Original Article

An evaluation of elution of leachable components from composite resins after light curing by light emitting diode and halogen light: An *in vitro* study

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Abstract

Objectives: The purpose of our study was to determine the amount of eluted triethyleneglycol dimethacrylate (TEGDMA) and to compare the eluted TEGDMA in different composite resins after light curing with conventional halogen light curing unit and light emitting diode (LED).

Materials and Methods: The present study was conducted on the two types of composite resins, which were divided equally into four groups – Group I: Denoted as Hybrid-LED, Group II: Denoted as Microhybrid-LED, Group III: Denoted as Hybrid-Halogen Group IV: Denoted as Microhybrid-Halogen. Polymerized specimens of hybrid and microhybrid composite resins were stored in air tight centrifuge tubes at 37°C for 24 h, then extract the monomers in high-performance liquid chromatography (HPLC) grade acetonitrile and water and incubated at 37°C for 24 h. All extracts were analyzed by HPLC. Eluted TEGDMA was detected by ultraviolet detector. The results obtained for TEGDMA were computed and analyzed using the one-way ANOVA and independent samples *F*-test at significance level 0.05.

Results and Conclusions: Elution of TEGDMA from all the samples of Group III (Hybrid-Halogen) was greatest and from Group II (Microhybrid-LED) was lowest. The sequence of TEGDMA elution was Group III > Group I > Group IV > Group II. From our results, we can conclude that the LED light curing unit may be more efficient than standard halogen light curing unit. The extractable quantities of composite resin components should be minimized. Furthermore, all ingredients of a dental composite should be declared by the manufacturers, in order to identify those substances in a product which may cause adverse side effects in patients and dental personnel.

Keywords: Composite resins; elution; high performance liquid chromatography; residual monomers; triethyleneglycol dimethacrylate

INTRODUCTION

As Dr. Ronald E. Goldstein states "Esthetic Dentistry is the art of dentistry in its purest form." The search for an ideal esthetic material for restoring teeth has resulted in significant improvement in both esthetic material and technique for using them. In the late 1960s, composite resins were introduced as an alternative to silicates and unfilled resins. Light cured resin-based composites are cured through a process called polymerization which is the conversion of the resin monomers into a polymer network. An important determinant of the clinical success of composite restorations is to conversion of all its monomer to polymer during the polymerization reaction. However, monomer conversion is never complete, and the degree of conversion varies between approximately 35% and 77% (Ferracane *et al*., 1994, 1995).[1-3] According to Chung and Greener, 1990; Knezević A, (2001), DC ranges from 43.5% to 73.8% when standard curing unit is used.[4,5]

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Therefore, a significant amount of residual monomer or short-chain polymer remains unbound in set dental composites and can be leached into aqueous media.[1] The effect on physical/mechanical properties and the results of toxicity testing of dental composites are variable with different immersion media. Solution of 75% ethanol/water and acetonitrile is recommended by the US Food and Drug Administration (FDA) as a food/oral simulating liquid.[6] Ethanol/water and acetonitrile both are organic solvent and, in this study, we selected Acetonitrile as immersion medium.

Among the main two monomers BisGMA and triethyleneglycol dimethacrylate (TEGDMA), the amount of leachable BisGMA has already been established in different studies.

According to Nalçaci *et al*., the amount of eluted BisGMA was in the range of 3.42–5.15 ppm after 24 h.[7] They conducted their study in reversed-phase high-performance liquid chromatography (HPLC) instrument and used methanol as solvent. Yap *et al*. also evaluate the elution of BisGMA by HPLC in 2004.[1] They used acetonitrile (also used in the present study) as the extraction medium. They found that average 9.59–12.74 ppm of BisGMA was eluted after 24 h. In 2008, Zhang and Yu evaluate the residual BisGMA after immersion the composite samples in various media and they established that after 24 h 2.1 ppm BisGMA in distilled water, 1.8 ppm BisGMA in artificial saliva and 8.13 ppm BisGMA in ethanol/water was eluted.[6]

From 40% to 50% of the monomers of many modern resin composites and other resinous materials are made up of the relatively hydrophilic TEGDMA (Ferracane *et al*., 1995)[3]. TEGDMA might easily penetrate dentin, causing pulpal irritation (Gerzina TM, 1996) and can diffuse rapidly and in high quantities through dentinal tubules, even in the presence of a positive pulpal pressure.[8] According to many studies, due to the low molecular weight of TEGDMA, it was eluted in the highest amount. Hence, keeping in mind, the toxicity level of TEGDMA further studies is needed to establish the amount of eluted TEGDMA. Knezević A (2001), Chung KH (1990), Eliades GC, (1987) did the qualitative analysis of TEGDMA in different composite resins after light curing.[4,5,9] However, qualitative analysis is not so clinically significant as quantitative analysis of TEGDMA. Studies related to quantitative analysis of TEGDMA are very limited. In almost all studies, it is found that elution and toxicity of TEGDMA are much more than high-molecular-weight BisGMA. Hence, the present study has been undertaken to evaluate the elution of TEGDMA in a BisGMA/TEGDMA containing composite resins after light curing by both light-emitting diode (LED) and Halogen light curing unit by reversed-phase HPLC.

