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IN VITRO EVALUATION OF RESIDUAL MONOMER RELEASED AFTER POLYMERIZATION OF RESTORATIVE MATERIALS USED IN PEDIATRIC DENTISTRY

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ABSTRACT

Introduction: The release of residual monomers from dental restorative materials is a critical factor influencing their biocompatibility and long-term performance. In pediatric dentistry, materials like composite resins, compomers, and giomers are commonly used due to their aesthetic and functional properties. The aim of this study was to compare the release of residual monomers from three common pediatric restorative materials via high-performance liquid chromatography (HPLC). Materials and methods: Three restorative materials (3M Filtek One Bulk Fill Composite, Dentsply Sirona Dyract XP Compomer, and Shofu Beautifil 2 Giomer) were used. The amount of residual monomer released at each time interval was determined

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via HPLC. Discs of the restorative materials to be examinedwere prepared using standard Teflon molds for each group (n=7), and the materials were polymerized using an LED light source according to the manufacturers' recommendations. The data were statistically analyzed via IBM SPSS V23. Results: The retention times for the Bis-GMA, UDMA, and TEGDMA standard monomers were 6.7, 5.6, and 4.4 min, respectively. Residual release of Bis-GMA and UDMA was detected from all the materials, whereas TEGDMA release was not observed in the giomer. Significant differences in monomer release quantities among the restorative materials were observed over time, including in total residual monomer release. Conclusion: Compared with the giomer and compomer materials, the composite material resulted in lower residual monomer release. Further research and measures are needed to minimize residual monomer release and improve polymerization in restorative materials.

KEYWORDS: Composite; Compomer; Giomer; High-performance liquid chromatography; Biocompatibility.

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EVALUACIÓN IN VITRO DEL MONÓMERO RESIDUAL LIBERADO TRAS LA POLIMERIZACIÓN DE MATERIALES RESTAURADORES UTILIZADOS EN ODONTOLOGÍA PEDIÁTRICA

RESUMEN

Introducción: La liberación de monómeros residuales de los materiales restauradores dentales es un factor crítico que influye en su biocompatibilidad y rendimiento a largo plazo. En odontología pediátrica, materiales como las resinas compuestas, los compómeros y los giómeros se utilizan comúnmente debido a sus propiedades estéticas y funcionales. El objetivo de este estudio fue comparar la liberación de monómeros residuales de tres materiales restauradores pediátricos comunes mediante cromatografía líquida de alta resolución (HPLC). Materiales y métodos: Se utilizaron tres materiales restauradores (3M Filtek One Bulk Fill Composite, Dentsply Sirona Dyract XP Compomer y Shofu Beautifil 2 Giomer). La cantidad de monómero residual liberado en cada intervalo de tiempo se determinó mediante HPLC. Se prepararon discos de los materiales restauradores a examinar utilizando moldes de teflón estándar para cada grupo (n=7), y los materiales se polimerizaron utilizando una fuente de luz LED según las recomendaciones del fabricante. Los datos se analizaron estadísticamente mediante IBM SPSS V23. Resultados: Los tiempos de retención para los monómeros estándar Bis-GMA, UDMA y TEGDMA fueron de 6,7, 5,6 y 4,4 min, respectivamente. Se detectó liberación residual de Bis-GMA y UDMA en todos los materiales, mientras que no se observó liberación de TEGDMA en el giómero. Se observaron diferencias

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significativas en las cantidades de liberación de monómero entre los materiales restauradores a lo largo del tiempo, incluyendo la liberación total de monómero residual. Conclusión: En comparación con los materiales giómero y compómero, el material compuesto resultó en una menor liberación residual de monómero. Se necesitan más investigaciones y medidas para minimizar la liberación residual de monómero y mejorar la polimerización en los materiales restauradores.

PALABRAS CLAVE: Compuesto; Compómero; Giómero; Cromatografía líquida de alta resolución; Biocompatibilidad.

INTRODUCTION

Polymers are large molecules composed of repeating monomer units, and their physical and mechanical properties vary according to monomer type and molecular configuration (1–3). Structurally, polymers may be branched, cross-linked, or linear (4). Complete polymerization requires all carbon double bonds to react; however, this process is often incomplete. Monomers that fail to integrate into the polymer chain are referred to as residual monomers (5), and their complete elimination after polymerization is not always possible (6). Although typically present at low concentrations, residual monomers may pose toxic risks, particularly in medical and food-related applications (7).

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Resin-based dental restorative materials are primarily composed of polymer matrices formed by monomers such as bisphenol A-glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA), the latter improving material flow and polymer formation (8–12). In dentistry, incomplete polymerization of these materials may result in residual monomer release into the oral environment. These substances can dissolve in oral fluids and negatively affect surrounding tissues. Insufficient polymerization has been associated with reduced mechanical strength and durability, as well as cytotoxic, mutagenic, estrogenic, and inflammatory effects on pulpal, gingival, and oral mucosal tissues (8,13).

With the increasing use of resin-based materials, concerns regarding their biological safety have grown. Previous studies have demonstrated that residual monomers released from resin materials may induce cytotoxic effects, including cell death in in vitro models (14). Therefore, optimizing polymerization protocols and applying appropriate post-polymerization procedures are essential to reduce residual monomer content (15).

The aim of this study was to evaluate the amount of residual monomer released from composite, compomer, and giomer restorative materials commonly used in pediatric dentistry. Monomer release was analyzed using high-performance liquid chromatography (HPLC) at predetermined

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time intervals: 6 h, 24 h, 7 days, 15 days, 21 days, 24 days, and 30 days.

