



Effect of Thermocycling and Bleaching on Hardness of two Resinous Dental Materials

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ABSTRACT

Aim: this in vitro study was conducted to evaluate the effect of thermocycling and bleaching on hardness of two composites. **Methods:** a total of 20 samples were used in this study divided into 2 groups (n=20) according to type of composite used Bis-GMA based and Bis-GMA free; each group was divided into 2 subgroups (n=5) bleached and non-bleached and all subgroups were subjected to thermocycling then vickers hardness test. **Results:** The results showed that there were significant differences between all groups and subgroups. The highest mean hardness number ($32.44\text{VHN} \pm 1.61$) was recorded in the non bleached non thermocycled BIS-GMA based composite. The lowest mean hardness number ($16.06\text{ VHN} \pm 0.93$) was recorded with the bleached and thermocycled BIS-GMA free composite. **Conclusion:** Conjugation of bleaching and thermocycling have marked decreasing effect on surface hardness of dental resin composite. Bis-GMA based composite can resist scratching after bleaching and thermocycling more than Bis-FMA free composite.

Keywords: Hardness, bleaching, thermocycling, Bis-GMA free, composite

1. Introduction

Dental caries remains one of the most prevalent chronic conditions across all age groups globally. Despite being largely preventable, it continues to pose a significant public health challenge. Resin-based composite materials have become the restoration of choice for both clinicians and patients, primarily due to their superior aesthetics, favorable handling characteristics, and excellent sculptability. Furthermore, composite formulations capable of releasing fluoride offer the additional therapeutic benefit of mitigating recurrent caries (Angelova *et al.*, 2023; Sun *et al.*, 2026).

Chemically, resin-based composites consist of an organic monomer matrix reinforced by inorganic filler particles. Bisphenol A-glycidyl methacrylate (Bis-GMA) represents the most widely utilized monomer in commercial formulations. However, concerns regarding its biocompatibility have emerged, as degradation products may exert estrogen-like endocrine-disrupting effects that pose potential health risks. Consequently, Bis-GMA-free dental composites have been developed as alternative formulations to eliminate these biocompatibility concerns while maintaining clinical performance (Cho *et al.*, 2022; Pratap *et al.*, 2019).

Surface hardness defined as a material's resistance to permanent indentation or scratching is a critical mechanical property directly tied to the degree of polymerization, overall mechanical integrity, and long-term clinical durability of resin restorations. Within the dynamic oral cavity, restorative materials are subjected to different masticatory forces and continuous aqueous degradation. Because fluid absorption and thermal stress compromise the mechanical stability of resin networks over time, artificial aging protocols such as thermocycling are essential for accurately predicting long-term material performance *in vitro* (. (Nair *et al.*, 2022; Samuel *et al.*, 2022).

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Vital tooth bleaching is frequently employed to improve the aesthetic appearance of discolored teeth and adjacent restorations. Bleaching agents rely on peroxide compounds that act as strong oxidizers, forming free radicals that cleave chromophores into low molecular weight molecules. While aesthetically beneficial, these oxidative reactions can alter the physical and surface properties of resin composites including microhardness, roughness of the surface, stability and translucency of color depending on the regimen utilized either in-office, home-applied, or combination protocols (Redwan *et al.*, 2025). The literature regarding the impact of bleaching agents on composite microhardness remains contradictory, with studies reporting both decreased and increased surface hardness following exposure.

Comparative evaluations of monomer compositions have demonstrated that Bis-GMA-based composites exhibit significantly higher microhardness values than organically modified ceramic (ORMOCER)-based formulations. Similarly, assessments comparing Bis-GMA-based materials to modified urethane dimethacrylate (UDMA)-based composites revealed lower hardness values in the UDMA-based groups (Comba *et al.*, 2020; Colombo *et al.*, 2020).

Conversely, another study reported comparable hardness values between Bis-GMA-based and urethane dimethacrylate (UDMA)-based composites when evaluated via Vickers microhardness testing (Ludovichetti *et al.*, 2022).

The clinical safety of Bis-GMA has come under increasing scrutiny due to its potential to release BPA derivatives. Bis-GMA exhibits marked cytotoxicity and genotoxicity, particularly in hepatic cells, where it compromises cell viability and morphology, suppresses cell proliferation, and triggers apoptotic pathways. Furthermore, low-dose exposure to BPA during critical developmental windows—such as gestation and early infancy has been linked to neurodevelopmental impairments and neurobehavioral disorders, including attention-deficit/hyperactivity disorder (ADHD), autism spectrum disorders, and depressive conditions (Costa and Cairrao 2024; Sendra *et al.*, 2023).

