Evaluation of Monomer Leaching from a Resin Cement Through Dentin

ABSTRACT

The aim of the study was to evaluate the elution of Triethylene glycol dimethacrylate (TEGDMA), Urethane dimethacrylate (UDMA), Bisphenol A glycerolate dimethacrylate (BisGMA), and Bisphenol A (BPA), from a dual -cured resin cement through human dentin, under constant positive pulpal pressure. Ten human dentin disks were adjusted into a custom made testing device and transparent glass slabs were luted with Variolink II cement, under a steady pressure. The device was filled with Ringer's solution and a pressure of 14.1 cm H_2O was applied. Eluates were retrieved from each one of the ten specimens at 9 time intervals. All the samples were analyzed by High Performance Liquid Chromatography (HPLC). TEGDMA was detected from the second and UDMA was detected from the fourth time interval. The highest average concentration of TEGDMA and UDMA was detected in the 3 day time interval. Time had a significant effect on their elution. BPA and BisGMA were not detected in any sample at any time interval. The clinical relevance of the present study is that the concentration of the eluted monomers, under the conditions that were chosen, did not reach toxic levels for the pulp.

INTRODUCTION

In vivo radioactive tracer experiments have demonstrated that radioactive compounds can move either direction across dentin: from the systemic circulation to dentin and vice versa,[1,2] so dentin is considered as "a permeable barrier".[3] The thousands of dentinal tubules that transverse the dentin allow ions, fluids, minute particles and bacteria to penetrate through dentin.[3,4] Dentin's permeability varies, even in different areas of the same tooth section and this phenomenon has been attributed to distinct differences in the structure of dentin, from area to area.[5] So, factors like the age of the tooth, the presence or the absence of the smear layer, the proximity of the dental material to the pulp, and also the diameter, the density and the length of the dentinal tubules have an effect on dentin's permeability. The area of dentin in close contact with a dental material is a significant factor that it should be considered in permeability studies and in reality, the area of dentin which is available for diffusion is defined by the diameter, the density and the length of the dentinal tubules.[3,4] On the other hand, some compounds penetrate through dentin more easily than others. The easiness of a compound to travel across dentin depends upon several factors like the molecular size, the charge, the hydrophobic or hydrophilic nature, the chemical affinity of the compound with the dentin, the diffusion coefficient and the applied concentration of the compound.[3,6] Over more, the outward movement of the dentinal tubule fluid has a competitive action against the passage of the compounds.[7,8] Reduced percentages of several compounds were detected when a positive pulpal pressure was applied.[9,10] Thus, the positive pulpal pressure is a factor that should be considered in *in vitro* permeability studies. In human teeth, the positive pulpal pressure has been estimated at about 14.1 cm H_2O.[11]

Keywords

Bisphenol A (BPA)
Bisphenol A Glycerolate Dimethacrylate (BisGMA)
Dentin Permeability
Triethylene Glycol Dimethacrylate (TEGDMA)
Urethane Dimethacrylate (UDMA)

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From this point of view, a dental material in contact with dentin may cause adverse effects, if it releases compounds in toxic concentration. In the oral environment, dental polymeric materials are found to elute compounds through two main mechanisms: The first mechanism concerns the elution of the unreacted monomers or the oligomers[12-16] but also the additives and the ions from the fillers.[17-19] The second mechanism concerns the degradation of the dental polymeric materials by hydrolysis and/or enzyme catalysis from the saliva and the enzymes in the oral environment. Degradation is a major problem, as it lasts for the entire life of the material and leads to the formation of several by-products.[19,20] Thereafter, the eluted compounds and the by-products of degradation can enter the human body by ingestion, by diffusion through the gastro dentin or by inhalation. As an example, Bisphenol A (BPA) is not a component of the dental resins but several researchers have detected BPA in the saliva of patients who were treated with several composites and sealants.[21-23] The elution of BPA was attributed to composite resin degradation in the oral environment or to impurities in the original monomer, which are left after its synthesis. Composite resins contain some compounds which are derivatives of BPA,[24] like Bisphenol A glycerolate dimethacrylate (BisGMA), Bisphenol A dimethacrylate (BisDMA), Bisphenol A ethoxylated dimethacrylate (BisEMA), etc. It has been shown that BisDMA is rapidly converted to BPA in whole saliva solutions[25] while the hydrolysis of BisGMA does not produce BPA.[26] On the other hand, the commercial BisGMA which is used for dental composite resin synthesis is found to contain traces of BPA.[27] Since the estrogenicity of BPA is well established,[28] BPA was included in the study.