MATERIALS AND METHODS

Sample preparation

Four groups of sample were prepared:

- Group I: Hybrid-LED
- Group II: Microhybrid-LED
- Group III: Hybrid-Halogen
- Group IV: Microhybrid-Halogen.

The Hybrid-A2 shade (Spectrum–Dentsply) and Microhybrid-A2 shade (Esthet X– Dentsply) composite material were placed in customized stainless steel molds with cylindrical recesses 6.5 mm in diameter and 1 mm in height by the help of plastic filling instrument. They were sandwiched between two glass slides to ensure smooth surfaces, minimize the inhibition of polymerization by oxygen and extrude excess material through the application of pressure. A constant uniform pressure was maintained to achieve 1 mm thickness of sample. The top slide was removed and the material was cured from the top surface keeping the curing tip at a constant distance of 1 mm from the surface of composite sample using either a standard halogen light (Heraeus Kulzer) or a standard LED curing unit (Heraeus Kulzer) - both for 40 s.

All samples were prepared in a temperature-controlled room (23°C). A curing radiometer was used to measure the intensity of the standard halogen curing unit before each application in Group III and Group IV. In the case of Group I and Group II, the light intensity stated by the standard LED unit manufacturer was accepted as being accurate. The LED unit's batteries were recharged according to the manufacturer's recommendations and the units were replaced in their chargers following polymerization of each sample.

Immediately after light polymerization, the specimens were removed from the customized stainless steel molds and sized with sandpaper and were placed in centrifuge tubes. The centrifuge tube openings were covered with aluminum foil and tied with elastics to prevent contamination of moisture. Then the tubes were stored at 37° C for 24 h.

Release of dental monomers

Five milliliters of HPLC-grade acetonitrile were added to each centrifuge tube and the mouth opening of each tube was tightened with rubber cork to prevent the evaporation of volatile material. The specimens were then incubated in incubator (Orbitek), at 37°C for 24 h.

The incubation solutions were then centrifuged in a centrifuge machine (Eltek TC 4100 D) at 15,000 × g for 10 min. The solutions were filtered through filter paper into a sample vial. To prevent the evaporation of monomers, the solutions were filtered within 2 min in a temperature-controlled room (15°C) in the closed glass chamber.

