

## Degree of conversion and plasticization of dimethacrylate-based polymeric matrices: influence of light-curing mode

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**Abstract:** This study analyzed the correlation between the degree of conversion (DC) and the plasticization (P) of experimental dimethacrylate-based polymeric matrices. Six polymeric matrices produced with Bis-GMA (G), UDMA (U) and TEGDMA (T) dimethacrylate monomers were studied: G, U, GT55, GT73, GU55, GU73 - (55 = 50/50wt% and 73 = 70/30wt%). Photoactivation was induced by camphoroquinone/ethyl *N,N*-dimethyl-4-aminobenzoate. The matrices were light-cured using two modes: Standard (S) - 850 mW/cm<sup>2</sup> for 20 s, and Gradual (G) - 100 up to 1000 mW/cm<sup>2</sup> for 10 s + 1,000 mW/cm<sup>2</sup> for 10 s. The degree of conversion (%) was obtained by using a FT-IR spectrometer equipped with an attenuated total reflectance crystal (ATR), and plasticization was estimated by the softening effect of ethanol. No correlation was found between the degree of conversion and plasticization ( $r = 0.32 / P = 82.35 \pm 0.85DC$ ). The means of DC% were: GT55 ( $66.18 \pm 3.29$ ) > U ( $59.34 \pm 5.61$ ) > GT73 ( $54.97 \pm 7.89$ ) > GU55 ( $50.60 \pm 6.26$ ) > GU73 ( $44.02 \pm 4.85$ ) > G ( $38.25 \pm 3.35$ ), ( $P < 0.05$ ). The matrices with TEGDMA showed the highest plasticization. The light-curing mode influenced only G > S plasticization ( $P < 0.05$ ). (J. Oral Sci. 50, 315-321, 2008)

**Keywords:** dimethacrylate monomers; degree of conversion; plasticization, light-curing mode.

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### Introduction

Resin-based composites are among the most widely used materials in restorative dentistry. In general, these materials are constituted of a polymeric matrix based on dimethacrylate monomers, such as 2,2-bis[*p*-(2'-hydroxy-3'methacryloxypropoxy)phenyl]propane (bis-GMA), 1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane (UDMA), and triethylene glycol dimethacrylate (TEGDMA), inorganic filler particles coated with a methyl methacrylate-functional silane coupling agent to bond the filler to the organic matrix, and a photoinitiator system to permit photoactivation by light sources (1,3). Bis-GMA is the most widely used monomer in polymeric matrices of resin-based composites (1). However, due to intermolecular hydrogen bonding, its high molecular weight and the presence of two aromatic rings in its molecule, this monomer shows a high viscosity and a low mobility, characteristics that could interfere with the degree of conversion of the polymeric matrix (4). Thus, low-molecular-weight monomers such as TEGDMA are added to reduce the polymeric matrix viscosity (5), thereby increasing the filler incorporation and degree of conversion of the composite (4,6). UDMA is an aliphatic high-molecular-weight monomer also used in polymeric matrices. This monomer has two imino groups (-NH-), which form intermolecular hydrogen bonds, increasing its viscosity. On the other hand, its aliphatic molecule gives the polymer chain great mobility, thereby increasing the degree of conversion and the crosslink

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density of the polymeric matrix (3). Other dimethacrylate monomers have been proposed to formulate polymeric matrices for resin-based composites (6). However, present day composite materials continue to be Bis-GMA, TEGDMA, and UDMA based.

Several light-curing sources and modes can be used to start the polymerization reaction of resin-based composites (7-9). Light-curing with higher irradiance leads to a higher degree of conversion. However, greater polymerization shrinkage stresses, and consequently greater gap formation, develop at the cavity wall-resin-based composite interfaces (10). Thus, gradual light-curing modes that use an initial low irradiance have been introduced to allow polymerization shrinkage to be reduced by stress relief during resin-based composite setting (11). Although some published studies have shown that these light curing modes do not reduce the degree of conversion of resin-composite or dental polymers (11,12), it has also been suggested that a more linear polymer network could be formed (13,14). This structure can increase the uptake of solvent by a polymer (15). Some published studies have shown that the chemical structure of dimethacrylate monomers can affect the amount of water or solvents absorbed by dimethacrylate-based dental resins and resin-based composites (16,17). Several drawbacks, such as a reduction in hardness, increased surface wear and decreased mechanical properties can occur due to this solvent uptake (18). These effects are derived from polymeric matrix plasticization by water and solvents.