Although an increase in the use of the resin cement materials is noticed nowadays, only a few studies examine the compounds that are eluted from resin cement materials and even fewer studies examine the trans-dentinal permeability of these compounds.[29-31] Tooth preparations for all-ceramic crowns require extensive preparation of the tooth structure. That leads to a decreased thickness of the remaining dentin and an increased area of contact, between the resin cement and the dentin. So, an "inflation" of the factor area of dentin available for diffusion is noticed when teeth are prepared for all-ceramic crowns. Over more, in the present study an *in vitro* model that attempts to simulate the positive pulpal pressure of natural teeth and the conditions of fixed prosthesis cementation is used.[31]

The aim of the present study is the detection of the monomers Bisphenol A (BPA), Triethylene glycol dimethacrylate (TEGDMA), Urethane dimethacrylate (UDMA), and Bisphenol A glycerolate dimethacrylate (BisGMA), from a dual cured resin cement (Variolink II) in the pulp chamber space. The hypothesis that was tested was: "The compounds BPA, TEGDMA, UDMA and BisGMA are able to leave the network of the resin cement, after the polymerization of the cement and pass through a dentin barrier in the "pulp chamber space".

MATERIALS AND METHODS

SPECIMEN PREPARATION

Ten healthy volunteers (18-30 years old) consented to donate their extracted third molars for the experimental purposes of this study. The Ethical Committee of the Dental School of the Aristotle University of Thessaloniki approved the study. All teeth (n=10) were caries and restoration free and after the extraction, were cleaned and stored in deionized water with 0.02% w/v thymol. From each tooth only one dentin disk (0.85±0.05 mm) was obtained under constant water coolant, just above the level of the pulp horns, by a low speed saw (ISOMET). The dentin disks were hand-sanded by a 600 grit silicon carbide paper under tap water and then, the disks were acid-etched on both sides with 35% phosphoric acid (Ultra etch) for 15 seconds and rinsed with water spray. Each dentin disk was attached, with a minimum quantity of aquarium marine silicone (Top sil) on the enamel margins of the dentin disk, on a custom made device[31] as shown in Figure 1. The area of the dentin of each disk was calculated by digital pictures (Canon EOS 600D and macro lens, Sigma 105 mm) with the use of a license free software program, Image J. A standard space (40 μm) for the resin cement was created on each specimen. Four stops from acrylic resin (Kallocryl) were constructed around the dentin disk on the glass Slab A. To simulate the positive pulpal pressure at 14.1 cm H_2O, the whole device was turned upside down and filled with Ringer's solution up to 14.1 cm height, counting from the internal surface of the disk. The total volume of Ringer's solution that was used for each specimen was 700 μL, including the three-way and the glass tube.