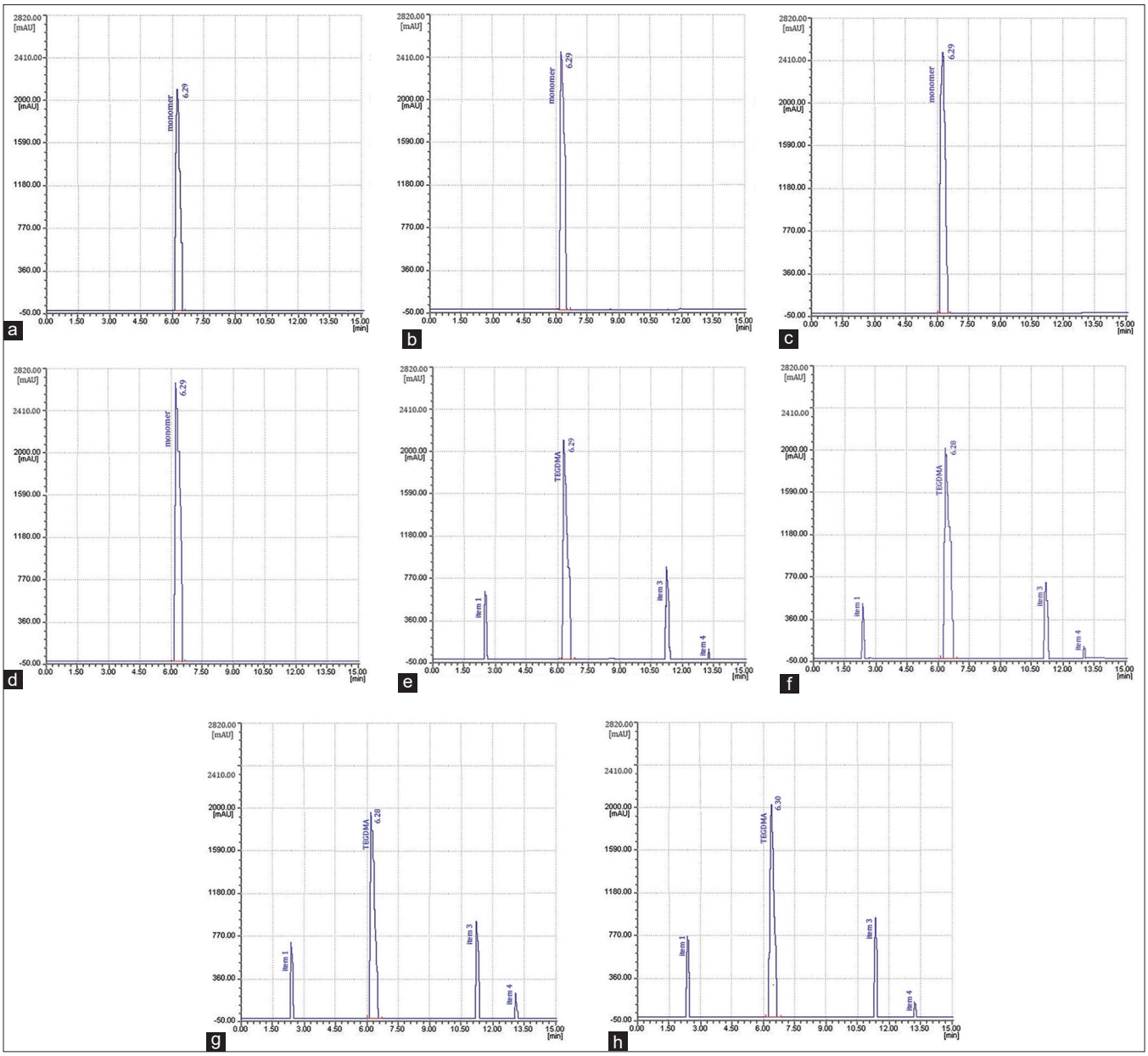

Figure 1: (a) High-performance liquid chromatography (HPLC) graph of 8 ppm concentration of standard triethyleneglycol dimethacrylate (b) HPLC graph of 10 ppm concentration of standard triethyleneglycol dimethacrylate (c) HPLC graph of 12 ppm concentration of standard triethyleneglycol dimethacrylate (d) HPLC graph of 20 ppm concentration of standard triethyleneglycol dimethacrylate (e) HPLC graph of Group I, number 1 sample (f) HPLC graph of Group II, number 1 sample (g) HPLC graph of Group III, number 1 sample (h) HPLC graph of Group IV, number 1 sample

Twenty microliters of each solution were injected at the room temperature each time into a reversed phase HPLC, (Cyberlab) instrument with a Symmetry Columns (C18, 5.0 μm, 4.6 mm × 250 mm).

In this study, HPLC – grade acetonitrile and HPLC – grade water were used as the mobile phase and ultraviolet (UV) detector (Orbitek) at 205 nm was used to detect the retention times (RTs) of the sample solutions as well as standard solutions.

Operation

The sample run was taken in a gradient mode in this study. Samples were eluted at a flow rate of 1.0 ml/min for the first 5 min using a solvent linear gradient of 50% acetonitrile in water to 100% acetonitrile, and then eluted at the same flow rate for 10 min with 100% acetonitrile. The concentration of acetonitrile was then gradually decreased for more than 5 min to 50% acetonitrile in water at a flow rate of 1.0 ml/min. The same conditions were held for the next 10 min to wash the column. The eluted TEGDMA

was detected by a UV detector (Cyberlab) at 205 nm. RT, peak areas, and UV absorbance of TEGDMA of each sample solutions were obtained from the resulting chromatogram [Figure 1e-h and Table 1].

Standard solution preparation

Standard concentrations of 8, 10, 12, and 20 ppm of TEGDMA were prepared. The standards were then injected into the reversed-phased HPLC instrument using the aforementioned conditions. RT, peak areas, and UV absorbance of each concentration of TEGDMA were obtained from the resulting chromatogram [Figure 1a-d and Table 1].

Calibration plots were obtained from the peak areas and concentrations of standard TEGDMA. The amount (concentration) of TEGDMA eluted from each sample solutions was subsequently quantified based on the peak areas against standard curves obtained for standard TEGDMA.

Statistical analysis

The results obtained for TEGDMA were computed and analyzed using the one-way ANOVA and independent samples *F*-test at the significance level of 0.05.

RESULTS

Procedure outline

From the resulting chromatogram, we got milliabsorbance unit (mAU) of the analyte (along the y-axis in the graph), RT (along the x-axis), and peak area (Half width × height) representing the amount of analyte expressed in graph. 1 mAU = 410 unit height in the graph. The RT obtained from the graph was fixed for standard TEGDMA and it is 6.29 min as per graph [Figure 1a-d].