Therefore, this study aimed to evaluate the surface microhardness of Bis-GMA-based and Bis-GMA-free resin composites subjected to bleaching and thermocycling. The null hypothesis tested was that neither the composite formulation nor the combination of bleaching and thermocycling would significantly affect the surface microhardness of the tested materials.

2. Materials and Methods

2.1 Ethical Approval:

This study was approved by the Research Ethics Committee of the Faculty of Dentistry, Minia University (Protocol No. 118, Decision No. 1090; 2025).

2.2 Sample size calculation:

Sample size was calculated according to Abouelmagd and Basheer (2022); Fares, (2023) and Putri and Elline (2021). Using G power statistical power analysis program (version 3.1.9.4) for sample size determination. The total sample size for hardness test was sufficient to detect a medium effect size (d) = 1.77 with an actual power ($1-\beta$ error) of 0.8 (80%) and a significance level (α error) 0.05 (5%) for two-sided hypothesis test. the total sample size for hardness test was 20 samples.

2.3 Specimen Allocation and Experimental Groups

Twenty composite specimens were divided into two equal main groups ($n = 10$) according to the monomer composition:

- **Group I:** Bis-GMA-based resin composite
- **Group II:** Bis-GMA-free resin composite

Each main group was subsequently divided into two subgroups ($n = 5$):

- **Subgroup A:** Non-bleached
- **Subgroup B:** Bleached

All subgroups were subjected to thermal aging (5,000 thermal cycles). Surface microhardness was evaluated for all specimens at baseline (prior to thermocycling/bleaching) and following experimental treatment using the Vickers microhardness test.

2.4 Materials:

The materials brand name, scientific name, composition, manufacturer and lot number in (Table 1).

Table 1: Materials used in this study.

Scientific name	Brand name	Composition	Manufacturer	Lot Number
Bis-GMA based Bioactive S-PRG resin material (Figure 1)	Beautiful Bulk Flowable	The monomer matrix: Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, The filler: Surface pre-reacted glass ionomer filler based on fluoroboroaluminosilicate glass (72.5wt%), Initiator camphorquinone and pigments	Shofu Dental Corporation, Kyoto, Japan.	092268
Bis-GMA free dental composite resin (Figure 2)	Tetric N Flow Bulk Fill	The monomer matrix: Monomethacrylates and dimethacrylates (28wt%), The filler: Barium glass, ytterbium trifluoride and copolymers (71wt%) additives, initiator ivocerin, metal oxides, stabilizers and pigments (<1.0wt%)	Ivoclar Vivadent AG, Schaan, Liechtenstein.	Z05B59
Dual bleach system (Figure 3)	D.M WHITE plus	hydrogen peroxide 37%, carbamide peroxide 10%, benzoyl peroxide, and sodium perborate	D.M Trust, Minia, Egypt	



Fig. 1: Bis-GMA based dental composite.



Fig. 2: Bis-GMA free dental composite.



Fig. 3: Bleaching agent.

2.4. Specimen Fabrication, Bleaching, Thermocycling, and Microhardness Testing

2.4.1. Specimen Preparation

Composite specimens were fabricated using a custom polytetrafluoroethylene (Teflon) mold measuring 15mm in diameter and 1mm in thickness (Figure 4). The mold was positioned on a glass microscope slide, and the composite resin was packed directly from its light-shielded delivery syringe. A second glass slide was pressed over the uncured material to extrude excess monomer and ensure a flat, smooth top surface. Each specimen was light-polymerized for 20 seconds through the glass slide according to the manufacturer's recommendations using a light-emitting diode (LED) curing unit (Woodpecker Medical Instrument Co., Guilin, China) operating at an irradiance of 1200 mW/cm². Radiometric output was monitored and validated prior to each curing cycle using a digital radiometer (Woodpecker Medical Instrument Co., Guilin, China).