Published studies have demonstrated a correlation between the degree of conversion and mechanical properties of dental resins or resin-based composites (19-22). Thus, the degree of conversion has often been used as an indicator of the behavior of resin-composite restorations. However, earlier studies showed that the degree of conversion was not sufficient for characterizing the 3-dimensional structure of resin-based composites, and that sites with different amounts of C=C bonds, present as pendant groups or residual monomer, could coexist in the same polymer (23,24). This heterogeneity would produce a polymer network with a lower crosslink density and probably poor

mechanical properties.

This study investigated the correlation between the degree of conversion (DC) and plasticization (P) of six experimental dimethacrylate-based polymeric matrices, and evaluated the influence of light-curing mode on both responses. The study hypotheses were: 1) that light-curing mode would not affect the DC, 2) that plasticization would be greater with gradual light-curing, and 3) that no correlation would be found between the DC and P.

## Materials and Methods

### Materials

Six experimental dimethacrylate-based polymeric matrices were analyzed in this study (Table 1). The monomers Bis-GMA, UDMA, and TEGDMA were used as received, and without purification (Aldrich Chemical Co., Milwaukee, WI, USA). After being weighed in an analytical balance (AW 220, Shimadzu, Tokyo, Japan), the monomers were mixed by centrifugation at 2800 rev.min<sup>-1</sup> for 30 min, at 60°C (SpeedMixer DAG 150FVZ-K, FlackTech Inc., Hauschild, Germany). In each polymeric matrix, 0.25 wt% of camphoroquinone and 1.0 wt% of ethyl *N,N*-dimethyl-4-aminobenzoate (EDMAB) were introduced to act as photosensitizer and reducing agent, respectively. These reagents were added to the matrices in dark containers and mixed by hand at 60 ± 0.5°C until the mixtures were homogeneous.

All the specimens in this study were photoactivated with a quartz-tungsten-halogen unit (Optilux 501, Kerr, Danbury, CT, USA). Two light curing modes were used: Standard (S) - 850 mW/cm<sup>2</sup> for 20 s (17 J/cm<sup>2</sup>) and Gradual (G) - 100 up to 1000 mW/cm<sup>2</sup> for 10 s + 1,000 mW/cm<sup>2</sup> for 10 s (≅ 16 J/cm<sup>2</sup>). The radiant exposure was calculated as the product of the irradiance of the curing unit, determined by a radiometer (model 100, Demetron Inc., Danbury, USA), and the time of irradiation. For the Gradual mode, the radiant exposure was obtained as the sum of mean irradiance over the first 10 s multiplied by 10 s at 10 J/cm<sup>2</sup>, corresponding to the radiant exposure in the last 10 s of light exposure.

### Degree of conversion - DC%

Spectra of the uncured and cured specimens of each polymeric matrix were recorded by a FT-IR spectrometer (Varian 3100 FT-IR, Varian Inc., Palo Alto, CA, USA), equipped with an attenuated total reflectance crystal – ATR (MIRacle ATR, Pike Technologies, WI, USA) operating with 120 scans at a resolution of 4 cm<sup>-1</sup>. A volume of 0.7 µl of each polymeric matrix was collected with a micropipette, and then compressed between two polyethylene strips and two glass slides to produce a thin