From the data based on peak area and concentration, we get regression equation [y = a + bx; where y stands for concentration, x stands for area, a and b are constant].

The standard curve was obtained based on Area and Concentration of Standard TEGDMA [Figure 2a and Table 2].

TEGDMA peak was identified from each chromatogram of each sample solutions by the RT (6.29 min), which is constant [Figure 1e-h].

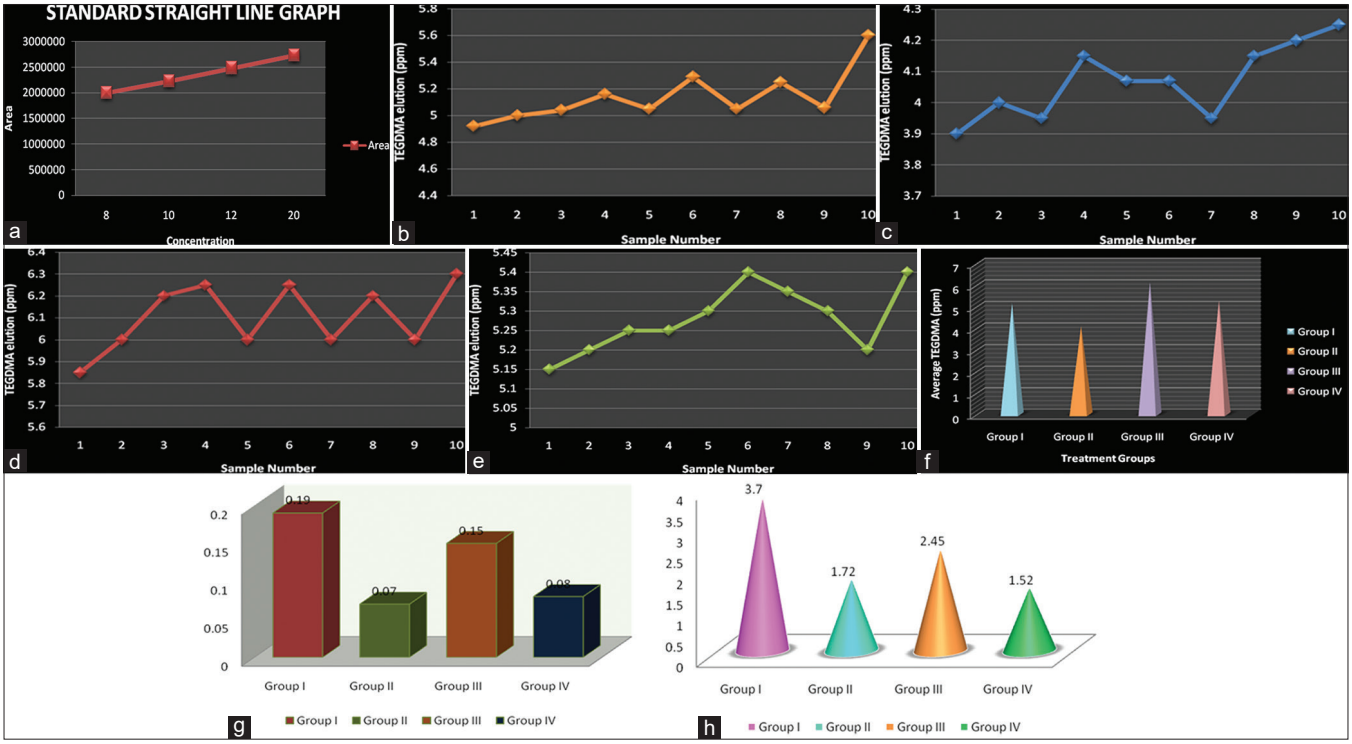

Figure 2: (a): Standard curve of peak area versus monomer concentration of standard monomer (Triethyleneglycol dimethacrylate [TEGDMA]) (Regression equation: y = 1660597 + 56176x), (b) Graphical representation of the individual TEGDMA elution of Group I (Hybrid-light emitting diode [LED]), (c) Graphical representation of the individual TEGDMA elution of Group II (Microhybrid-LED), (d) Graphical representation of the individual TEGDMA elution of Group III (Hybrid-Halogen), (e) Graphical representation of the individual TEGDMA elution of Group IV (Microhybrid-Halogen), (f) Average TEGDMA eluted in four groups of sample, (g) Average of standard deviation (SD) values of four Treatment groups, (h) Percentage of coefficient variation (CV) of four treatment groups

Now with the help of regression equation [y = a + bx], based on standard curve, we get the concentration (in ppm) of eluted TEGDMA of each composite sample.

The results for TEGDMA (of composite samples) were computed and analyzed using the one-way ANOVA and independent samples *F*-test at significance level 0.05.