The null hypotheses (H_0) of this study are as follows:

1. There was no significant difference in monomer release between the composite material and the compomer material.
2. There was no significant difference in monomer release between the compomer material and the giomer material.
3. There was no significant difference in monomer release between the composite material and the giomer material.

Materials and methods

Determination of disc size

To ensure the reliability of the study, a statistical power analysis was conducted via the G*Power program prior to the study. The effect size for residual monomer release after polymerization was determined to be 0.927. With a standard deviation of 2.97, a power of 0.80, and α set at 0.05, the minimum disc size required for each group was determined to be n=5. Therefore, a total of 120 disks were used, with 5 discs per group, to conduct the study.

Materials

This study evaluated residual monomer release from one composite, one compomer, and one giomer material

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commonly used in primary and permanent teeth: bulk-fill composite (Filtek Bulk Fill, 3M ESPE), compomer (Dyract XP, Dentsply Sirona), and giomer (Beautifil 2, Shofu) (Table 1).

Table 1. Materials used in the study and their chemical contents.

The commercial names of the components	The compositions
Bulk fill composite (Filtek bulk fill, 3M ESPE)	Free-form 20nm silica filler, 4 to 11nm zirconia filler, aggregated zirconia/silica cluster filler, agglomerate of 100nm particles of ytterbium trifluoride filler, AFM (Active Monomer), Audma, UDMA, and 1,12-dodecane-DMA, Bis-GMA, TEGDMA.
Compomer (Dyract XP, Dentsply Sirona)	UDMA, Bis-GMA, TEGDMA, camphorquinone, ethyl 4-(dimethylamino)benzoate, carboxylic acid modified dimethacrylate, BHT (Butylated hydroxytoluene), strontium-alumino-sodium-fluoro-phosphosilicate glass, highly dispersed silicon dioxide, UV stabilizer, strontium fluoride, titanium dioxide, and iron oxide pigments.
Giomer (Beautifil 2, Shofu)	Bis-GMA, inorganic glass fillers, aluminum oxide, silica, DL-camphorquinone, pre-activated glass ionomer.

In this study, a rainbow light curing device was used for the polymerization of the examined restorative materials. This device is rechargeable, with a power capacity of 2200 mAh and light intensity ≥1200 mW/cm² at 5 W, and it emits light in the 450–470 nm wavelength range. It offers polymerization in different modes and

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durations. A radiometer (Woodpecker LM-1) was used to measure the light intensity of the LED curing device used in the study. The colors of the examined materials were specified as A2, and polymerization was performed with a constant curing distance for each material.

To calculate residual monomer levels, a HPLC system (Agilent 1100 series) equipped with a G1311A QuatPump, a G1313A Standard Autosampler, and a G1365B UV–Vis detector was used. The HPLC system was calibrated with bisphenol A glycerolate dimethacrylate, triethylene glycol dimethacrylate, and diurethane dimethacrylate from Sigma Aldrich (Table 2).

Table 2. Characteristics of materials used in the HPLC calibration.

	Commercial Name	Manufacturer's Product Number	Molecular Weight	Molecular Formula
Bis-GMA	Bisphenol A glycerolate dimethacrylate	Sigma-Aldrich 494356	512.59 gr/mol	$C_{29}H_{36}O_8$
UDMA	Diurethane dimethacrylate	Sigma-Aldrich 436909	470.57 gr/mol	$C_{23}N_2O_7H_{37}$
TEGDMA	Triethylene glycol dimethacrylate	Sigma-Aldrich 261548	286.33 gr/mol	$C_{14}H_{22}O_6$

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Methods

Discs' Preparation

In this study, discs were created via premade Teflon molds with a height of 2 mm and a diameter of 5 mm. Restorative materials were applied to these molds, which were positioned on a flat glass plate, in equal amounts according to the manufacturer's recommendations. Excess material was removed by placing glass plates on the top and bottom surfaces of the molds and applying slight pressure to ensure even distribution and eliminate excess material.

Following the manufacturer's instructions, the LED light curing device was calibrated with a radiometer, and the materials were polymerized for 20 seconds. The discs were then polished underwater for 10 s using 3M polishing discs. After polishing, the discs were weighed on a precision scale and placed into bottles containing solvents.

Storage of Discs

A total of 120 disc from three different groups were placed into labeled, lightproof, 30 mL amber glass bottles with screw caps containing 80% ethanol and 20% water solutions. Each disc was placed in a 10 mL volume of the solution. At specific intervals (5 min, 6 h, 24 h, 7 days, 15 days, 21 days, 24 days, and 30 days), 1 mL discs were withdrawn from the bottles. After the extraction times were recorded, all the bottles were incubated at 37°C until the measurement days. At the designated intervals, 1 mL discs were extracted via

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Eppendorf pipettes and transferred into prelabeled 1.5 mL glass vials for residual monomer measurement.

Introduction of standard monomers to the HPLC device

Before HPLC studies are conducted, the instrument must be prepared according to the experimental conditions. This preparation involves determining and calibrating the chromatographic conditions of the device. Initially, the monomer solutions are diluted to a specific ratio and injected into the HPLC system to determine the retention times (the time required for a solute to pass through a chromatography column and reach the detector) and peak values of the monomers. An ethanol/water mixture was used as the mobile phase and injected into the HPLC device to create a suitable environment for monomer analysis.

Discs' Analyses

During the HPLC analysis, the system was set to room temperature with a mobile phase flow rate of 1 mL/min. Each disc was injected with 20 µL, and chromatograms were recorded at a wavelength of 204 nm, where monomers show maximum absorption. Calibration curves for each monomer were prepared using standard solutions. Given that the longest retention time for the monomers was 5 min, each chromatogram was obtained with a run time of approximately 7 min under an average pressure of 6.9 MPa. Measurements were taken on the basis of the peaks identified for the monomers of

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interest. The peak points and peak areas were analyzed to determine the amounts of unreacted monomers present.