Table 1 Composition of polymeric matrixes analyzed in this study

Polymeric Matrix	Composition (wt%)		
	Bis-GMA (G)	TEGDMA (T)	UDMA (U)
G	100		
U			100
GT55	50	50	
GT73	70	30	
GU55	50		50
GU73	70		30

film approximately 3.0 mm in diameter and 0.1 mm thick. Five films of each polymeric matrix were then light-cured in accordance with the two light-curing modes used, with the light tip in contact with the glass slide. FT-IR spectra of the cured specimens were recorded 24 h after dry storage at 37°C. The DC% was calculated from the ratio of the height of the absorbance peak of the aliphatic C=C bond (1,638 cm<sup>-1</sup>) relative to that of the carbonyl group (>C=O, 1,720 cm<sup>-1</sup>), used as an internal standard, obtained from the cured and uncured specimens, by means of the following equation:

$$DC(\%) = 100 \times [1 - (R_{\text{cured}} / R_{\text{uncured}})]$$

where R = peak at 1,638 cm<sup>-1</sup> / peak at 1,720 cm<sup>-1</sup>

### Plasticization measurement - P

Plasticization was estimated by the softening effect of ethanol. Single increments of each polymeric matrix were applied to an aluminum mold, 5 mm in diameter and 2 mm thick, covered with a polyester strip and a glass slide (0.1 mm thick) and light cured from the top, in accordance with the two light-curing modes (n = 5). After storage in air at 37°C for 24 h, the disks were embedded in epoxy resin with the top side in contact with a glass plate. After epoxy resin polymerization, the top disk surfaces were polished with 1,200-grit abrasive paper. The disks were loaded on a microhardness tester (model 2003, Buehler, Lake Bluff, USA) and five indentations were made on the top side with a Knoop diamond at a 100 g load and 15 s dwell time. The disks were then stored in 75% ethanol for 24 h and the procedure was carried out again. The plasticization (P) was estimated by using the difference between the mean values of Knoop diamond indentation depth ( $d_k$  - μm) after and before ethanol storage as described by Silva et al. (25).

$$P = d_{k \text{ after}} - d_{k \text{ before}}$$

### Statistical analysis

Statistical analysis was performed using Statgraphics 5.1 Software (Manugistics, Rockville, MD, USA). After the normal distribution of the errors and the homogeneity of variance had been verified by the Shapiro-Wilk and Levene tests, respectively, each test variable (DC and P) was analyzed separately by two-way ANOVA. The  $d_{k \text{ before}}$  data were subjected to one-way ANOVA. All the mean comparisons were made by the Student-Newman-Keuls test. Data were also subjected to linear regression analysis, with DC% as the independent variable and plasticization as the dependent variable. All statistical analyses were performed at a significance level of  $\alpha = 0.05$ .

## Results

Figure 1 shows a representative FT-IR spectrum recorded

for G polymeric matrix. Several absorbance peaks are evident between 500 cm<sup>-1</sup> and 4000 cm<sup>-1</sup>. Figure 2 shows the absorbance peaks used for calculating the DC(%): aliphatic C=C bond (1,638 cm<sup>-1</sup>) and >C=O (1,720 cm<sup>-1</sup>). The values of DC% obtained with the S and G light-curing modes are shown in Table 2. Two-way ANOVA detected a statistically significant influence of polymeric matrix independent factor ( $P < 0.05$ ). Comparisons by the Student-Newman-Keuls test showed the following order: GT55 > U > GT73 > GU55 > GU73 > G. For the light-curing mode, two-way ANOVA showed no significant difference (S = G,  $P = 0.3437$ ).

The  $d_{k \text{ before}}$  values are shown in Fig. 3. The means of binary mixtures of GT55 and GT73 light-cured using two modes were significantly higher than those for the other matrices ( $P < 0.05$ ). The plasticization results are displayed in Table 3. Two-way ANOVA showed significant differences for the two independent factors ( $P < 0.05$ ). Specimens light-cured with G mode showed higher plasticization than those light-cured with S mode ( $P < 0.05$ ). With regard to the polymeric matrix, the Student-Newman-Keuls test gave the following order: GT73 = GT55 > G = GU73 = GU55 = U.

Linear regression analyses (Fig. 4) showed no correlation between DC% and plasticization ( $r = 0.32 / P = 82.35 \pm 0.85DC$ ).

## Discussion

Most previous studies that have analyzed the degree of conversion of dimethacrylate-based materials have used the aromatic C=C bond peak (1608 cm<sup>-1</sup>) as an internal standard to calculate the degree of conversion (3,25,26). In the present study, however, the composition of U polymeric matrix was 100wt% UDMA, which has no aromatic rings in its molecule. Consequently, the carbonyl group peak (1,720 cm<sup>-1</sup>) was chosen as an internal standard to calculate the DC of all the polymeric matrices analyzed. This choice is supported by previous studies (27,28).

