.

The polished dentin surface was etched with a 35 wt% phosphoric acid etching gel (SBMP, 3M ESPE Dental Products) for 15 seconds, rinsed with deionized water for 15 seconds and blot-dried. Primer (SBMP, 3M ESPE Dental Products) was applied twice onto the moist dentin surface and was dried completely with air flow. The assigned experimental adhesive was applied and cured for 20 seconds using the same light-curing unit. A resin composite (Z-250, shade A2, 3M ESPE Dental Products) was built up in three increments to about 3.5 mm in thickness. Each increment was cured for 20 seconds according to the manufacturer's instructions.

After storage in deionized water at room temperature for 24 hours, the sample was trimmed to a rectangular shape using a low-speed diamond saw. In order to form the specimens into an hourglass-shape, two grooves were made along the dentin-resin interface with a diamond bur mounted in a low-speed press drill. The narrowest portion of the hourglass was about 1 mm wide. The specimen was then serially sectioned with a low-speed diamond saw into multiple slices about 0.6 mm thick. Six to twelve sections were obtained per tooth. The outer-most sections were kept for SEM evaluation.

Forty-eight hours after bonding, the specimen was attached to opposing arms of a testing device with a small amount of cyanoacrylate cement (Super Glue Gel, 3M). The whole assembly was set and tested in the UTM at a cross-head speed of 1.0 mm/min, and

the maximum load (kg) at fracture was recorded.

Examination of bonded interfaces

The bonded interfaces of randomly selected specimens from each group were replicated with epoxy resin after treatment with 6N HCl for 5 seconds and 3.5% NaOCl for 5 minutes. Samples were evaluated at various magnifications using a field-emission SEM (S-4700, HITACHI high technologies Co., Tokyo, Japan) operating at 15 kV.

Calculation of solubility parameters (δ_t)

In this study, the solubility parameters of the monomers were calculated for the total cohesive energy density (δ_t) by Hoy's method using the following formula^{18,19}:

$$\delta_t = \frac{F_t + B}{V}$$

where δ_t is Hoy's solubility parameter for the total cohesive force, F_t is the molar attraction function, V is the molar volume of the solvent molecule or the structural unit of the polymer, and B is a "base value" of 277. The F_t and V values were taken from the reference.¹⁹

Statistical Analysis

Since the data of the FS showed a normal distribution but did not satisfy homogeneity of variance, they were analyzed using the nonparametric Kruskal-Wallis test. Multiple comparisons were further conducted using the Mann-Whitney test with a significance level of 0.05. The data of the DC was not normally distributed (Shapiro-Wilk normality test) and thus the Kruskal-Wallis analysis of rank and the median test were applied again for the statistical analysis of nonparametric procedures. The data of the MTBS was analyzed using a mixed-level repeated measure analysis of variance (ANOVA) and a mixed-level repeated measure analysis of covariance (ANCOVA) using the general linear model (GLM) procedure to consider the inter-specimen differences. While inter-location differences in the same tooth

were not significant, inter-tooth differences were statistically significant ($p < 0.0001$).

RESULTS

Table 1 shows the viscosities and the DC of the experimental adhesives immediately after curing and 48 hours after curing, the FS of the cured experimental adhesives, and the MTBS of the adhesive to dentin. By adding only 20 wt% of TEGDMA, the Bis-M-GMA/TEGDMA mixture showed a lower viscosity than that of the Bis-GMA/TEGDMA 60/40 mixture. According to the content of Bis-M-GMA, the viscosities of the experimental adhesives were controlled from very thin 0.07 Pa · s to very thick 3.65 Pa · s, comparable to that of commercially available SBMP ($\eta = 0.38$ Pa · s). Immediately after curing, the DC of the 0/100/0 adhesive was significantly lower than those of the other groups ($p < 0.05$), but after 48 hours, there was no statistically significant difference between the DCs of the experimental adhesives ($p > 0.05$). Although there were no significant differences in the DC after 48 hours of curing, the experimental adhesive systems that did not contain Bis-GMA (0/60/40, 0/80/20, and 0/100/0 adhesives) exhibited

a lower FS value than did those containing Bis-GMA (60/0/40 and 40/30/30 adhesives) ($p < 0.05$). Among those that did not contain Bis-GMA, the experimental adhesives that contained very little to no TEGDMA (0/80/20 and 0/100/0 adhesives) also showed significantly lower MTBS than did the experimental adhesives containing a higher content of TEGDMA ($p < 0.05$).