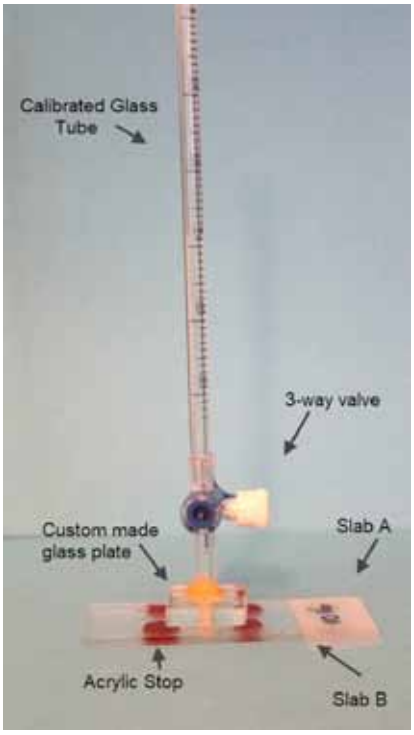

Figure 1: Illustration of the *in vitro* model

On the dentin disk of each specimen a transparent glass slab (Slab B) was cemented with the dual cured resin cement. In the present study, the multi component adhesive (Syntac) was used in conjunction with the low viscosity, transparent resin cement. Also, a one component primer which mediates a bond between the luting cements and the dental materials (Monobond Plus), was used on the glass slabs B prior to cementing. The composition is shown in Table 1.[32] Following precisely the instructions of the manufacturer,[32] a thin layer of the primer (Syntac Primer) was applied to dentin with a microbrush and after 15 seconds, air dried with the air syringe. Then, a thin layer of the adhesive (Syntac Adhesive) was applied to dentin for 10 seconds and then, it was air dried with the air syringe. Finally, a thin layer of the bonging agent (Heliobond) was applied and then immediately it was air dried. Equal amounts of catalyst and base of the resin cement were mixed on a mixing pad for 10 seconds and then they were applied to the dentin disk. A glass slab B, which was previously treated with a light film of the one component primer which mediates a bond between the luting cements and the dental materials for 60 seconds, was placed on the top with a light finger pressure at first. Then, a digital force gauge (Chatillon, DFE Series Digital force gauge) to apply a force of 25 N. The resin cement was cured for 40 seconds from the lateral side of the specimen. Then, the specimen was removed from the digital force gauge device and cured for another 40 seconds. The tip of the curing device was hold in contact with the transparent glass slab B. All specimens were stored in an oven at 37˚C. At 5 minutes, 20 minutes, 1 hour, 2 hours (early elution) and 21 hours, 3 days, 7 days, 10 days, and 21 days (late elution) time intervals, the whole eluent from each of the ten specimens was retrieved and sent for analysis and then, each specimen was immediately filled with fresh Ringer's solution.

CHROMATOGRAPHIC CONDITIONS

A method suitable for the chromatographic separation of the five compounds HEMA (Cas No 868-77-9), BPA (Cas No 80-05-7), TEGDMA (Cas No 109-16-0), UDMA (Cas No 72869-86-4) and BisGMA (Cas No 1565-94-2) was used, although the resin cement contain just the four of them.[31] In brief, the HPLC system was consisted of a photodiode array detector SPD-M6A set at 210 nm, a class-M10A computing integrator, an automatic Injector SIL-9A, a FCV-9A solvent mixing system and an LC9AD pump by Shimadzu. The chromatographic separation was achieved within 7.9 min under the following conditions: **Flow rate:** 1.5 mL/min. **Detection:** at UV 210 nm. **Column:** Perfectsil target ODS 3 (25 cm 4.6 mm id, 5 μM). **Mobile phase:** Gradient Elution: 45% CH_3CN/55% H_2O from 0 to 2 min and 88% CH_3CN/12% H_2O from 2 to 8 min, followed by 3min equilibration time. The retention time for HEMA was 3.2 min, for BPA 5.9 min, for TEGDMA 6.6 min, for UDMA 7.4 min and for BisGMA 7.9 min. A typical chromatogram of an 8 mg/L standard solution is shown in the Figure 2.

Table 1. Monobond Plus, Syntac Primer, Syntac Adhesive, Heliobond, Variolink II Base and Catalyst composition (in wt%).

Monobond Plus	
Ethanol	50-100%
Methacrylated phosphoric acid ester	1-<2.5%
Syntac primer	
Dimethacrylates:(Triethylene glycol dimeth., Polyethylene glycol dimeth.)	25%
Maleic acid	4%
Solvent: Acetone	71%
Stabilizer	< 0.1%
Syntac adhesive	
Dimethacrylates: Polyethylene glycol dimethacrylate	35%
Maleic acid	< 0.01%
Glutaraldehyde	5%
Water	60%
Heliobond	
Bisphenol A glycerolate dimethacrylate	59.5%
Triethylene glycol dimethacrylate	39.7%
Stabilizers and Catalysts	0.8%
Variolink II (Base)	
Ytterbium trifluoride	≤25%
Bisphenol A glycerolate dimethacrylate	10-<20%
Urethane dimethacrylate	2.5-<10%
Triethylene glycol dimethacrylate	2.5-<10%
Variolink II (Catalyst)	
Bisphenol A glycerolate dimethacrylate	50-100%
Ytterbium trifluoride	20-<25%
Urethane dimethacrylate	2.5-<10%
Triethylene glycol dimethacrylate	2.5-<10%
Dibenzoyl peroxide	0.1-<1%