The radiant exposures generated by the two light-curing modes in the present study ( $\cong 16 \text{ J/cm}^2$  and  $17 \text{ J/cm}^2$ ) were based on the findings of Emami and Söderholm (26), who showed that small radiant exposures ranging from 5 to 15 J/cm<sup>2</sup> were sufficient to produce adequate DC% at the top and bottom surfaces of 2-mm-thick specimens of Z250 resin composite. The absence of a significant difference between the DC% produced by the S and G light-curing modes was expected, and proved the first of our study hypotheses. This finding was attributed to the slight difference between the radiant exposures generated by the two modes (S = 17 J/cm<sup>2</sup> and G  $\cong 16 \text{ J/cm}^2$ ), and is supported by the study of Rahiots et al. (9), who also found no differences between the DC%

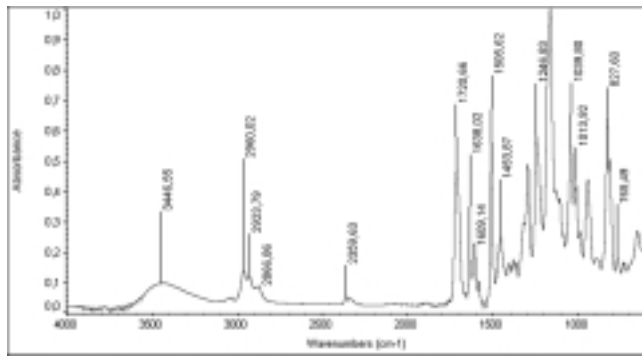


Fig. 1 FT-IR spectra recorded for G polymeric matrix. Several absorbance peaks can be seen between 2,000 cm<sup>-1</sup> and 500 cm<sup>-1</sup>.

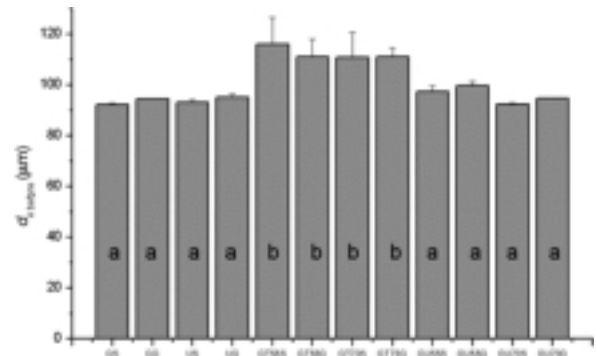


Fig. 3 Mean values of  $d_{k \text{ before}}$ . Columns with the same letters do not differ significantly ( $\alpha = 0.05$ )

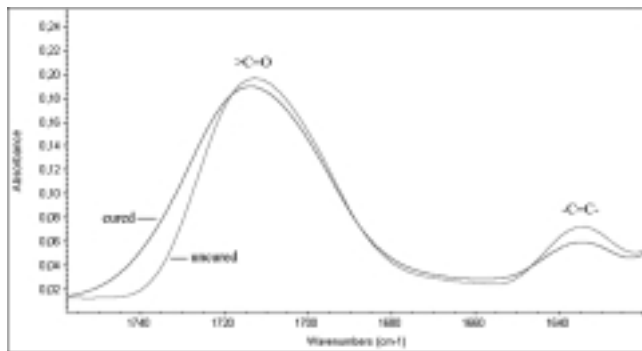


Fig. 2 FT-IR spectra of the cured and uncured specimens of the G polymeric matrix showing the absorbance peaks of the aliphatic C=C bond (1,638 cm<sup>-1</sup>) and carbonyl group (1,720 cm<sup>-1</sup>) used to calculate the DC%.