Statistical Analyses

The data were analyzed via IBM SPSS V23. The Shapiro–Wilk test was used to assess data normality. For normally distributed data with three or more groups, one-way ANOVA was used for comparisons, and Tamhane's T2 test was employed for multiple comparisons. For nonnormally distributed data with three or more groups, the Kruskal–Wallis test was used, and the Dunn test was applied for multiple comparisons. Repeated-measures ANOVA was used for within-group comparisons over time with normally distributed data, with multiple comparisons adjusted via the Bonferroni correction. For nonnormally distributed data, the Dunn test was used for within-group comparisons over time.

Results were expressed as mean ± standard deviation and median (minimum–maximum). Statistical significance was set at $p < 0.05$.

Results

The retention times of the standard monomers were determined as 6.7 min for Bis-GMA (Figure 1), 5.6 min for UDMA (Figure 2), and 4.4 min for TEGDMA (Figure 3). All disc samples were analyzed according to these retention times.

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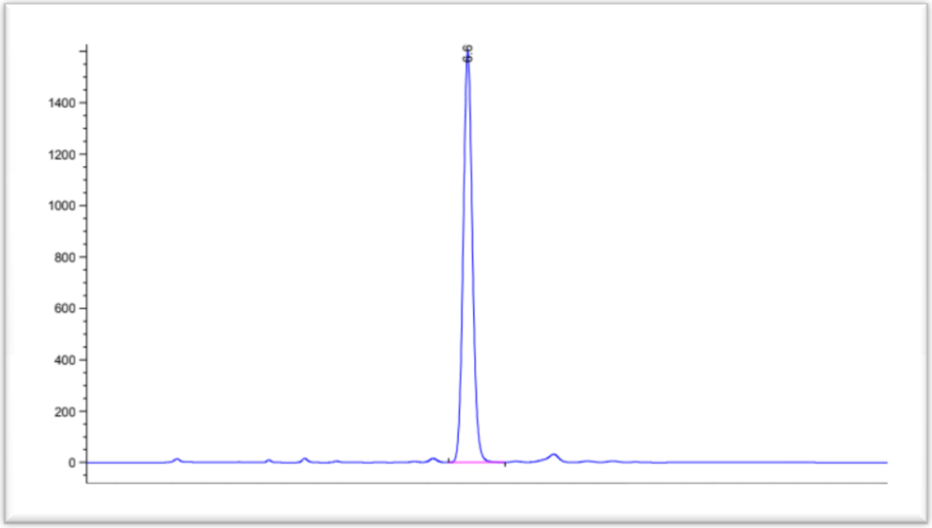

Figure 1. Retention time and peak area for the BIS-GMA monomer.

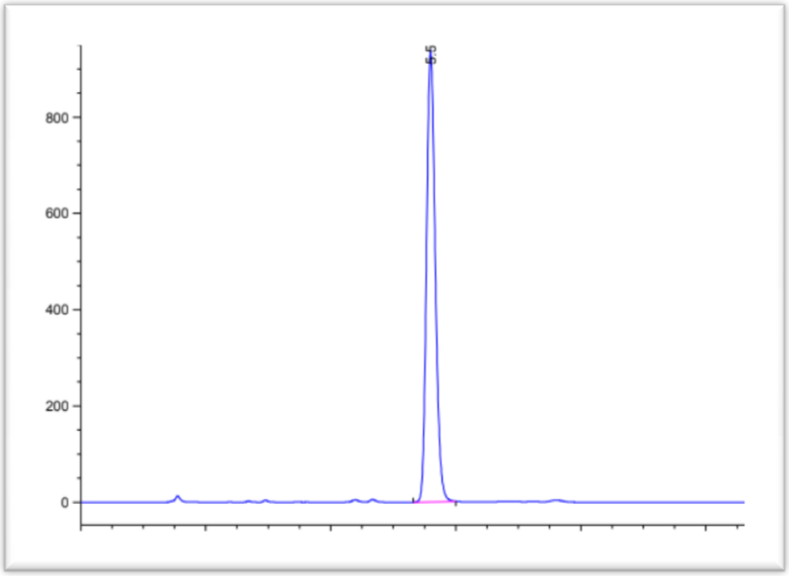

Figure 2. Retention time and peak area for the UDMA monomer.

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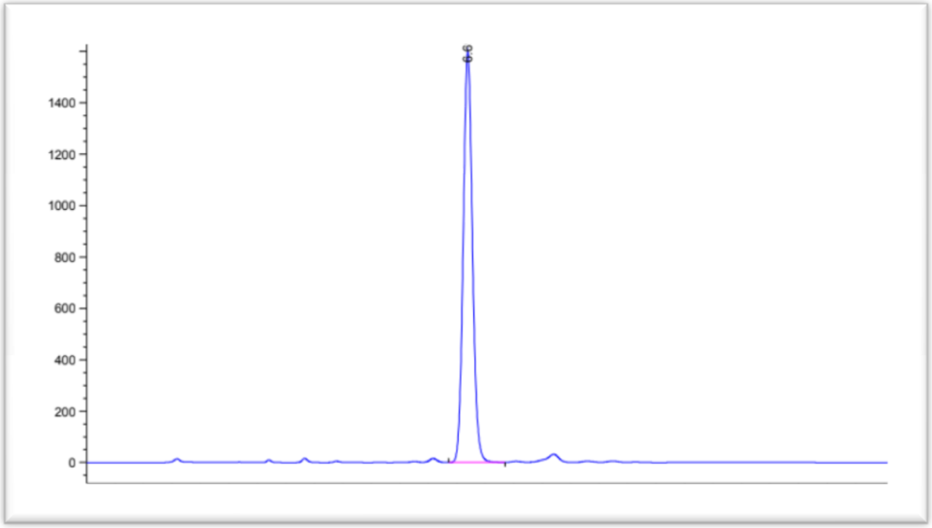

Figure 3. Retention time and peak area for the TEGDMA monomer.