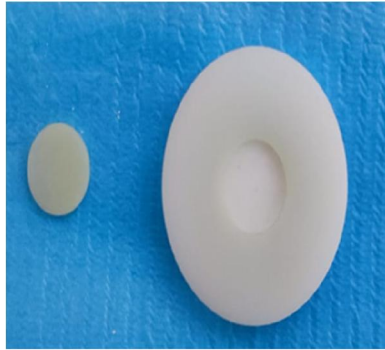


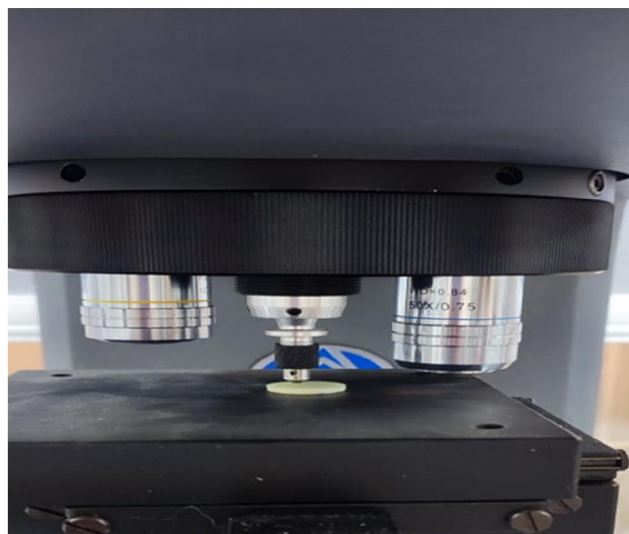
Fig. 4: Teflon mold and a prepared sample.

2.4.2. Surface Microhardness Assessment

Surface microhardness was evaluated using a Vickers microhardness tester (Wilson Tukon 1102; Buehler, Germany) figure 5 fitted with a square-based diamond pyramid indenter. A static load of 50 g was applied perpendicular to the specimen surface with a dwell time of 15 seconds. Indentation diagonals were measured automatically via the integrated optical microscope, and Vickers hardness numbers (VHN) were automatically calculated by the system software. Three indentations were placed on each surface, positioned at least 1mm apart to prevent stress field interference, and averaged to obtain a single representative value per specimen (figure 6).



A



B

Fig. 5: A; Vickers Hardness machine. B; Assessment of the hardness of a sample.

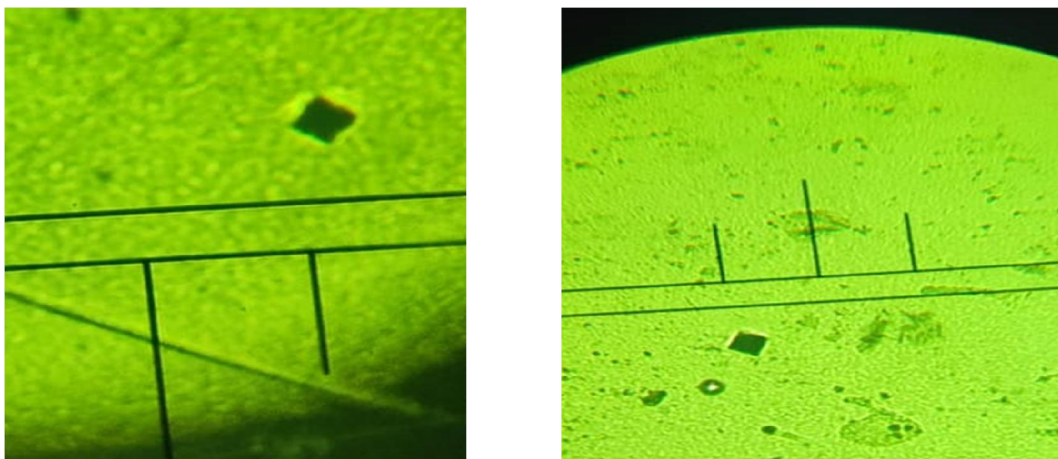


Fig.6: Micrograph shows the square indentations on the samples surfaces.

2.4.3. Thermal Aging Protocol

Specimens assigned to thermal aging were subjected to 5,000 thermal cycles in an automated thermocycling unit (SD Mechatronik, Westerham, Germany) (figure 7). The temperature range was maintained between 5°C and 55°C, with a dwell time of 30 seconds in each water bath and a transfer time of 5 text seconds between baths. Following thermocycling, microhardness testing was performed on the untreated surface using the standardized parameters described above.



Fig.7: Thermocycling machine.