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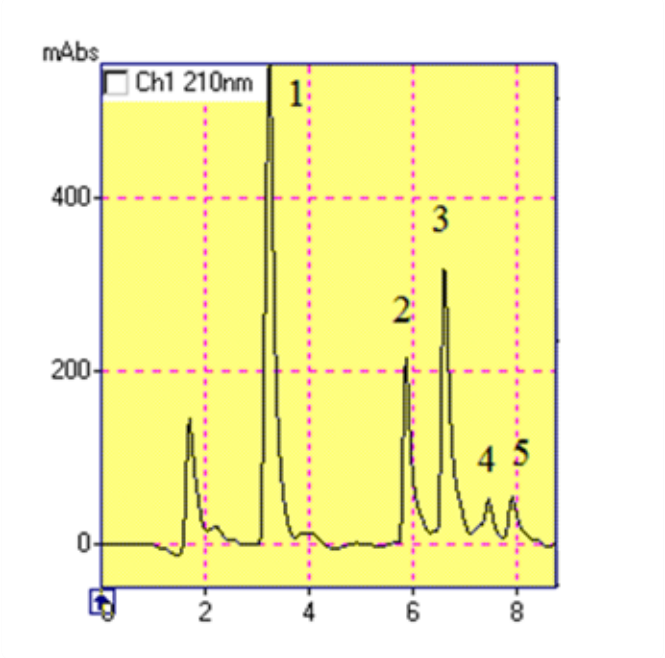

Figure 2: Typical chromatogram of 8 mg/L standard solution: (1) HEMA, (2) BPA, (3) TEGDMA, (4) UDMA, (5) BisGMA

STATISTICAL ANALYSIS

Data for the concentration of the TEGDMA and UDMA compounds was summarized by computing minimum and maximum values, means, standard errors (SE), and standard deviations (SD) per time interval. Then, the effect of time on the TEGDMA's and on the UDMA's concentration was tested by the Analysis of Variance (ANOVA) method with repeated measures (9 time intervals). First, the results of the multivariate approach to repeated measures analysis were evaluated (MANOVA). Following, Mauchly's test of sphericity was assessed. These results were used mainly for estimating the correct standard errors for testing the statistical significance of the differences between time intervals relative to mean values of TEGDMA and UDMA concentration. Greenhouse–Geisser adjustment to the degrees of freedom of the time effect and the corresponding error mean square was adopted, because the sphericity assumption did not hold. Mean concentration of TEGDMA between time intervals were compared with the Bonferroni criterion. The same criterion was used to compare the mean concentrations of UDMA between time intervals. Prior to MANOVA, the effect of the fluctuations of the dentin disk thickness and the resin cement amount was tested by the Analysis of Co Variance (ANCONA) method. In all hypothesis testing procedures the significance level was predetermined at $p \leq 0.05$. All statistical analyses were performed with the SPSS v.15.0 statistical software (SPSS Inc., Chicago, IL, USA).

RESULTS

Only TEGDMA and UDMA were detected from the examined compounds. BPA and BisGMA were not detected at any sample of any time interval. A representative chromatogram of the 7th day sample of a specimen is illustrated in Figure 3.