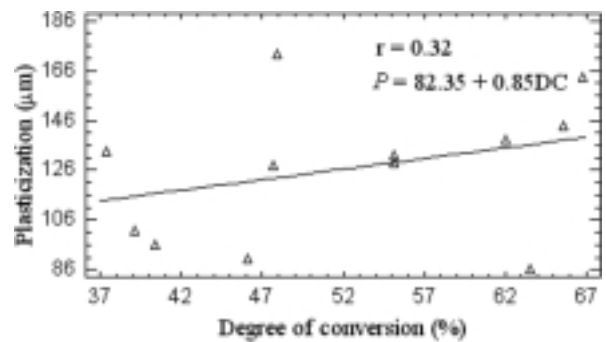


Fig. 4 Regression line of plasticization (P) plotted against the degree of conversion (DC).

Table 2 Means (%) and standard deviation ( $\pm$ SD) of degree of conversion for all experimental groups subject to two light-curing modes

Polymeric matrix	Light-curing mode	
	Standard	Gradual
G	<sup>Aa</sup> 39.2 $\pm$ 1.8	<sup>Aa</sup> 37.3 $\pm$ 4.7
U	<sup>Ab</sup> 63.5 $\pm$ 3.6	<sup>Bb</sup> 55.2 $\pm$ 3.6
GT55	<sup>Ab</sup> 65.6 $\pm$ 4.8	<sup>Ac</sup> 66.8 $\pm$ 1.7
GT73	<sup>Ab</sup> 62.0 $\pm$ 1.9	<sup>Bb</sup> 48.0 $\pm$ 2.0
GU55	<sup>Aa</sup> 46.1 $\pm$ 3.9	<sup>Bb</sup> 55.2 $\pm$ 4.5
GU73	<sup>Aa</sup> 40.4 $\pm$ 2.5	<sup>Ab</sup> 47.7 $\pm$ 3.6

Means with the same capital letter are not statistically different within the same line. Means with the same lower case letter are not statistically different within the same column ( $P < 0.05$ ).

Table 3 Means (µm) and standard deviation ( $\pm$ SD) of Knoop diamond indentation depth used to estimate the plasticization for all experimental groups subject to two light-curing modes

Polymeric matrix	Light-curing mode	
	Standard	Gradual
G	<sup>Aa</sup> 101.9 $\pm$ 4.8	<sup>Ba</sup> 133.9 $\pm$ 7.4
U	<sup>Aa</sup> 86.8 $\pm$ 10.6	<sup>Ba</sup> 129.4 $\pm$ 5.5
GT55	<sup>Ab</sup> 144.3 $\pm$ 23.5	<sup>Bb</sup> 163.9 $\pm$ 30.1
GT73	<sup>Ab</sup> 138.4 $\pm$ 9.8	<sup>Bb</sup> 173.2 $\pm$ 20.9
GU55	<sup>Aa</sup> 90.9 $\pm$ 2.8	<sup>Ba</sup> 132.2 $\pm$ 16.0
GU73	<sup>Aa</sup> 96.6 $\pm$ 2.2	<sup>Ba</sup> 128.4 $\pm$ 0.9

Means with the same capital letter are not statistically different within the same line. Means with the same lower case letter are not statistically different within the same column ( $P < 0.05$ ).



produced by an exponential (Gradual) and a continuous (Standard) light-curing mode with a minor difference between their radiant exposures. From a clinical viewpoint, this finding is of crucial importance. For more than 10 years, gradual light-curing modes have been used to diminish the harmful effects of shrinkage stress generated during the resin composite polymerization reaction (12). Thus, if these light-curing modes do not affect the degree of conversion, they could be considered more adequate for light-curing of resin composites. This was the basis of the first hypothesis of the present study.

The G homopolymer had the lowest DC% value. This may be explained by the high Bis-GMA viscosity, derived from the intermolecular hydrogen bonding formed by the hydroxyl groups, and the presence of the isopropylidene-diphenoxy ring structure that stiffens the Bis-GMA molecule (2,29). These features drastically reduce the mobility of Bis-GMA, consequently decreasing the polymeric network flexibility (3), and thus the DC% of the G homopolymer.