The following formulas have been obtained from the data on area and concentration of standard TEGDMA:

Regression equation: y = 1660597 + 56176 × [y = a + bx]

Where "x" stands for concentration

And 'y' stands for area (in the present study)

Regression coefficient "a" (constant) =1,660,597

Regression coefficient "b" (constant) =56,176

Correlation coefficient "r" (interdependence between "x" and "y") =0.93562

As the correlation coefficient ("r" value) here is >0.5, the two variables namely concentration level and area are directly proportional. Hence, for the unit increase of concentration level (x), the area increases as given by the Regression equation.

Average concentration x = 12.5

Average area y = 2362797.75

Estimated values (calculated approximate value) of y (area) based on x (concentration) following regression equation of "y" and "x" viz. ŷ (estimated area) =1660597 + 56176x

The amount (concentration) of TEGDMA eluted from each sample solutions is quantified based on peak areas against the standard curves obtained for standard TEGDMA (Regression equation: y = 1660597 + 56176x).

The graphical representations of the same data are shown individually in Figure 2b-e for the Groups I, II, III, and IV, respectively. The same observations for the respective groups have been subjected to the statistical analysis. For each group, the average TEGDMA elution, standard deviation (SD), coefficient variation, and range of TEGDMA elution were calculated. To evaluate and compare the groups among themselves for statistical significance, the average difference between the TEGDMA elution was subjected to student *F*-test [Table 4].

Critical difference (CD) for comparison among the mean values is obtained by using the formula:

Table 1: Retention time (RT), Half width, Height & Area of Eluted TEGDMA of Standard solutions & Sample solutions obtained from HPLC graph

Name	Concentration (ppm)	RT (min)	Half width	Height	Area
a. High-performance liquid chromatography 8 ppm concentration of standard triethyleneglycol dimethacrylate					
TEGDMA	8	6.29	9.29	215,464	2,001,657
b. High-performance liquid chromatography 10 ppm concentration of standard triethyleneglycol dimethacrylate					
TEGDMA	10	6.29	9.07	246,048	2,231,654
c. High-performance liquid chromatography 12 ppm concentration of standard triethyleneglycol dimethacrylate					
TEGDMA	12	6.29	10.02	248,065	2,485,612
d. High-performance liquid chromatography 20 ppm concentration of standard triethyleneglycol dimethacrylate					
TEGDMA	20	6.29	9.97	274,049	2,732,268
e. High-performance liquid chromatography Group I, number 1 sample					
TEGDMA		6.29	9.02	214,743	1,936,983
f. High-performance liquid chromatography Group II, number 1 sample					
TEGDMA		6.28	9.16	205,206	1,879,683
g. High-performance liquid chromatography Group III, number 1 sample					
TEGDMA		6.28	9.89	201,124	1,989,114
h. High-performance liquid chromatography Group IV, number 1 sample					
TEGDMA		6.30	9.55	204,178	1,949,903

TEGDMA: Triethyleneglycol dimethacrylate, RT: Retention time

Table 2: Standard concentration, area and estimated area of standard monomer (triethyleneglycol dimethacrylate)

Concentration (X) (ppm)	Area (Y) (obtained in the present study)	Area (estimated) (Ŷ) (as per regression equation)
8	2,001,657	2,110,005
10	2,231,654	2,222,357
12	2,485,612	2,334,709
20	2,732,268	2,784,117

TEGDMA: Triethyleneglycol dimethacrylate

$$CD = t_{36} \times \sqrt{\frac{2MSE}{10}}$$

= 0.12, 0.16, 0.21

At 5%, 1%, and 0.1% level of significance [Results depicted in Table 5].

DISCUSSION

The present study was conducted on two types of composite material Hybrid (Spectrum – Dentsply) and Microhybrid (Esthet X – Dentsply), both of A_2 shade and two types of light curing units LED – 600 mW/cm^2 (Heraeus Kulzer) and Halogen – 750 mW/cm^2 (Heraeus Kulzer). Each type of composite sample was of 6.5 mm in diameter and 1 mm in height and each sample were light cured for 40 s either by LED or Halogen light

keeping the curing tip at a constant distance of 1 mm from the surface of composite sample.

The elution of TEGDMA was evaluated by the help of C18 reversed phase HPLC instrument. Although there are many other studies to evaluate elution of leachable components or residual monomers from composite resins, studies with the help of HPLC are very limited.

The analytical method HPLC used in our investigation is able to detect monomers that leach out of cured composite resins within the limits of the detection system. HPLC is an alternative and more sensitive method than other methods like, gas chromatography/mass spectrometry. It is a very powerful and commonly used separation method and preferred to gas chromatography, because it gives a greater level of control over the separation process.