HPLC Findings According to Material Type and Measurement Period

Bis-GMA and UDMA release was detected in all tested restorative materials, whereas TEGDMA release was not detected in the giomer group. Comparisons of Bis-GMA peak area values according to measurement times are presented in Table 3, and statistically significant differences were observed among the groups at all measurement periods ($p < 0.05$).

Comparisons of UDMA peak area values are shown in Table 4, demonstrating statistically significant differences between the groups throughout all measurement periods ($p < 0.001$).

TEGDMA release values according to the number of measurements for each group

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are presented in Table 5. Statistically significant differences in TEGDMA release were detected during several measurement periods.

Table 3. Comparison of BIS-GMA peak area values according to materials.

Monomer	Variable	COMPOSITE		COMPOMER		GIOMER		Test ist.	p
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)		
BIS-GMA	Area 5th min	1.44 ± 0.55	1.24 (1.10 - 2.42) [a]	16.92 ± 1.51	16.34 (15.42 - 19.36) [ab]	482.74 ± 89.46	464.39 (384.50 - 600.97) [b]	12.500 [2]	**0.002**
BIS-GMA	Area 6th h	2.54 ± 1.29 [a]	2.00 (1.53 - 4.75)	54.77 ± 25.61 [b]	62.19 (10.54 - 77.00)	1083.36 ± 275.62 [c]	1129.00 (761.25 - 1465.73)	43.392 [1]	**0.001**
BIS-GMA	Area 24th h	2.01 ± 0.41 [a]	2.03 (1.56 - 2.56)	45.10 ± 22.34 [b]	47.24 (7.95 - 63.75)	1223.03 ± 186.94 [c]	1284.30 (1025.55 - 1462.23)	103.069 [1]	**<0.001**
BIS-GMA	Area 7th day	3.57 ± 0.73 [a]	3.38 (2.66 - 4.63)	9.94 ± 0.92 [b]	9.94 (8.60 - 11.13)	1209.74 ± 235.72 [c]	1294.30 (860.06 - 1417.95)	126.617 [1]	**<0.001**
BIS-GMA	Area 15th day	160.63 ± 58.94 [a]	134.94 (119.23 - 261.56)	39.79 ± 20.11 [b]	45.26 (6.27 - 59.65)	1706.52 ± 255.48 [c]	1702.83 (1451.54 - 1995.72)	102.119 [1]	**<0.001**
BIS-GMA	Area 21st day	427.51 ± 147.38 [a]	330.88 (316.08 - 629.54)	14.96 ± 2.12 [b]	14.54 (12.22 - 17.94)	2495.73 ± 665.80 [c]	2841.81 (1615.17 - 3107.30)	48.258 [1]	**<0.001**
BIS-GMA	Area 24th day	121.86 ± 108.10 [a]	161.73 (6.19 – 237.63)	38.36 ± 21.27 [a]	45.19 (7.95 - 63.75)	1554.89 ± 285.55 [b]	1677.99 (1101.48 - 1781.56)	63.654 [1]	**<0.001**
BIS-GMA	Area 30th day	11.46 ± 2.47 [a]	12.26 (8.76 – 13.69)	23.29 ± 1.05 [b]	23.02 (22.12 - 24.64)	1594.43 ± 307.51 [c]	1723.91 (1115.36 - 1834.27)	102.759 [1]	**<0.001**

[1]One-way ANOVA, [2] Kruskal-Wallis H test, a-c: Groups with the same letter have no significant difference between them.

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Table 4. Comparison of UDMA peak area values according to materials.

Monomer	Variable	COMPOSITE		COMPOMER		GIOMER		Test ist.	p
		Mean± SD	Median (Min - Max)	Mean± SD	Median (Min - Max)	Mean± SD	Median (Min - Max)		
UDMA	Area 5th min	10.82 ± 2.66 [a]	9.19 (8.91 - 14.99)	159.53 ± 17.50 [b]	167.47 (129.71 - 171.87)	267.92 ± 56.00 [c]	268.87 (203.30 - 350.52)	202.990 [1]	**<0.001**
UDMA	Area 6th h	14.80 ± 4.29 [a]	12.89 (10.40 - 19.94)	394.91 ± 45.71 [b]	394.17 (347.03 - 454.64)	515.91 ± 147.43 [b]	523.54 (356.13 - 725.18)	177.832 [1]	**<0.001**
UDMA	Area 24th h	10.09 ± 5.56	7.57 (6.67 - 19.81) [a]	276.98 ± 108.96	226.17 (184.03 - 450.41) [ab]	506.91 ± 90.33	519.23 (397.27 - 634.09) [b]	11.580 [2]	**0.003**
UDMA	Area 7th day	45.15 ± 62.03	18.43 (14.49 - 156.07) [a]	767.22 ± 728.08	424.55 (285.04 - 2042.64) [b]	449.79 ± 91.35	490.19 (324.00 - 532.05) [b]	9.420 [2]	**0.009**
UDMA	Area 15th day	22.17 ± 11.09 [a]	18.28 (12.46 - 40.28)	318.16 ± 96.07 [b]	277.97 (236.50 - 466.65)	558.21 ± 105.07 [c]	497.60 (469.17 - 721.04)	77.400 [1]	**<0.001**
UDMA	Area 21st day	54.84 ± 60.54	29.67 (21.11 - 162.35) [a]	615.03 ± 64.10	612.78 (533.01 - 710.43) [ab]	884.35 ± 243.73	959.97 (568.63 - 1203.48) [b]	10.820 [2]	**0.004**
UDMA	Area 24th day	20.73 ± 7.04 [a]	21.93 (11.64 - 30.80)	279.02 ± 113.03 [b]	226.17 (184.03 - 460.58)	474.37 ± 98.07 [b]	520.45 (337.97 - 567.31)	58.712 [1]	**<0.001**
UDMA	Area 30th day	24.82 ± 5.21 [a]	23.85 (19.05 - 30.80)	381.56 ± 46.69 [b]	395.18 (305.57 - 430.38)	475.19 ± 105.13 [b]	498.41 (323.61 - 568.30)	168.321 [1]	**<0.001**