The representative SEM images of the experimental adhesives 60/0/40 and 0/60/40, which had higher MTBS, showed an intimately adapted resin-dentin interface and well-developed resin tags (Figures 2a, and 2b). However, in the images of the 0/100/0 adhesive, sparse resin tags were short and gap was observed between the hybrid layer and the underlying dentin (Figure 2c). The calculated values of Hoy's solubility parameter of the monomers used in the experimental adhesives and the ingredients of the SBMP primer are shown in Table 2. The Hoy's solubility parameter of Bis-M-GMA was the lowest among the evaluated monomers (Table 2). As a result, the difference from those of polyalkenoic acid and HEMA, which might be major constituents of the primed surface, was the greatest.

Table 1. The ratios of monomers in the experimental adhesives used in this study, viscosity (η), the degree of conversion immediately after curing and at 48 hours after curing (DC, $n^* = 6 - 9$), the flexural strength (FS, $n = 10$) of the bulk adhesives and the microtensile bond strength (MTBS, $n = 23 - 30$) of the adhesives to dentin

Bis-GMA/ Bis-M-GMA/ TEGDMA (wt%)	η (Pa · s)	DC (%), median		FS (MPa)	MTBS (MPa)
		Immediately after curing	48 hr after curing		
60/0/40	0.45	70.6 ^a	85.5 ^a	82.2 (10.0) ^{a**}	44.8 (12.6) ^a
40/30/30	0.42	73.2 ^a	87.7 ^a	75.0 (7.3) ^a	45.2 (8.3) ^a
0/60/40	0.07	73.4 ^a	88.4 ^a	37.0 (4.3) ^b	44.5 (10.5) ^a
0/80/20	0.30	72.1 ^a	86.8 ^a	35.0 (2.3) ^b	33.4 (6.9) ^b
0/100/0	3.65	58.8 ^b	73.6 ^a	40.3 (3.5) ^b	27.0 (9.6) ^b
SBMP***	0.38	68.9	86.0	80.0 (16.6)	45.3 (9.6)

Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane; Bis-M-GMA, 2,2-bis[4-(2-methoxy-3-methacryloyloxy propoxy) phenyl] propane; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; DC, degree of conversion; FS, flexural strength; MTBS, microtensile bone strength.

*n is the number of specimens.

**The numbers in parentheses are standard deviations. Mean or median values with the same superscript within a column are not significantly different ($p > 0.05$).