2.4.4. Bleaching Protocol and Sequential Treatment

Specimens assigned to Subgroup B were treated with the bleaching agent (figure 8). The bleaching gel was dispensed and mixed with the provided catalyst powder using a micro-applicator brush for 20 seconds until a homogenous paste was obtained. A uniform layer 1-2 mm thick was applied to the specimen surface, gently agitated to ensure complete contact, left undisturbed for 30 minutes, and thoroughly rinsed under running deionized water. Microhardness measurements were then obtained from the bleached surface. For specimens designated for dual treatment, the opposing surface was bleached, subjected to the thermocycling protocol, and subsequently evaluated for surface microhardness.

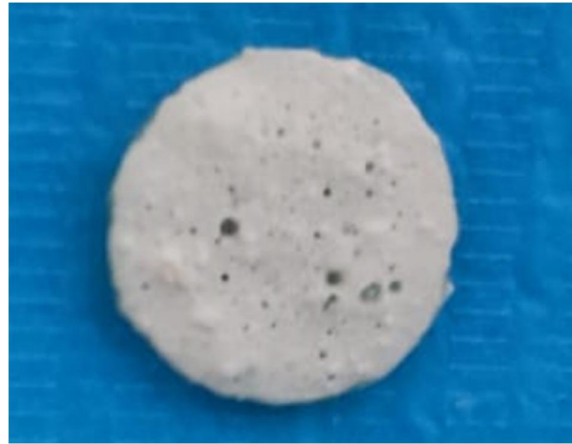


Fig. 8: Bleaching gel added to the surface of a specimen

2.5 Statistical analysis:

Data were collected, tabulated, and analyzed using IBM SPSS Statistics (Version 26.0; IBM Corp., Armonk, NY, USA). The normality of data distribution was assessed, confirming a parametric distribution. Continuous variables were summarized using descriptive statistics, expressed as mean and standard deviation (SD). Intergroup comparisons of surface microhardness values were analyzed using analysis of variance (ANOVA), followed by Fisher's Least Significant Difference (LSD) post-hoc test for pairwise comparisons. Paired (dependent-samples) *t*-tests were performed to compare baseline (pre-treatment) and post-treatment microhardness values within the same group. Correlations between experimental parameters were evaluated using Pearson's correlation coefficient (*r*). The level of statistical significance was set at $P \leq 0.05$.

3. Results

Mean values and standard deviations for surface microhardness across all experimental groups and subgroups are listed in Table 2. Statistically significant differences were observed among all tested groups and subgroups ($p < 0.05$).

The highest mean surface microhardness (32.44 ± 1.61 VHN) was recorded in the baseline (non-bleached, non-thermocycled) Bis-GMA-based composite control group. Conversely, the lowest mean surface microhardness (16.06 ± 0.93 VHN) was observed in the dual-treated (bleached and thermocycled) Bis-GMA-free composite subgroup (Figure 9).

Pearson's correlation analysis revealed a statistically significant, strong positive correlation ($r = 0.967 < 0.001$) between non-thermocycled and thermocycled group microhardness values (Figure 10).

Table 2: Mean and standard deviation of hardness number (VHN) between groups:

Subgroups	A (Non bleached)		P-value Non Vs Thermocycled	B (bleached)		P-value Non Vs Thermocycled
	Non-thermocycled	Thermocycled		Non-thermocycled	Thermocycled	
Group I	32.44±1.61	25.48±1.36	0.00*	27.92±3.26	22.34±2.38	0.00*
Group II	22.08±1.09	19.8±1.82	0.01*	18.66±1.71	16.06±0.93	0.01*
P- value I Vs II	0.00*	0.00*		0.00*	0.00*	

- $P \leq 0.05$ means significant difference
- The p value control VS bleached before thermocycling were 0.024 and 0.005 for groups I and II respectively
- The p value control VS bleached after thermocycling were 0.034 and 0.003 for groups I and II respectively