[1]One-way ANOVA, [2] Kruskal-Wallis H test, a-c: Groups with the same letter have no significant difference between them.

Table 5. Comparison of TEGDMA peak area values monomers according to groups.

Monomer	Valiable	COMPOSITE		COMPOMER		Test ist.	p
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)		
TEGDMA	Area 5th min	111.19 ± 41.61	98.31 (74.03 - 182.60)	30.24 ± 3.65	28.90 (26.96 - 35.75)	4.334 [1]	**0.002**
TEGDMA	Area 6th h	229.03 ± 89.75	190.22 (151.98 - 374.82)	148.91 ± 12.98	148.40 (137.40 - 170.21)	1.975 [1]	0.117
TEGDMA	Area 24th h	161.12 ± 47.85	141.38 (127.23 - 241.64)	129.67 ± 24.92	120.37 (107.07 - 172.01)	1.303 [1]	0.229
TEGDMA	Area 7th day	243.05 ± 34.92	242.45 (208.43 - 296.48)	316.67 ± 25.34	329.22 (273.84 - 335.53)	-3.815 [1]	**0.005**
TEGDMA	Area 15th day	259.54 ± 61.10	236.67 (212.23 - 359.51)	332.86 ± 40.83	331.98 (292.36 - 394.44)	-2.231 [1]	0.056
TEGDMA	Area 21st day	553.70 ± 261.70	494.26 (292.06 - 994.45)	620.50 ± 16.63	628.39 (598.18 - 638.29)	-0.570 [1]	0.585
TEGDMA	Area 24th day	361.33 ± 61.64	376.60 (293.29 - 439.44)	208.64 ± 173.10	128.93 (107.07 - 515.21)	20.000 [2]	0.151
TEGDMA	Area 30th day	375.73 ± 68.03	392.15 (307.92 - 467.59)	530.93 ± 14.70	529.62 (511.37 - 545.41)	-4.986 [1]	**0.006**

[1]Independent Samples t-Test, [2] Mann-Whitney U Test.

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Comparison of Monomer Release Values Among Materials

For Bis-GMA, significant differences among the materials were observed at all evaluated time points, including early (5 min), intermediate (6 h, 24 h, 7 and 15 days), and late periods (21, 24, and 30 days). These differences were mainly observed between the giomer material and the other restorative materials (Table 6).

Table 6. Comparison of BIS-GMA monomer result values according to groups.

Monomer	Valiable	COMPOSITE		COMPOMER		GIOMER		Test ist.	p
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)	Mean± SD	Median (Min - Max)		
BIS-GMA	Result 5th min	0.04 ± 0.02	0.04 (0.03 - 0.07) [a]	0.48 ± 0.04	0.47 (0.44 - 0.55) [ab]	13.05 ± 2.55	12.52 (10.24 - 16.42) [b]	12.500 [2]	**0.002**
BIS-GMA	Result 6th h	0.07 ± 0.04 [a]	0.06 (0.04 - 0.14)	0.98 ± 0.43 [b]	1.05 (0.30 - 1.47)	30.18 ± 7.86 [c]	31.48 (20.99 - 41.08)	42.663 [1]	**0.001**
BIS-GMA	Result 24th h	0.06 ± 0.01 [a]	0.06 (0.04 - 0.07)	0.71 ± 0.36 [b]	0.63 (0.23 - 1.10)	34.16 ± 5.33 [c]	35.91 (28.53 - 40.98)	98.287 [1]	**<0.001**
BIS-GMA	Result 7th day	0.10 ± 0.02 [a]	0.10 (0.08 - 0.13)	0.28 ± 0.03 [b]	0.28 (0.25 - 0.32)	33.78 ± 6.72 [c]	36.19 (23.81 - 39.72)	124.140 [1]	**<0.001**
BIS-GMA	Result 15th day	4.58 ± 1.68 [a]	3.85 (3.40 - 7.46)	0.56 ± 0.30 [b]	0.57 (0.18 - 0.98)	47.95 ± 7.29 [c]	47.85 (40.68 - 56.20)	106.088 [1]	**<0.001**
BIS-GMA	Result 21st day	12.19 ± 4.20	9.44 (9.02 - 17.96) [ab]	-0.15 ± 0.34	-0.31 (-0.37 - 0.45) [a]	70.46 ± 18.99	80.33 (45.35 - 87.90) [b]	12.500 [2]	**0.002**
BIS-GMA	Result 24th day	3.48 ± 3.08 [a]	4.61 (0.18 - 6.78)	0.52 ± 0.40 [a]	0.57 (0.07 - 1.10)	43.63 ± 8.14 [b]	47.14 (30.69 - 50.09)	64.118 [1]	**<0.001**
BIS-GMA	Result 30th day	0.33 ± 0.07	0.35 (0.25 - 0.39) [ab]	0.09 ± 0.34	-0.07 (-0.09 - 0.70) [a]	44.75 ± 8.77	48.45 (31.09 - 51.59) [b]	10.519 [2]	**0.005**

[1] One-way ANOVA, [2] Kruskal-Wallis H Test, a-c: Groups with the same letter have no significant difference between them.