***SBMP was not included in the statistical evaluation due to its different formulation.

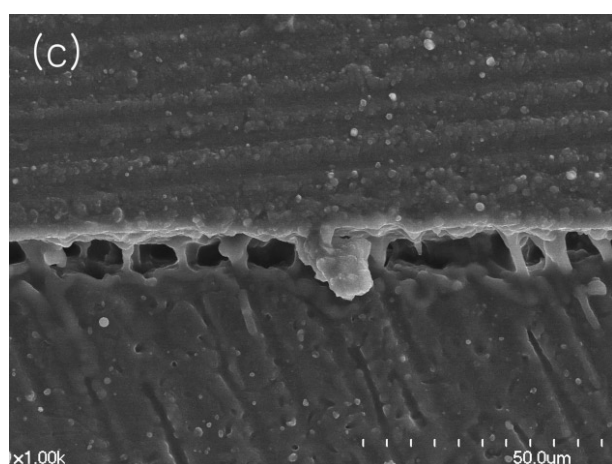
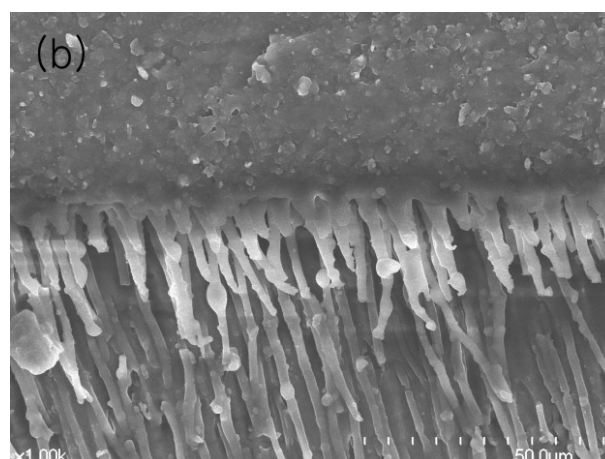
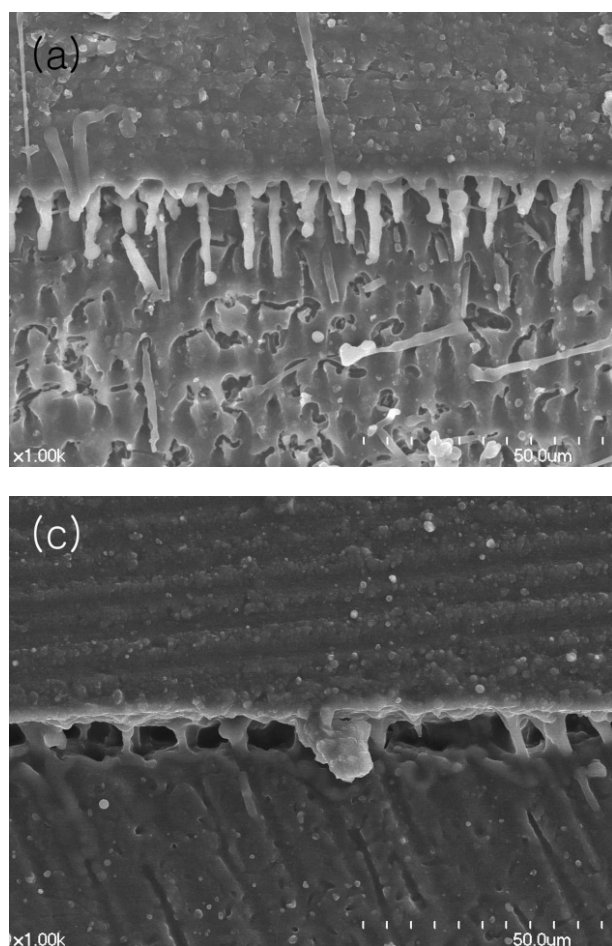


Figure 2. Representative SEM images of the resin-dentin interfaces bonded with the experimental adhesives of 60/0/40 (a), 0/60/40 (b) and 0/100/0 (c). The SEM images of the experimental adhesives 60/0/40 and 0/60/40, which had higher MTBS, showed an intimately adapted resin-dentin interface and well-developed resin tags (a and b). However, in the images of the 0/100/0 adhesive, sparse resin tags were short and gap was observed between the hybrid layer and the underlying dentin (c). SEM, scanning electron microscopy; MTBS, microtensile bone strength.

Table 2. The solubility parameters (δ_i) calculated by the Hoy method

	Bis-GMA	Bis-M-GMA	TEGDMA	HEMA	Poly alkenoic acid	Water
Solubility parameter (J/cm^3) ^{1/2}	18.6	16.9	20.5	24.2	22.0	48.0

Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane; Bis-M-GMA, 2,2-bis[4-(2-methoxy-3-methacryloyloxy propoxy) phenyl] propane; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate.

DISCUSSION

As the most commonly used base monomer in current dental composite restorative formulations,¹³ Bis-GMA exhibits very high viscosity because of the hydrogen bonding interactions that occur between the hydroxyl groups.^{20,21} Thus, it must be diluted with a less viscous monomer like TEGDMA.^{14,22} The current generation of dentin adhesives has been criticized as being too hydrophilic and absorbing too much water.²³ This lowers their stiffness when com-

pared to those of more hydrophobic resins.²⁴ On the contrary, although a novel hydrophobic monomer Bis-M-GMA has a higher molecular weight than Bis-GMA, it exhibits a dramatically lower viscosity due to the absence of hydrogen bonding as a result of substituting methoxy groups for the hydroxyl groups. A partial or complete replacement of Bis-GMA with Bis-M-GMA was expected to minimize the need for diluent TEGDMA and its adverse effects. With at most 20% TEGDMA, the viscosities of the experimental adhesives containing Bis-M-GMA were com-

parable to those of the SBMP adhesive. If dentin is bonded with more hydrophobic resins, they would absorb less water, plasticize less, and produce more durable bonds.²⁵ Therefore, the experimental adhesives made of hydrophobic Bis-M-GMA were expected to satisfy these characteristics. It was reported that filler-loaded dental composites made from Bis-M-GMA and TEGDMA mixtures had better mechanical strength, lower water uptake and lower curing shrinkage than those of Bis-GMA and TEGDMA mixtures.¹⁴