Pelka *et al*.; Munksgaard *et al*.; Yap *et al*.; Nalçaci *et al*.; Moharamzadeh *et al*.; Zhang and Xu.[1,6,7,10-12] In all these studies, HPLC was used for elution of leachable components of composite resins.

It could be shown that different solvents and different curing methods lead to different results concerning the amount of leached nonreacted monomers. In accordance with Ferracane JL (1995)[3], the amount of remaining monomers/initiators eluted from light curing dental filling materials *in vitro* is dependent on the elution medium and ranges between 0.5%–2% weight in water and 2%–6% weight in 70% ethanol. In an aqueous extraction medium, the quantitative and qualitative analysis of substances is more difficult. Using organic solvents such as ethanol or methanol, there is a significantly improved elution and a distinct increase in the amount of each substance detected.

Several factors contribute to the process of elution from dental composites, such as size and chemical composition of the leachable substance and chemistry of the solvent.[3] The rate and extent of elution have been reported to be greater in an organic solvent when compared to elution into pure water.[2,3,13,14] Solution of 75% ethanol/water and acetonitrile are recommended by US FDA as a food/oral simulating liquid.[6] For this reason, in order to measure the elution of TEGDMA, organic solvents are preferred.

Pelka *et al*. evaluated the elution parameters and HPLC detection of single components from resin composite. They used water: Acetonitrile (50:50 vol/vol) as the extraction medium of composite resins.[10] Moharamzadeh *et al*. have done HPLC analysis of components released from dental composites with different resin compositions using different extraction media. They used water: Acetonitrile (30:70 vol/vol) as the reference extraction media.[11] Acetonitrile, which has been used in other HPLC analysis of dental composites (Yap AU 2004),[1] was selected as the solvent and mobile phase for the present study, as TEGDMA is hydrophilic and the results of our pilot study showed that monomer and acetonitrile solvent peaks were well separated.

Using a C18 reverse-phase column, the RTs (the time between sample injection and an analyte peak reaching a detector at the end of the column in HPLC) depended only on the polarity. Molecules with a high polarity demonstrate with HPLC shorter RTs than apolar molecules. Pelka *et al*. in 1999 and Nalçaci *et al*. in 2006 have used C18 reversed phase HPLC instrument for time-based elution of TEGDMA from resin composite.[7,10] In the present study we have also used reversed phase HPLC (Cyberlab) instrument with a Symmetry Columns (C18, 5.0 μm, 4.6 mm × 250 mm) for elution of TEGDMA. RT increases with hydrophobic-nonpolar - surface area. TEGDMA is hydrophilic and so its retention should be less. In this investigation, a RT of about 6.29 min for TEGDMA was exhibited.

As per several authors[1,12] to ensure uniform and maximum polymerization, in our present study, we have used 1 mm thick composite samples. Moreover, to minimize the effect of colorants on light penetration, we have selected A_2 shade as per the study of Nalçaci *et al*. in 2006 and Bayne *et al*. in 1994 for all the samples.[7,15] (In the present study, a standard LED light and a standard halogen light with an irradiation time of 40 s [as per manufacturer's recommended cure time] were used).

The data were obtained in the present study as [Tables 3 and Figure 1 f-h]:

Average TEGDMA elution in Group I samples was 5.14 ppm, SD of 0.19, and the Coefficient of variation (CV %) of 3.70.

In Group II, average value of TEGDMA elution of about 4.07 ppm, SD of 0.07, and the critical value (CV %) of 1.72.

Group III gave the average value of 6.12 ppm, SD of 0.15, and the critical value (CV %) of 2.45.

In Group IV, average value of eluted TEGDMA obtained was 5.28 ppm, SD of 0.08, and the critical value (CV %) of 1.52.

This is depicted in Table 3. From these data, we can say that standard LED light is more efficient than Standard Halogen light as more amount of average TEGDMA was eluted from the hybrid as well as microhybrid composite samples (6.12 and 5.28 ppm, respectively) after light curing by Standard Halogen light, than Standard LED light (where average TEGDMA elution were 5.14 and 4.07 ppm, respectively from hybrid and microhybrid composite).