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UDMA release values showed statistically significant differences among the materials at early (5 min, 6 h), intermediate (24 h, 7 and 15 days), and late measurement periods (21, 24, and 30 days) (Table 7).

Table 7. Comparison of UDMA monomer result values according to groups.

Monomer	Valiable	COMPOSITE		COMPOMER		GIOMER		Test ist.	p
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)		
UDMA	Result 5th min	0.57 ± 0.14 [a]	0.49 (0.47 - 0.79)	6.75 ± 0.93 [b]	7.17 (5.17 - 7.41)	12.49 ± 2.97 [c]	12.54 (9.07 - 16.86)	132.010 [1]	**<0.001**
UDMA	Result 6th h	0.78 ± 0.23 [a]	0.68 (0.55 - 1.06)	19.21 ± 2.42 [b]	19.18 (16.68 - 22.38)	25.62 ± 7.81 [b]	26.03 (17.16 - 36.70)	150.114 [1]	**<0.001**
UDMA	Result 24th h	0.53 ± 0.29	0.40 (0.35 - 1.05) [a]	12.97 ± 5.77	10.28 (8.05 - 22.15) [ab]	25.14 ± 4.78	25.80 (19.34 - 31.88) [b]	11.580 [2]	**0.003**
UDMA	Result 7th day	2.39 ± 3.28	0.98 (0.77 - 8.26) [a]	38.93 ± 38.55	20.78 (13.40 - 106.45) [b]	22.12 ± 4.84	24.26 (15.46 - 26.48) [b]	9.420 [2]	**0.009**
UDMA	Result 15th day	1.17 ± 0.59 [a]	0.97 (0.66 - 2.13)	15.15 ± 5.09 [b]	13.02 (10.83 - 23.01)	27.86 ± 5.56 [c]	24.65 (23.15 - 36.48)	66.623 [1]	**<0.001**
UDMA	Result 21st day	2.90 ± 3.21	1.57 (1.12 - 8.60) [a]	30.87 ± 3.39	30.75 (26.53 - 35.92) [ab]	45.13 ± 12.90	49.13 (28.41 - 62.02) [b]	10.820 [2]	**0.004**
UDMA	Result 24th day	1.10 ± 0.37 [a]	1.16 (0.62 - 1.63)	13.08 ± 5.98 [b]	10.28 (8.05 - 22.69)	23.42 ± 5.19 [b]	25.86 (16.20 - 28.34)	49.618 [1]	**<0.001**
UDMA	Result 30th day	1.31 ± 0.28 [a]	1.26 (1.01 - 1.63)	18.51 ± 2.47 [b]	19.23 (14.48 - 21.09)	23.46 ± 5.57 [b]	24.69 (15.44 - 28.40)	140.866 [1]	**<0.001**

[1]One-way ANOVA, [2] Kruskal-Wallis H Test, a-c: Groups with the same letter have no significant difference between them.

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For TEGDMA, significant differences were primarily detected between the composite and compomer materials at early and late measurement periods, while no significant differences were observed at some intermediate time points. TEGDMA was not detected in the giomer group at any measurement period (Table 8).

Table 8. Comparison of TEGDMA monomer result values according to groups.

Monomer	Valiable	COMPOSITE		COMPOMER		Test ist.	p
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)		
TEGDMA	Result 5th min	6.18 ± 2.33	5.46 (4.10 - 10.18)	1.64 ± 0.20	1.57 (1.46 - 1.95)	4.334 [1]	**0.002**
TEGDMA	Result 6th h	12.78 ± 5.03	10.61 (8.47 - 20.96)	8.29 ± 0.73	8.27 (7.65 - 9.49)	1.975 [1]	0.117
TEGDMA	Result 24th h	8.98 ± 2.68	7.87 (7.08 - 13.49)	7.22 ± 1.40	6.70 (5.95 - 9.59)	1.303 [1]	0.229
TEGDMA	Result 7th day	13.57 ± 1.96	13.54 (11.63 - 16.57)	17.70 ± 1.42	18.40 (15.30 - 18.75)	-3.815 [1]	**0.005**
TEGDMA	Result 15th day	14.50 ± 3.42	13.21 (11.84 - 20.10)	18.61 ± 2.29	18.56 (16.33 - 22.06)	-2.231 [1]	0.056
TEGDMA	Result 21st day	30.98 ± 14.67	27.65 (16.32 - 55.69)	34.73 ± 0.93	35.17 (33.48 - 35.72)	-0.570 [1]	0.585
TEGDMA	Result 24th day	20.20 ± 3.45	21.06 (16.39 - 24.58)	11.64 ± 9.70	7.17 (5.95 - 28.83)	20.000 [2]	0.151
TEGDMA	Result 30th day	21.01 ± 3.81	21.93 (17.21 - 26.16)	29.71 ± 0.82	29.63 (28.61 - 30.52)	-4.986 [1]	**0.006**

[1] Independent Samples t-Test, [2] Mann-Whitney U Test.