Even though the DC was the same as the other groups, except the 0/100/0 adhesive, the adhesives containing traditional Bis-GMA (60/0/40 and 40/30/30 adhesives) showed significantly higher FS than did those without the monomer (0/60/40, 0/80/20, and 0/100/0 adhesives). The experimental adhesives with higher contents of Bis-M-GMA and lower contents of TEGDMA (0/80/20 and 0/100/0 adhesives) showed significantly lower MTBS values than those with higher contents of TEGDMA. The 0/60/40 experimental adhesive exhibited significantly higher MTBS than those with lower contents of TEGDMA (0/80/20 and 0/100/0 adhesives) in spite of its similar flexural strength to them. The bond strength approached to those of the adhesives having at least 30 wt% of TEGDMA (60/0/40 and 40/30/30 adhesives). Based on the DC and FS measurements, a highly cross-linked and strong Bis-GMA backbone might be needed for polymer strength. However, when an appropriate amount of TEGDMA was added to control the viscosity and to increase the wetting characteristics, the adhesive made of Bis-M-GMA, instead of Bis-GMA, also exhibited comparable dentin bond strength to those of the adhesive containing Bis-GMA/TEGDMA mixture.

Several investigations have revealed that the mechanical properties of restorative resins are dependent not only on the degree of conversion of the double bonds, but also on the nature of the involved monomer molecules.^{8,26,27} A high cross-linking density in the polymer creates a dimensionally stable material and enhances the mechanical properties of the polymer.²⁸ The addition of Bis-M-GMA led to an increase in the mean molecular weights of the monomers in the experimental adhesive. This might

lower the cross-linking density and adversely affect the mechanical properties of the adhesive.^{28,29} Stiff Bis-GMA molecules appeared to influence the FS of experimental adhesives.³

Even in the Bis-M-GMA/TEGDMA mixtures, at least 30 wt% TEGDMA might be necessary to maintain the DC, mechanical strength, and bond strength. First, TEGDMA plays an important role in cross-linking. When Bis-GMA was used in fractions of more than 60 wt% in a Bis-GMA/TEGDMA mixture, that is, less than 40 wt% TEGDMA, autodeceleration of the polymerization reaction decreased the DC owing to the increased viscosity in the adhesive mixtures.⁴ However, in the Bis-M-GMA/TEGDMA mixture, a decrease of TEGDMA content to 20 wt% had no influence on the DC of the experimental adhesive due to low viscosity of Bis-M-GMA. Although the molecular weight of Bis-M-GMA is greater than that of Bis-GMA, Bis-M-GMA is relatively free from diffusion limitations due to the absence of hydrogen bonds. However, the 0/100/0 experimental adhesive showed a significantly lower conversion rate immediately after curing. This suggests that dilution with a highly mobile monomer like TEGDMA is needed to allow the higher conversion rate of Bis-M-GMA.

Secondly, small TEGDMA molecules can infiltrate into the hybrid layer where they copolymerize with the adhesion promoters of the primer.³⁰ Since SBMP primer does not contain a photoinitiator, polymerization of the primer can be triggered by the radicals transmitted from the polymerizing adhesive resin. Bis-GMA and Bis-M-GMA are bulk molecules and do not have free mobility. Relatively small TEGDMA molecules effectively transmit reactive chains to the primer. In the case of 0/80/20 and 0/100/0 adhesives with very little to no TEGDMA, the primer intermingled with the decalcified collagen network could not be fully polymerized. As a result, the bonded complex could not resist the polymerization shrinkage stress of the overlying composite resin.³ On the contrary, in SEM observation, the 0/60/40 adhesive showed intimately adapted resin-dentin interfaces without such a gap observed in the 0/100/0 adhesive. This gives an explanation for the significantly higher bond strength of the 0/60/40 experi-

mental adhesive in spite of its low flexural strength similar to those of the 0/80/20 and 0/100/0 adhesives.