The results of our study can be correlated with the results obtained by Nalçaci *et al*., where they have evaluated

Table 3: Triethyleneglycol dimethacrylate elution (concentration in ppm) from the samples of treatment groups

Sample number	Group I		Group II		Group III		Group IV	
	Area (Y)	Concentration (X)	Area (Y)	Concentration (X)	Area (Y)	Concentration (X)	Area (Y)	Concentration (X)
1	1,936,983	4.92	1,879,683	3.90	1,989,114	5.85	1,949,903	5.15
2	1,941,477	5.00	1,885,301	4.00	1,997,484	6.00	1,952,712	5.20
3	1,943,724	5.04	1,882,492	3.95	2,008,888	6.20	1,955,521	5.25
4	1,950,465	5.16	1,893,727	4.15	2,011,679	6.25	1,955,577	5.25
5	1,944,286	5.05	1,889,233	4.07	1,997,653	6.00	1,958,329	5.30
6	1,957,768	5.29	1,889,345	4.07	2,011,804	6.25	1,963,947	5.40
7	1,944,342	5.05	1,882,548	3.95	1,997,658	6.00	1,961,138	5.35
8	1,955,521	5.25	1,893,783	4.15	2,009,450	6.20	1,958,891	5.30
9	1,944,848	5.06	1,896,536	4.20	1,997,661	6.00	1,953,274	5.20
10	1,975,183	5.60	1,899,289	4.25	2,014,337	6.30	1,964,510	5.40
Average	1,949,460	5.14	1,888,480	4.07	2,003,585	6.12	1,955,927	5.28
SD	11,030	0.19	6084	0.07	9454	0.15	1,957,380	0.08
CV		3.70		1.72		2.45	4859	1.52
Range		4.92–5.60		3.90–4.25		5.85–6.30		5.15–5.40

CV in percentage=SD/average ×100. From these values, it is evident that elution of TEGDMA from all the samples of Group III is the greatest and from Group II is the lowest. The sequence of TEGDMA elution is Group III > Group IV > Group I > Group II. SD: Standard deviation, CV: Coefficient of variation, TEGDMA: Triethyleneglycol dimethacrylate

Table 4: Analysis of variance of the data on triethyleneglycol dimethacrylate release

Source	DF	Sum of squares	Mean sum of square	*F*
Between groups	3	21.24275	7.08092	405.20
Within groups	36	0.62910	0.01748	-
Total	39	21.87185	-	

F value with 3, 36 DF is highly significant ($P<0.001$)

time-based elution of TEGDMA from resin composite with LED and QTH lights.[7] Mills *et al*. gave the similar findings of significantly deeper levels of polymerization in medium-shade hybrid and micro-filled composites cured using LED light when compared to QTH light. These authors also pointed out that the narrow emission peaks of blue LED units indicate that they are more effective than QTH light units.[16]

Our study agrees with the findings of Vandewalle *et al*. who investigated the effect of light dispersion of LED curing lights on resin composite polymerization and observed that the latest generation of LED curing lights provides degree of conversion ratios similar to or better than the halogen curing light.[17]

Bala *et al*., Moon *et al*., and Yap *et al*. also conducted their study in the same line and gave same results of higher degree of polymerization by LED curing unit than Halogen curing unit.[1,18,19]

Knezević *et al*. compared the degree of conversion and temperature rise during polymerization of composite resin samples with halogen and blue diodes. They found that the degree of conversion as well as rise of temperature for all the materials they used is higher in case of illumination with standard halogen light curing units, than with blue LEDs. The intensity of the tested halogen curing unit used was much higher (750 mW/cm^2) than that of the LEDs (150–400 mW/cm^2).[4] However, in our study, we use LED light curing unit of 600 mW/cm^2 intensity and halogen curing unit of 750 mW/cm^2 intensity and as per our result curing performance of LED was significantly better than halogen light. Our results vary from the results of above study may be because of great difference of curing intensity used by them.

Now, if we concentrate on the elution of TEGDMA from the two types of composite resins (hybrid and microhybrid composite) used in the present study, the results showed that the amount of average TEGDMA elution is as follows [Table 6].

Spectrum is a sub-micron hybrid composite and Esthet-X is a microfilled composite. The two composites are differ in their particle size distribution. Spectrum contains hybrid and sub-micron filler particles (particle size are <1 μm and 10–20 nm, respectively), where Esthet-X contains hybrid and microfilled particles (particle size are <1 μm and 0.04 μm, respectively).

Leachable components are released due to degradation or erosion over time, the leaching process being determined not only by the degradation process itself but also diffusivity through the material. Chemical degradation is caused by hydrolysis or enzymatic catalysis. Nonspecific esterases, human saliva derived esterase and pseudocholinesterase may catalyze the biodegradation of composite resins.[9,20]

From the result of our study, it was seen that the amount of elution of TEGDMA was more in Hybrid composite (spectrum) than Microhybrid composite (Esthet-X) using both LED and Halogen light curing agents. As spectrum contains much less filler particle size than Esthet-X, so there may be more degradation of spectrum over time may occurred than Esthet-X and so the amount of eluted TEGDMA was also more.