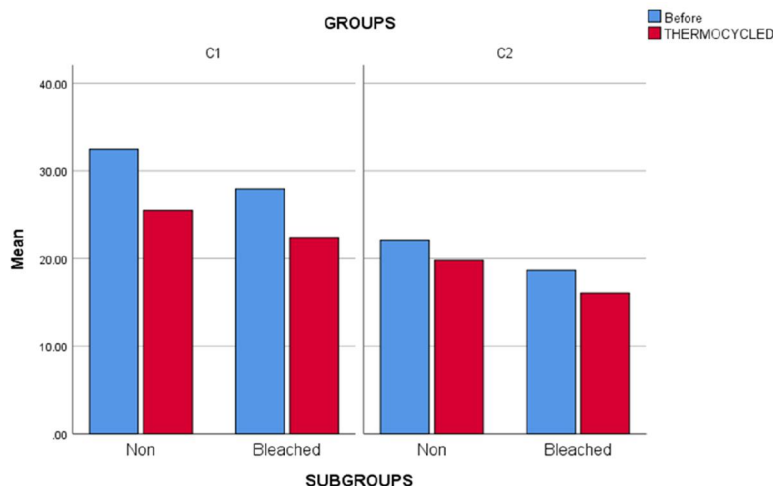


Fig.9: Bar chart represents the results of hardness test between different groups and subgroups.

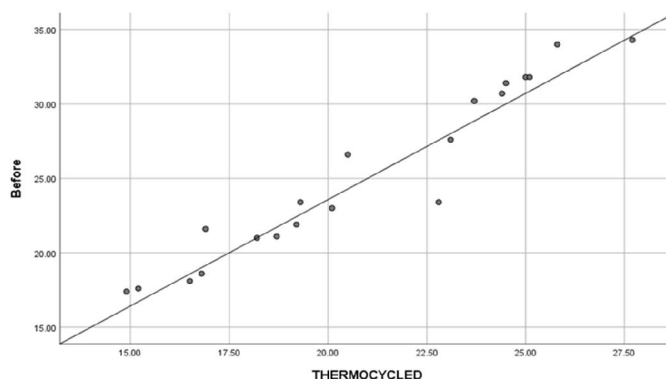


Fig.10: A curve shows correlation coefficients between non-thermocycled and thermocycled group.

4. Discussion

The surface microhardness of dental resin-based composites serves as a fundamental mechanical parameter reflecting cross-link density, degree of conversion, monomer architecture, and structural resistance to in-service wear and scratching. In the dynamic intraoral environment, restorative materials undergo continuous hydrothermal fluctuations and periodic chemical insults, such as exposure to oxidative bleaching gels (Ciccone-Nogueira *et al.*, 2007; Fernandes *et al.*, 2020). The present *in vitro* study evaluated the influence of thermocycling (5,000 thermal cycles) and high-concentration peroxide bleaching on the surface microhardness of a traditional Bis-GMA-based bulk-fill composite (*Beautifil Bulk Flowable*) and a Bis-GMA-free bulk-fill formulation (*Tetric N-Flow Bulk Fill*). Based on the statistical findings, both the monomer composition and the experimental treatments significantly altered the surface Vickers microhardness ($p < 0.05$). Consequently, the null hypothesis, that neither the composite monomer formulation nor the combination of bleaching and thermocycling would affect surface microhardness, was rejected.

At baseline (non-bleached, non-thermocycled), the Bis-GMA-based composite achieved a significantly higher mean microhardness (32.44 ± 1.61 VHN) compared to the Bis-GMA-free composite (22.08 ± 1.09 VHN) ($p = 0.00$). This mechanical superiority can be attributed to the rigid aromatic backbone of the Bis-GMA molecule, which contains rigid bisphenol-A core rings and strong hydrogen-bonding hydroxyl (OH) side groups that enhance structural rigidity and organic network cross-linking. Conversely, *Tetric N-Flow Bulk Fill* replaces Bis-GMA with alternative aliphatic dimethacrylates and monomethacrylates, which possess greater conformational flexibility and reduced inter-chain hydrogen bonding.

These baseline results agree with Melo *et al.* (2024), who reported that Bis-GMA-containing composite formulations maintained higher initial microhardness and greater structural stability than

Bis-GMA-free resins. The higher initial hardness observed in the Bis-GMA group also aligns with Bayraktar *et al.* (2021) who observed that rigid aromatic monomer systems consistently outclassed flexible aliphatic networks under Vickers microhardness testing.

On the other hand, the present baseline findings disagree with Fernandes *et al.* (2020), who found no statistically significant baseline microhardness differences between traditional Bis-GMA composite matrices and modified urethane dimethacrylate (UDMA)-based resins. This discrepancy likely stems from differences in filler loading fractions, filler particle size distribution, and curing light irradiance across different experimental designs.