Dickens et al. (30) recently showed that the viscosity of Bis-GMA, UDMA, and TEGDMA was 1369, 28, and 0.05 Pa.s, respectively. These authors considered that in dimethacrylate resins with initially low viscosity, the onset of diffusion limitation and autoacceleration are delayed during the polymerization reaction, resulting in a high degree of conversion. These findings appear to support the DC% data for binary mixtures, i.e., GT73/GT55 and GU73/GU55, observed in the present study. From Table 2, it can be seen that the increase in the TEGDMA or UDMA content of binary mixtures tended to parallel an increase in the DC%. It is obvious that this behavior resulted from the decrease in the viscosities of the matrices with an increased TEGDMA or UDMA content. Reinforcing the role of monomer viscosity in the DC% of dimethacrylate-based matrices, Chung et al. (31) showed that an increase of TEGDMA from 0% to 40% increased the DC% of a TTEMA-TEGDMA binary mixture from 17% to 48%, i.e. TTEMA, tris[4-(2'-hydroxy-3'-methacryloyloxypropoxy)phenyl]methane, is a high-molecular-weight monomer possessing an aromatic ring like that of Bis-GMA.

In addition to the role of viscosity, the second significantly higher DC% presented by U homopolymer could also be explained by an increase in reactivity of UDMA, derived from some alternative polymerization pathway. As reported previously (3), the chain transfer reactions produced by the -NH- groups with UDMA polymer increased the mobility of radical sites in the network, offering an alternative path for the polymerization reaction to continue until the glassy stage was reached.

In this study, plasticization was determined on the basis of the softening effect of ethanol. When a polymer is immersed in a suitable solvent (i.e., with similar solubility parameters), the solvent enters the polymer network through the intermolecular spaces, forms secondary bonds with the polymer chain, pulls apart the polymer entanglements and reduces the interchain interactions (14). The reduction in hardness is a consequence of polymer plasticization (15). In addition, several published studies have shown that storage in solutions of 75-100% ethanol for 24 h causes softening of resin composites and dimethacrylate polymeric matrices (13,14,18,23,25). On this basis, it was expected that all matrices analyzed in the present study would undergo plasticization.

The G light-curing mode led to a high degree of plasticization, and was significantly different from the S mode. Thus, the second hypothesis of this study was supported. Indeed, the high degree of dimethacrylate monomer softening by ethanol after gradual (two-step)-light curing has been reported previously. According to Asmussen and Peutzfeldt (13), gradual curing may create few centers of polymer growth, resulting in a more linear polymer with low crosslink density. In a low cross-linked polymer, the polymer-solvent interaction overcomes the polymer chain primary bonds, increasing the hole-free volume between polymer chains. Thus, the uptake of solvent will increase and so will the plasticization (32). Based on this, it is reasonable to hypothesize that although there was no significant difference in DC% between S and G mode, the polymer structures formed were different from each other.

Although Bis-GMA/TEGDMA binary mixtures presented the highest DC%, they also showed the highest plasticization (Tables 2 and 3). Allied to the inherent heterogeneity shown by networks derived from the free radical polymerization of dimethacrylate monomers, TEGDMA, because of its high flexibility, leads to a marked degree of pendant double bond primary cyclization (33). This feature may contribute to the formation of a more heterogeneous network, which would increase the diffusion of ethanol in the nanopores of the network, thus increasing the degree of plasticization. Published studies (28,34); have indeed shown that the greater the content of TEGDMA, the higher the water and salivary sorption of Bis-GMA/TEGDMA binary mixtures. From this viewpoint, it is reasonable to assume that, in this study, the GT55 and GT73 polymeric matrices adsorbed more ethanol than the others.

The lack of correlation between the DC and P (Fig. 4) proved the third hypothesis of the present study. This finding disagrees with the results of previous studies

(28,34), which found a correlation between the degree of conversion of dimethacrylate-based polymers and their softening by ethanol. However, it is important to clarify that in these studies the authors used only different proportions of Bis-GMA/TEGDMA binary mixtures. From this, it is clear that not only the degree of conversion, but also the chemical structure of the polymeric matrix must be taken into account to predict the plasticization behavior of the material.

In summary, the results of this study show that although the degree of conversion is a good indicator for predicting the behavior of resin-based composites, other properties such as plasticization must also be considered for this purpose. In addition, it was confirmed that gradual light-curing modes may create a less cross-linked dimethacrylate-based polymeric matrix.

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