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Within-Group Time-Dependent Changes

Within-group analyses revealed significant time-dependent changes in monomer release. In the composite group, statistically significant differences over time were observed for Bis-GMA, UDMA, and TEGDMA ($p < 0.05$). In the compomer group, significant temporal differences were detected for all monomers ($p < 0.05$). In the giomer group, significant time-dependent differences were observed for Bis-GMA and UDMA, whereas TEGDMA was not detected at any time point (Table 9).

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Table 9. Comparison of time-dependent residual monomer release within material groups.

Monomer	Valiable	COMPOSITE		COMPOMER		GIOMER	
		Mean ± SD	Median (Min - Max)	Mean ± SD	Median (Min - Max)	Mean ±SD	Median (Min – Max)
BIS-GMA	Result 5th min	0.04 ± 0.02	0.04 (0.03 - 0.07) B	0.48 ± 0.04	0.47 (0.44 - 0.55) AB	13.05 ± 2.55 A	12.52 (10.24 - 16.42)
	Result 6th h	0.07 ± 0.04	0.06 (0.04 - 0.14) BC	0.98 ± 0.43	1.05 (0.30 - 1.47) B	30.18 ± 7.86 AB	31.48 (20.99 - 41.08)
	Result 24th h	0.06 ± 0.01	0.06 (0.04 - 0.07) BC	0.71 ± 0.36	0.63 (0.23 - 1.10) AB	34.16 ± 5.33 B	35.91 (28.53 - 40.98)
	Result 7th day	0.10 ± 0.02	0.10 (0.08 - 0.13) ABC	0.28 ± 0.03	0.28 (0.25 - 0.32) AB	33.78 ± 6.72 AB	36.19 (23.81 - 39.72)
	Result 15th day	4.58 ± 1.68	3.85 (3.40 - 7.46) AC	0.56 ± 0.30	0.57 (0.18 - 0.98) AB	47.95 ± 7.29 B	47.85 (40.68 - 56.20)
	Result 21st day	12.19 ± 4.20	9.44 (9.02 - 17.96) A	-0.15 ± 0.34	-0.31 (-0.37 - 0.45) A	70.46 ± 18.99 AB	80.33 (45.35 - 87.90)
	Result 24th day	3.48 ± 3.08	4.61 (0.18 - 6.78) AC	0.52 ± 0.40	0.57 (0.07 - 1.10) AB	43.63 ± 8.14 B	47.14 (30.69 - 50.09)
	Result 30th day	0.33 ± 0.07	0.35 (0.25 - 0.39) ABC	0.09 ± 0.34	-0.07 (-0.09 - 0.70) AB	44.75 ± 8.77 B	48.45 (31.09 - 51.59)
	Test ist.	33.000 [1]		15.930 [1]		17.340 [2]	
	p	**<0.001**		**0.026**		**0.001**	
UDMA	Result 5th min	0.57 ± 0.14	0.49 (0.47 - 0.79) B	6.75 ± 0.93	7.17 (5.17 - 7.41) B	12.49 ± 2.97	12.54 (9.07 - 16.86)
	Result 6th h	0.78 ± 0.23	0.68 (0.55 - 1.06) AB	19.21 ± 2.42	19.18 (16.68 - 22.38) AB	25.62 ± 7.81	26.03 (17.16 - 36.70)
	Result 24th h	0.53 ± 0.29	0.40 (0.35 - 1.05) B	12.97 ± 5.77	10.28 (8.05 - 22.15) B	25.14 ± 4.78	25.80 (19.34 - 31.88)
	Result 7th day	2.39 ± 3.28	0.98 (0.77 - 8.26) AB	38.93 ± 38.55	20.78 (13.40 - 106.45) AB	22.12 ± 4.84	24.26 (15.46 - 26.48)
	Result 15th day	1.17 ± 0.59	0.97 (0.66 - 2.13) AB	15.15 ± 5.09	13.02 (10.83 - 23.01) AB	27.86 ± 5.56	24.65 (23.15 - 36.48)
	Result 21st day	2.90 ± 3.21	1.57 (1.12 - 8.60) A	30.87 ± 3.39	30.75 (26.53 - 35.92) A	45.13 ± 12.90	49.13 (28.41 - 62.02)
	Result 24th day	1.10 ± 0.37	1.16 (0.62 - 1.63) AB	13.08 ± 5.98	10.28 (8.05 - 22.69) AB	23.42 ± 5.19	25.86 (16.20 - 28.34)
	Result 30th day	1.31 ± 0.28	1.26 (1.01 - 1.63) AB	18.51 ± 2.47	19.23 (14.48 - 21.09) AB	23.46 ± 5.57	24.69 (15.44 - 28.40)
	Test ist.	24.000 [1]		24.575 [1]		11.786 [2]	
	p	**0.001**		**0.001**		**0.002**	
TEGDMA	Result 5th min	6.18 ± 2.33 A	5.46 (4.10 - 10.18)	1.64 ± 0.20	1.57 (1.46 - 1.95) A		
	Result 6th h	12.78 ± 5.03 AB	10.61 (8.47 - 20.96)	8.29 ± 0.73	8.27 (7.65 - 9.49) ABC		
	Result 24th h	8.98 ± 2.68 A	7.87 (7.08 - 13.49)	7.22 ± 1.40	6.70 (5.95 - 9.59) AC		
	Result 7th day	13.57 ± 1.96 A	13.54 (11.63 - 16.57)	17.70 ± 1.42	18.40 (15.30 - 18.75) ABC		
	Result 15th day	14.50 ± 3.42 AB	13.21 (11.84 - 20.10)	18.61 ± 2.29	18.56 (16.33 - 22.06) ABC		
	Result 21st day	30.98 ± 14.67 AB	27.65 (16.32 - 55.69)	34.73 ± 0.93	35.17 (33.48 - 35.72) B		
	Result 24th day	20.20 ± 3.45 B	21.06 (16.39 - 24.58)	11.64 ± 9.70	7.17 (5.95 - 28.83) ABC		
	Result 30th. day	21.01 ± 3.81 B	21.93 (17.21 - 26.16)	29.71 ± 0.82	29.63 (28.61 - 30.52) BC		
	Test ist.	8.539 [2]		31.692 [1]			
	p	**0.021**		**<0.001**			

[1] Friedman Test, [2] Repeated Measures ANOVA, A-C: There is no significant difference between times with the same letter within each group.