Table 5: Comparison of average triethyleneglycol dimethacrylate release in four groups

Between groups	Group II		Group III		Group IV	
	Difference	Significance	Difference	Significance	Difference	Significance
Group I	1.07	***	1.21	***	2.05	***
Group II			0.14	*	0.98	***
Group III					0.84	***

*Significance at 5% level ($P<0.05$), ***Significance at 0.1% level ($P<0.001$). Hence, Group I is significantly different from Groups II, III, and IV ($P<0.001$). Groups II and III are significantly different from Group IV ($P<0.001$). Moreover, Group II is 5% ($P<0.05$) significantly different from Group III. TEGDMA: Triethyleneglycol dimethacrylate

Table 6: Average TEGDMA elution (ppm) ±SD of the samples of treatment groups

	Average TEGDMA elution (ppm) ±SD	
	Hybrid composite (spectrum)	Microhybrid composite (esthet-X)
LED	5.14±0.19	4.07±0.07
Halogen	6.12±0.15	5.28±0.08

SD: Standard deviation, LED: Light-emitting diode, TEGDMA: Triethyleneglycol dimethacrylate

A study to substantiate and support our present study was undertaken by Bala *et al*.[18] where they found the same findings of higher degree of conversion, i.e., less leachable components in microhybrid composite (Esthet-X) than hybrid composite (Filtek Z 250) and also higher degree of conversion in the above mentioned composites after light curing by LED than Halogen.

According to Tanaka *et al*.,[21] 6.21 wt% of TEGDMA was eluted from set dental composite of 3 mm thickness after 30 s curing and 1.88 wt% was eluted after 50 s irradiation. They analyzed the eluted TEGDMA by gas-liquid chromatography. Spahl *et al*. also reported TEGDMA as a toxic substance and eluted at a range of 0.04–2.3 wt%.[18] However, the above authors did not use the most sensitive instrument i.e. HPLC for elution of TEGDMA which were used in our study.

Nalçaci *et al*.[7] also conducted a study in the same line using hybrid (Charisma) and Microfilled (Filtek) composite and evaluated the time based elution of TEGDMA from resin composite cured with LED and QTH lights. Two millimeter thick samples were polymerized from the top and bottom surfaces, then immersed in methanol. HPLC was used to measure the amount of monomers released from the samples. Data were analyzed using the two-way ANOVA and Duncan's tests with a significance level of 0.05. They concluded that under the condition of their study standard QTH curing appear to result in higher levels of TEGDMA elution from the cured surface layer of resin composites than from standard LED curing. Our results [Table 3] agreed with the above findings. They found that after immersion of the composite samples for 24 h in methanol the amount of eluted mean TEGDMA from the Hybrid composite samples was in a range of 4.07 ± 0.07 by Halogen LCU and 4.20 ± 0.08 by LED LCU. However, the range of average TEGDMA elution in our present study [Table 3] was differ from Nalcaci *et al*.[7] and this may because of either the difference of thickness of composite samples used (In our study we have used 1 mm thick composite sample to achieve maximum polymerization), or because of difference in extraction media (in our study, the extraction media was Acetonitrile) or because of difference in manufacturing the hybrid composite (in our study, hybrid composite of Spectrum-Dentsply was used).

Therefore, as this was an *in vitro* study, to simulate the clinical conditions and for better assessment of elution of TEGDMA from composite resins after light curing, further clinical studies might be carried out in future.

The results of this *in vitro* study should be viewed as preliminary because of the limitations of a laboratory trial.

As in our study, limited variables were taken. Only two types of composite resins and two types of light curing devices were taken to conduct this *in vitro* study. And also among various leachable monomers from composite resins, we had evaluated only residual TEGDMA. Hence, further studies are required to evaluate the rates of elution of monomers over extended time periods from various resin composites polymerized using different curing methods.

SUMMARY AND CONCLUSION

From our results, we can conclude that monomer TEGDMA is released in detectable amounts and may be sufficient to cause an adverse reaction. The type of extraction media, type of composite resins, and the type of light curing units have a significant effect on the detection of monomer released from composite resins. The LED light curing unit may be more efficient than standard halogen light-curing unit.

The hydrolytic disintegration of TEGDMA to methacrylic acid, the toxicological aspects of methacrylic acid regarding repeated dose toxicity, *in vitro* genotoxicity, and carcinogenicity are seriously connected with a higher risk for local and possible systemic allergenic reactions. Systemic load and contact may occur when pure monomers are extensively distributed in the oral cavity or when resin matrix fillings are milled to replace them. The extractable quantities of composite resin components should be minimized. Hence, it should be the aim of future studies

to replace TEGDMA with more biocompatible diluents' monomers.

Therefore, as this was an *in vitro* study, to simulate the clinical conditions and for better assessment of elution of TEGDMA from various types of composite resins after light curing by different light sources, further clinical studies should be carried out in future.

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Conflicts of interest
There are no conflicts of interest.

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