This might be related to the wetting characteristics of the adhesives to primed dentin. It was revealed that the adhesive resin should have specific wetting characteristics matching those of the conditioned dentin in order to ensure optimum bonding.^{31,32} The wetting behavior may be expressed in terms of the solubility parameter (δ) and polarity of the adhesive resin.³¹ In this study, the total cohesive energy density (δ_t) for each monomer was calculated using the Hoy's method to compare the wetting characteristics of the experimental adhesives to those of the primed dentin.¹⁸ According to Hildebrand, mutually miscible liquids may have similar cohesive energy densities.³³ Using cohesive energy as an additive property for common small molecules, many authors, including Hoy, have developed group contribution models for solubility parameter calculations using atomic groups.¹⁸ In addition to these methods for determining the solubility parameters of individual molecular components, it has also been demonstrated that the solubility parameters of a mixture of more than one component can also be calculated by incorporating the volume-wise contributions of the solubility parameters of the individual components of the mixture.³⁴ Transferring this concept to dentin bonding, an adhesive resin with appropriate solubility parameters will be able to penetrate and adapt to the underlying primed dentin. It has been reported that resin-dentin bond strengths were related to the solubility parameters of dentin adhesives, and the maximum bond strength presumably occurred when the solubility parameter of the resin was close to that of the primed surface.^{25,31,32,35,36} The results of this study coincide with those of previous reports. As shown in Table 2, the ingredients of the SBMP primer and Bis-M-GMA have relatively high δ_t values and the lowest δ_t value, respectively. The δ_t value of demineralized dentin has been reported to be higher than 22.5 (J/cm³)^{1/2} for various concentrations of water and peptides.²⁵ 0/80/20 and 0/100/0 adhesives have relatively lower δ_t values compared with other groups due to the increased Bis-M-GMA and decreased TEGDMA. This can also lead to gap formation and it

can be exaggerated by polymerization shrinkage stress as shown in SEM observations (Figure 2c). Molecular mobility and a relatively high δ_t value of TEGDMA contributed to the copolymerization between the adhesive resin and the underlying primed dentin. Therefore, the incompatibility in solubility parameters between the ingredients of the adhesives and the primer would cause difficulties in the wetting of the adhesive to primed dentin. In addition to the lower FS of the adhesives containing no Bis-GMA, a greater difference in the solubility parameter of Bis-M-GMA from the primed dentin might attribute to the lower MTBS.

In this study, the experimental dental adhesives made of Bis-M-GMA did not show sufficient mechanical or bond strengths. An overall fraction of at least 30% TEGDMA was required to maintain proper bond strength of the adhesive. As a stiff base monomer, Bis-GMA was necessary for maintaining mechanical strength, and it could not be completely replaced by Bis-M-GMA. Therefore, the hypothesis of this study was rejected. In order to achieve good dentin bond strength, compatibility in solubility parameters and efficient copolymerization between the adhesive and the primed dentin were required, as well as high flexural strength of densely cross-linked adhesive layer.

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국문초록

2,2-Bis(4-(2-methoxy-3-methacryloyloxy propoxy) phenyl) propane을 함유한
상아질 접착레진의 물성이 접착강도에 미치는 영향

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연구목적: 치과용 접착제에서 기저단량체로 사용되는 Bis-GMA를 Bis-M-GMA로 대체한 접착레진이, Bis-GMA/TEGDMA를 사용한 접착제와 비교할 만한 물성과 접착강도를 가지는지 알아보려고 하였다.

연구 재료 및 방법: 다양한 구성비의 Bis-GMA, Bis-M-GMA 및 TEGDMA를 함유한 접착레진을 제작하여, 점도(η), 중합률(DC) 및 굴곡강도(FS)를 측정하고, 상아질에 대한 미세인장접착강도(MTBS)를 측정하였다. 해석을 위해 각 단량체들의 용해도 상수(δ_i)를 비교하였고, 접착계면을 주사전자현미경으로 관찰하였다.

결과: Bis-M-GMA의 점도는 3.65 Pa·s로 크게 감소하였다. Bis-GMA를 함유하지 않는 접착레진은 Bis-GMA를 함유한 접착레진에 비해 낮은 굴곡강도를 보였다($p < 0.05$). TEGDMA 함량이 낮은 접착레진은 TEGDMA 함량이 높은 접착레진에 비해 낮은 미세인장접착강도를 보였으며($p < 0.05$), 주사전자현미경 사진에서 균열이나 틈이 관찰되었다.

결론: 높은 접착강도를 얻기 위해서는 접착레진의 높은 굴곡강도와 초기 중합률 뿐 아니라, 접착레진과 primer 처리된 상아질 사이의 용해도 상수의 적합성도 요구되었다.

주요단어: 물성; 상아질 접착제; 용해도 상수; 접착력; Bis-GMA 유도체