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Discussion

Resin-based restorative materials are widely used in dentistry due to their favorable aesthetic and mechanical properties. However, biocompatibility remains a critical concern, particularly in pediatric dentistry, as incomplete polymerization may result in the release of residual monomers with potential cytotoxic effects. The degree of monomer-to-polymer conversion is influenced by material composition, curing conditions, and clinical application techniques.

During polymerization, reduced molecular mobility within the forming cross-linked polymer network may prevent complete monomer conversion, leading to the elution of residual monomers such as Bis-GMA, UDMA, and TEGDMA. These monomers have been associated with adverse biological effects, including cytotoxicity and inflammatory responses, highlighting the importance of optimizing polymerization protocols.

Numerous studies have investigated the release of residual monomers from resin-based materials and the factors influencing this process. However, data focusing specifically on materials commonly used in pediatric dentistry and evaluated under standardized polymerization conditions remain limited. Therefore, this study aimed to quantitatively assess residual monomer release following polymerization using HPLC at multiple time points. The standardized in vitro design, including the use of Teflon

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molds, controlled curing conditions, and statistically validated disc dimensions, enhances the reliability of the findings. Focusing on pediatric restorative materials is clinically relevant, as children may be more susceptible to the potential biological effects of residual monomers due to ongoing tissue development.

Manojlovic et al. (16) reported lower residual monomer release with QTH light sources than with LED units, attributing this to increased heat generation and enhanced free radical mobility. In contrast, Nalcaci et al. (17) observed greater monomer release with standard QTH devices and suggested that light intensity alone is insufficient for effective polymerization. In light of these conflicting findings, the present study focused not on comparing curing technologies but on assessing residual monomer release under standardized polymerization conditions. Accordingly, all materials were cured using an LED unit (Rainbow Light Curing) for 20 s, with a constant curing distance maintained.

Hansel et al. (18) investigated the biological effects of monomers used in composite materials and reported that TEGDMA may increase bacterial counts and that insufficient polymerization could lead to marginal leakage and pulp necrosis. **Although their study focused on biological outcomes rather than quantitative monomer release**, these findings highlight the clinical relevance of residual monomers released from restorative materials. In the present study, residual monomer release from one

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composite, one giomer, and one compomer material was quantitatively evaluated over time, and the composite material demonstrated lower residual monomer release compared with the other materials, which may be considered favorable in terms of potential biological risk.

Sideridou and Achilia (19) examined the elution of two light-cured composite resins in a 75% ethanol/water solution over six time periods and detected residual monomer release at all intervals, with the highest elution occurring during the initial period but continuing for up to one month. **Consistent with these findings**, the present study employed an 80% ethanol/water solution and detected monomer release at all eight measurement intervals, with elution persisting until the final day of observation.

Mazzaoui et al. (20) evaluated monomer elution from different composite resins and a glass ionomer cement in various ethanol/water solutions and reported that monomer release continued for up to three months, with the highest release occurring on the first day. In contrast, the present study assessed monomer release over a one-month period and demonstrated that release persisted throughout the observation period with varying levels over time.

Örtengren et al. (21) reported that Bis-GMA and UDMA were released in lower amounts than TEGDMA from composite materials. However, the present study demonstrated

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material-dependent release patterns, with higher Bis-GMA elution observed in the composite material and more pronounced TEGDMA release in the other materials. Archegas et al. (22) reported that Bis-GMA generally exhibited low elution, with most monomer release occurring within the first 7 days, whereas the present study identified the 21st day as the time point with the highest monomer release.

Nazar et al. (23) reported that increased layer thickness results in greater residual monomer release, with UDMA elution exceeding that of Bis-GMA. In the present study, standardized 2-mm-thick Teflon molds were used and specimen weights were controlled to minimize the effect of thickness, allowing monomer release to be attributed mainly to material-related factors.

Dundar et al. (24) demonstrated long-term monomer release from bulk-fill composites. In agreement with these findings, the Filtek bulk-fill composite in the present study showed the highest monomer release, particularly on day 21, with notable release also observed on days 7 and 15.

Conclusions

Although resin-based restorative materials are generally considered stable, degradation over time may lead to the release of residual monomers into the oral environment. Variations in monomer-to-polymer conversion can influence this release. To minimize residual monomer exposure and improve clinical outcomes,

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strategies such as optimized light-curing protocols, incremental layering, and appropriate material selection may be applied. Additionally, reducing the toxic effects of residual monomers requires strict adherence to manufacturer instructions, the use of isolation techniques (e.g., rubber dam), and removal of the oxygen inhibition layer by polishing after placement.

Considering the limitations of this in vitro study, dynamic oral conditions—including salivary flow, pH fluctuations, and enzymatic activity—could not be fully simulated, which may have affected the observed monomer release. Moreover, the ethanol–water solution used does not completely reflect the complexity of the oral environment. Finally, the 30-day evaluation period may not represent long-term monomer release, indicating the need for future longitudinal studies.

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