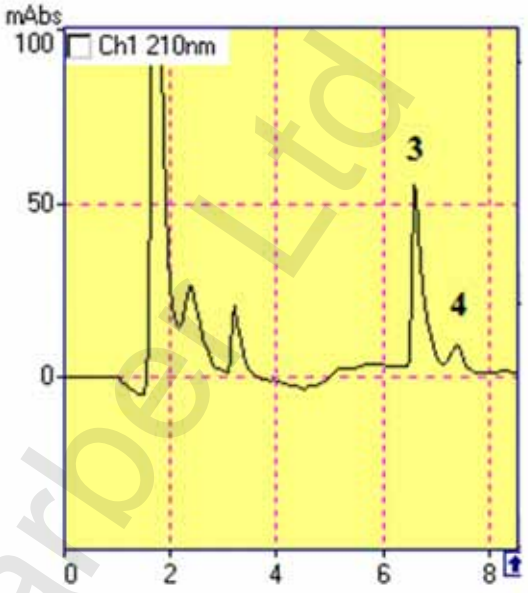

Figure 3: Chromatogram of a specimen at 7 d. (3) TEGDMA, (4) UDMA

TEGDMA was detected from the second-time interval (20 minutes) until the eighth-time interval (10 days), in all of specimen samples, besides two samples. At the last time interval (21 days), TEGDMA was detected, in very low concentrations, in only four samples. The results were different for UDMA. UDMA was detected from the fourth-time interval (2 hours) and then, until the eighth-time interval (10 days) in several specimen samples. At the last time interval (21 days), UDMA was detected in only three of the specimen samples.

Descriptive statistics for the dentin disk thickness and the resin cement amount are given in Table 2. ANCOVA method revealed that the fluctuation of the dentin disk thickness and the resin cement amount, did not affect the results of the study. (TEGDMA: p=0.115 and p=0.589 respectively. UDMA: p=0.064 and p=0.292 respectively.)

Table 2. Descriptive statistics for the dentin disk thickness and the resin cement amount

	N	Min	Max	Mean	SE	SD
Resin Cement Amount (mm^3)	10	2.31	3.90	2.63	0.06	0.19
Dentin Disk Thickness (mm)	10	0.76	0.91	0.85	0.02	0.05

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Time had a significant effect on TEGDMA's concentration according to the results of the ANOVA with the repeated measures method [$F(3.12, 28.13)=16.033$ $p<0.001$]. The highest mean concentration of TEGDMA was observed at the sixth-time interval (3 days) and this value statistically differs from the mean concentrations of 5 minutes, 20 minutes, 1 hour, 2 hours, 10 days and 21 days. Descriptive statistical indices for TEGDMA and the results of the *post hoc* analysis are given in Table 3. In the column "mean concentration" of the Table 3, the mean values that are followed by common letter(s) in parenthesis, are not statistically significant different, according to the Bonferroni criterion at p<0.05. The graphic representation of the mean concentration of TEGDMA in relation to time is shown in Table 5. The early elution of TEGDMA, from 20 minutes to 2 hours, seems to follow a linear descending trend which follows the equation:

$y = -0.1418x + 0.5239$ $R^2 = 0.9993$ (x=time, y= concentration)

The late elution of TEGDMA, from 21 hours to 21 days, follows the equation:

$y = -1.0256x^2 + 7.3296x - 12.15$ $R^2= 0.9585$ (x=time, y= concentration)

Also, time had a significant effect on UDMA's concentration according to the results of the ANOVA with the repeated measures method [F(3.2, 28.95)=18.115 p<0.001]. The highest mean concentration of UDMA was observed on the sixth-time interval (3 days) and this value statistically differs from the mean concentration of 5 minutes, 20 minutes, and 1 hour. Descriptive statistical indices for UDMA and the results of the *post hoc* analysis are given in Table 4. In the column "mean concentration" of the Table 4, mean values that are followed by common letter(s) in parenthesis, are not statistically significant different according to the Bonferroni criterion at p<0.05. The graphic representation of the mean concentration of UDMA in relation to time is shown in Table 6 and follows the equation:

$y = -0.1677x^4+2.0513x^3-9.5458x^2+20.236x-15.652$, $R^2=0.9895$

DISCUSSION

In this study, the trans-dentinal permeability of some of the constituents of the resin cement was evaluated with regards to different time intervals. In general, our results seem to agree with the results of other researchers who also state that the elution of monomers from resin materials decline from a certain time interval and then,[12,33,34] but further explanations are given below. TEGDMA was detected in most of the samples. At the first-time interval (5 minutes) TEGDMA was not detected in any of the samples while at the second-time interval (20 minutes), TEGDMA was detected in nine of the ten samples. Hamid & Hume[35] detected TEGDMA in the pulp chamber at 14.4 min, while Gerzina & Hume,[10, 36] detected TEGDMA in the pulp chamber at 43.2 min. This discrepancy can be attributed to differences of the Detection Limit (LOD) of the analytic method or to the different amount of TEGDMA that was eluted from the different materials. At the fifth-time interval (21 hours), an increase of the mean concentration of TEGDMA was noticed and the mean concentration was further increased at the sixth-time interval (3 days). As these two time intervals were more extended than the previous time intervals, the elution media stayed in contact with the dentin disk for longer time periods, 19 hours and 51 hours respectively. During these extended time periods the mean concentration of TEGDMA that was detected through dentin, increased. On the contrary, in the following time intervals a descending trend of the mean concentration of TEGDMA was noticed, although those time intervals were even greater.

Table 3. Descriptive statistics of the concentration of TEGDMA

Time Intervals	N	Min (mg/L)	Max (mg/L)	Mean (mg/L)	SE	SD
(1) 5 minutes	10	0.00	0.00	0.00(z)	0	0
(2) 20 minutes	10	0.00	1.60	0.34 (a,b,c,d,e,z)	0.14	0.45
(3) 1 hour	10	0.19	0.58	0.27 (b,c,e)	0.04	0.12
(4) 2 hours	10	0.00	0.44	0.23 (b,d,e)	0.04	0.12
(5) 21 hours	10	0.22	1.40	0.71 (a,c)	0.10	0.32
(6) 3 days	10	0.32	1.70	0.95 (a)	0.14	0.44
(7) 7 days	10	0.20	1.70	0.83 (a,b)	0.15	0.47
(8) 10 days	10	0.20	1.10	0.49(c,d)	0.10	0.31
(9) 21 days	10	0.00	0.43	0.13(e,z)	0.06	0.18

Mean values followed by common letter(s) in parenthesis, are not statistically significant different according to the Bonferroni criterion at p<0.05.

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Table 4. Descriptive statistics of the concentration of UDMA

Time	N	Min (mg/L)	Max (mg/L)	Mean (mg/L)	SE	SD
(1) 5 minutes	10	0.00	0.00	0.00(b)	0	0
(2) 20 minutes	10	0.00	0.00	0.00 (b)	0	0
(3) 1 hour	10	0.00	0.00	0.00 (b)	0	0
(4) 2 hours	10	0.00	1.58	0.6 (a,b)	0.21	0.67
(5) 21 hours	10	0.00	1.42	0.95 (a)	0.12	0.39
(6) 3 days	10	0.00	1.4	1.04 (a)	0.12	0.38
(7) 7 days	10	0.00	1.36	0.97 (a)	0.12	0.39
(8) 10 days	10	0.00	1.28	0.72(a,b)	0.16	0.51
(9) 21 days	10	0.00	1.4	0.33(a,b)	0.17	0.55

Mean values followed by common letter(s) in parenthesis, are not statistically significant different according to the Bonferroni criterion at $p<0.05$

After the repeated renewals of the Ringer's solution, the cement probably, "depleted" in TEGDMA. At the end of the experiment (21 days), TEGDMA was not detected in 60% of the samples and for the remaining 40%, the elution of TEGDMA continued, but the concentration was too low. To sum up, after the sixth-time interval (3 days), the rate of elution of TEGDMA gradually slowed down and the whole phenomenon seemed to end for the 60% of the specimens on the 21st day.