Subjecting specimens to 5,000 thermal cycles resulted in a statistically significant reduction in surface microhardness across all tested subgroups ($p < 0.05$). For non-bleached samples, thermocycling caused microhardness to drop from 32.44 VHN to 25.48 VHN in Group I (Bis-GMA-based) and from 22.08 VHN to 19.80 VHN in Group II (Bis-GMA-free). Hydrothermal degradation occurs through two primary mechanisms: water sorption causing resin matrix plasticization, and mismatched thermal expansion coefficients between inorganic fillers and the polymer matrix. Water uptake expands the distance between polymer chains, disrupting non-covalent secondary bonds and accelerating silane coupling agent hydrolysis at the filler–matrix interface.

The microhardness reduction following thermal cycling agrees with Ramachandran *et al.* (2021) and Thomaidis *et al.* (2024), who demonstrated that 5000 thermal cycles reliably induce polymer swelling, surface micro-cracking, and soft-layer erosion in flowable and bulk-fill composites. Pearson's correlation analysis in this study further confirmed a strong positive correlation ($r = 0.967$, $p < 0.001$) between pre- and post-thermocycling states, reinforcing that initial surface integrity directly governs post-aging mechanical resilience.

Conversely, these findings disagree with Aldhafyan *et al.* (2025) and Lima *et al.* (2023) who reported that high-density bulk-fill composites exhibited negligible microhardness loss after thermocycling. They attributed this thermal stability to optimized silanization and lower resin matrix volumes, which minimized water ingress.

Chemical oxidation via the dual-bleach system (37% Hydrogen Peroxide and 10% Carbamide Peroxide) produced significant softening of composite surfaces ($p < 0.05$). Prior to thermocycling, bleaching reduced microhardness from 32.44VHN to 27.92 VHN in the Bis-GMA group ($p = 0.024$) and from 22.08 VHN to 18.66 VHN in the Bis-GMA-free group ($p = 0.005$). The oxidative action of free radicals (OH and OOH) released by high-concentration peroxides causes polymer chain scission, cleaves ester bonds within methacrylate networks, and promotes filler elution from the resin surface.

These results agree with Melo *et al.* (2024) who reported significant surface softening and increased surface roughness in hybrid and flowable resin composites following exposure to 35-40% hydrogen peroxide gels. The higher susceptibility of the Bis-GMA-free matrix to chemical oxidation. They noted that aliphatic chains without aromatic steric protection undergo accelerated matrix erosion when exposed to carbamide and hydrogen peroxide free radicals.

However, these observations disagree with Mohammadi *et al.* (2020), who concluded that short-term, high-concentration in-office bleaching did not significantly decrease the Vickers microhardness of microhybrid composite resins. They suggested that short exposure times prevent free radicals from penetrating deeply into well-polymerized matrix networks.

The lowest surface microhardness recorded in the entire study (16.06 ± 0.93 VHN) was observed in the dual-treated (bleached and thermocycled) Bis-GMA-free subgroup. This demonstrates a clear synergistic mechanical degradation when chemical oxidation is combined with thermal stress. Bleaching agents initially induce superficial polymer scission and micro-void formation. When these compromised surfaces undergo thermocycling, water ingress into the micro-voids is accelerated, promoting deep matrix plasticization, micro-fracturing, and particle debonding.

This dual-degradation pattern agrees with Özyay *et al.* 2022 and Seo *et al.* 2015 who verified that sequential exposure to peroxide bleaching and thermal fluctuations causes cumulative surface degradation that exceeds the effect of either treatment alone.

In contrast, these findings disagree with Boussès *et al.* (2021); Kalaivani *et al.* (2023); Nezir *et al.* (2025) who found no additive softening effect when thermocycling was combined with tooth whitening protocols on high-fill microhybrid composites, asserting that dense inorganic filler packing shields the matrix from combined hydrothermal and oxidative damage.

While Bis-GMA-free composites offer enhanced biocompatibility by eliminating bisphenol-A (BPA) precursors and potential endocrine-disrupting degradation products, clinicians must weigh this safety benefit against their lower baseline microhardness and heightened sensitivity to environmental aging. The marked hardness loss in Bis-GMA-free formulations after bleaching and thermocycling suggests an increased susceptibility to intraoral abrasive wear, surface staining, and bacterial plaque retention. Therefore, when placing Bis-GMA-free bulk-fill restorations in high stress-bearing areas or in patients undergoing frequent vital tooth bleaching, clinicians should exercise caution and recommend protective surface repolishing protocols (Schmalz and Widbiller 2022).

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