The findings for UDMA were rather similar, but UDMA compound was detected from the fourth-time interval (2 hours) and thereafter. UDMA's concentration gradually increased until the sixth-time interval (3 days) and then slowly decreased, until the end of the experiment. The higher Limit of Detection (LOD) of the chromatographic method for UDMA and/or the higher molecular weight of the compound might explain the finding that UDMA was not detected until the fourth-time interval (2 hours) while TEDMA was detected much earlier. In a previous study,[6] UDMA was not detected through a dentin disk, while BisGMA, a hydrophobic compound with even greater molecular weight, manage to pass through dentin. To our knowledge, there is no other study for the trans-dentinal diffusion of UDMA. In the present paper, a different *in vitro* model was used, the study considered a more extended time period and the results were different. Also, Örtengren *et al* examined the elution of monomer from the same resin cement in water.[13] They detected quantifiable concentration of TEGDMA and UDMA and just detectable concentration of BisGMA. Combining their results with the results of our study, a possible explanation is given to the question why UDMA was detected and BisGMA was not.

BPA and BisGMA were not detected in any sample. This, it might mean that these compounds were not present in the eluate, or their concentrations were too low, below the limit of the detection of the method. An unknown compound was also detected at 3.3 minutes. This compound was not HEMA since the resin cement does not contain HEMA and over more, the UV spectra of these compounds were very different to each other. Over more, in Figure 4 a chromatogram from the control specimen is presented, to show that there are no peaks to interfere with the examined compounds. The control specimen was a specimen that it was constructed in the same way like the other specimens, but neither cement nor adhesives were applied on it and the renewal of the solution followed the same time intervals.

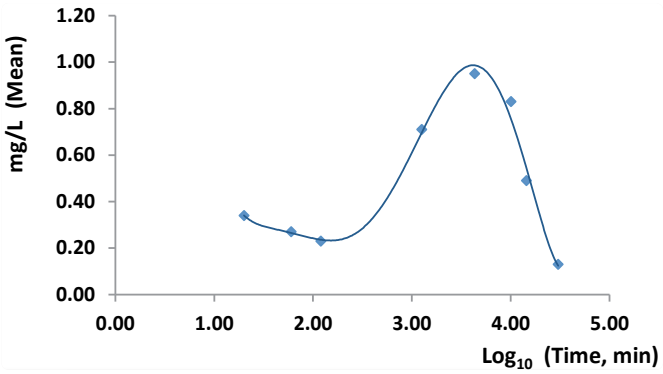

Figure 4: Mean concentration of TEGDMA in relation with time

The *in vitro* model used in the present study is quite relevant to the conditions of fixed prosthesis cementation. The mean surface area of the disks' dentin, as it was calculated by the Image J software program from the digital photos, was 65.86 mm^2 ± 1.54. Tiu *et al.*[37] have calculated the mean surface area of several types of teeth (anterior and posterior) that were prepared for complete crowns by general dentists. The mean surface area of the prepared teeth ranged from 33.97 mm^2 to 105.44 mm^2 for the cone frustum formula and from 41.75 mm^2 to 117.50 mm^2 for the right truncated pyramid formula.[37] So, the disks obtained from big third molar teeth in the present study, had area relevant with the surface area of teeth prepared for complete crowns. The steady force of 25 N, applied by a digital force gauge, was selected because it falls within the range of the forces that are applied, during clinical cementation of dental crowns.[38] Also, the combination of a low seating force (25 N) with a 40 μm space for the cement it is proposed because it seems to offer a quick seating of a restoration with minimal distortion.[39] The simulation of the positive intrapulpal pressure of human teeth 11 at 14.1 cm H_2O, was achieved by filling the specimens with Ringer's solution up to 14.1 cm height, following the type of hydrostatic pressure $P=\varepsilon$ x *h:* and the amount of the resin cement that was applied to each specimen was expressed as volume of the cement following the type $V=S$ x *h* which calculates the volume of a cylinder.[31]

In conclusion, the hypothesis stated at the start of the study is partially accepted. From all the examined compounds, only TEGDMA and UDMA were identified in the pulp chamber space. Since concentration higher than 0.5 – 5 mM (143 mg/L- 1430 mg/L) for TEGDMA and 0.05 mM (23.5 mg/L) for UDMA is considered to be toxic according to the different cell lines[40] the concentration that was detected was below the toxic levels. Under the conditions that were chosen in the present study, the resin cement did not elute monomers in toxic concentration.

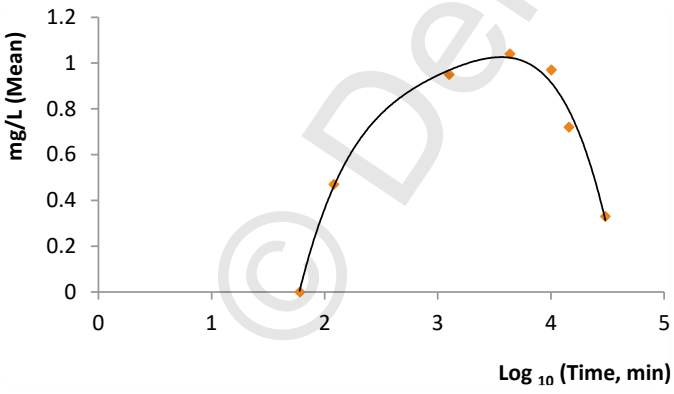

Figure 5: Mean concentration of UDMA in relation with time

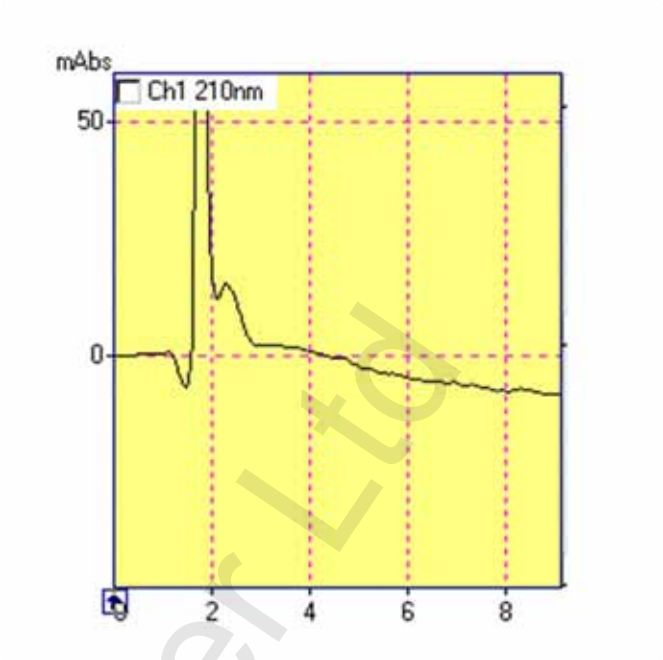

Figure 6: Blank chromatogram at 2 h

CONCLUSIONS

1. BPA and BisGMA compounds were not detected through the dentin disk in any eluate of the specimens' samples while TEGDMA and UDMA compounds were detected in several specimens' samples.
2. The concentration of TEGDMA and UDMA compounds that was eluted from the dual cured resin cement through a 0.85 mm dentin disk and under a pulpal pressure of 14.1 cm H_2O, did not reach toxic levels for the pulp.
3. Time had a significant effect on the concentration of TEGDMA and UDMA

CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest

'MANUFACTURERS DETAILS'.

- Syntac Primer, Syntac Adhesive, Heliobond, Monobond plus, Variolink II catalyst and base : all by Ivoclar Vivadent, Liechtenstein
- Low speed saw, ISOMET, Buehler GmbH.
- 35% phosphoric acid, Ultra etch, Ultradent GmbH
- Aquarium marine silicone, Top sil, Mercola, Grecce
- Acrylic resin, Kallocryl, Speiko GmbH
- Light curing unit, Power blue, Heraeus Kultzer GmbH
- Oven, Memmert, GmbH Co

- Photodiode array detector SPD-M6A, class-M10A computing integrator, automatic Injector SIL-9A, FCV-9A solvent mixing system, LC9AD pump: all by Shimadzu GmbH.
- Column, Perfectsil target ODS 3 (25 cm 4.6 mm id, 5 µM) MZ-Analysentechnick GmbH

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