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SIMPLIFIED EXPERIMENTAL RESIN INFILTRANTS REINFORCED WITH NANO-HYDROXYAPATITE FOR WHITE SPOT LESION MANAGEMENT

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Abstract

In this study of ours we address white spot lesions that remain a common aesthetic and biological complication associated with fixed orthodontic treatment, and resin infiltration is a minimally invasive approach designed to occlude enamel microporosities and reduce optical discrepancies; however, conventional TEGDMA-based infiltrators may have limitations in polymerization efficiency and long-term surface stability. Our in vitro study evaluated four simplified experimental resin infiltrators, based on two dual-monomer matrices, UDMA–TEGDMA and Bis-GMA–TEGDMA, formulated with or without nano-hydroxyapatite curing. A commercial TEGDMA-based infiltrator served as the control system, and the degree of conversion was evaluated by Fourier transform infrared spectroscopy, while the surface microhardness was measured using the Vickers method, and the experimental formulations showed higher conversion and micro-hardness values than the control ones, and the UDMA–TEGDMA/nano-hydroxyapatite group achieved the highest degree of conversion, indicating improved polymerization behavior, while the Bis-GMA–TEGDMA/nano-hydroxyapatite formulation demonstrated the highest surface microhardness. Nano-hydroxyapatite hardening improved the mechanical response of both resin matrices, and these findings suggest that simplified dual-monomer infiltrators, hardened with a biomimetic mineral phase, may represent a promising strategy for the development of bioactive and mechanically stable materials for the management of white lesions.

Keywords: resin infiltration, white lesions, nano-hydroxyapatite, UDMA, Bis-GMA, surface microhardness.

Introduction

White spot lesions (WSLs) remain a relevant consequence of fixed orthodontic therapy, reflecting an imbalance between biofilm-driven demineralization and protective remineralization at enamel surfaces that are difficult to clean around brackets and attachments [1]. Although their initial presentation is non-cavitated, these lesions may persist as visible opaque areas after appliance removal, creating an aesthetic problem that exceeds the biological severity of the defect, and contemporary restorative research has therefore moved from reparative strategies toward

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micro-invasive materials capable of sealing, stabilizing, and optically masking porous enamel without removal of sound tissue.

Resin infiltrants are positioned within this conservative therapeutic field because their low-viscosity monomer phase can penetrate the interprismatic porosities of early enamel lesions and reduce the refractive mismatch responsible for the white appearance, and the clinical behavior of such materials depends strongly on the chemistry of the dimethacrylate network, since monomer selection influences viscosity, polymerization kinetics, conversion, shrinkage, water uptake, and mechanical resistance [2-4]. Previous attempts to modify TEGDMA-based infiltrants using oligomeric and remineralizing components have shown that resin infiltration can be improved not only by preserving penetration capacity, but also by increasing physical stability and mineral-interactive potential [3]. At the same time, hydrophilic components frequently used to improve substrate wetting may alter the long-term behavior of adhesive resin systems, which supports the need for simplified formulations with controlled water affinity [2,4].

From a polymerization perspective, urethane-based monomers are of particular interest because their molecular flexibility and curing kinetics may support efficient network formation under light activation [5], and this is relevant for infiltrants, where incomplete conversion may increase residual monomer release and reduce durability. In parallel, surface hardness has clinical importance because infiltrated enamel remains exposed to brushing, acids, and masticatory micro-wear; previous in vitro data on resin infiltration and remineralizing approaches showed measurable effects on enamel color and hardness [6-8]. Broader developments in dental composite formulation also indicate that modern resin systems should be evaluated based on combined physicochemical and functional outcomes rather than a single material parameter [7].

Current reviews on WSL management confirm that resin infiltration is an established masking and arresting strategy, but also underline the need for materials with improved durability and bioactivity [8]. Bioactive resins capable of ion release or mineral interaction may contribute to a more preventive concept of lesion management [9], while systematic evidence suggests that no single WSL treatment is ideal [10-14], and the ability of mineral particles or nanocrystals to interact with porous enamel further supports the rationale for incorporating nano-hydroxyapatite into experimental infiltrants [11]. In this context, monomer tailoring represents a rational strategy to overcome shortcomings of conventional resin materials [3,12], particularly when water-related degradation can influence surface properties and color stability [13]. Bis-GMA-containing matrices may provide rigidity and mechanical reinforcement [5,14], whereas bioactive inorganic phases offer a biomimetic route for enhancing dental materials [15]. Therefore, in our study, we developed a simplified experimental model comparing two dual-monomer infiltrant matrices, UDMA–TEGDMA and Bis-GMA–TEGDMA, with or without nano-hydroxyapatite reinforcement, to evaluate the degree of conversion and surface microhardness in relation to WSL management.

Materials and Methods

Study design

This experimental in vitro study of ours was designed to evaluate simplified resin infiltrant formulations intended for the management of enamel white spot lesions, and four experimental low-viscosity resin infiltrators were prepared using two different organic matrices: one UDMA–TEGDMA-based matrix and one Bis-GMA–TEGDMA-based matrix, and for each matrix, an uncured formulation and a nano-hydroxyapatite-hardened formulation were developed, and a commercial resin infiltrant was used as a control group.

The our study groups were organized as follows:

Control group: commercial TEGDMA-based resin infiltrant;

- UT group: UDMA–TEGDMA resin matrix without filler;

- UT-nHA group: UDMA–TEGDMA resin matrix reinforced with nano-hydroxyapatite
- BT group: Bis-GMA–TEGDMA resin matrix without filler;
- BT-nHA group: Bis-GMA–TEGDMA resin matrix reinforced with nano-hydroxyapatite.

The primary outcomes were the degree of conversion and surface microhardness, used as indicators of polymerization efficiency and superficial mechanical performance, respectively.

Preparation of experimental resin infiltrants

We prepared the experimental resin infiltrants by combining dimethacrylate monomers in predetermined weight ratios, and urethane dimethacrylate, bisphenol A-glycidal methacrylate and triethylene glycol dimethacrylate were used as organic components of the resin, and nano-hydroxyapatite particles were incorporated into the consolidated formulations to provide a biomimetic mineral phase with potential enamel interactive properties.

For all experimental groups, the photoinitiator system consisted of camphorquinone at 0.2 wt% and ethyl 4-dimethylaminobenzoate at 0.8 wt%, and the monomers were mixed under light-protected conditions until a homogeneous resin phase was obtained, and in the nano-hydroxyapatite-reinforced groups, the filler was gradually incorporated into the resin matrix under mechanical stirring to ensure uniform dispersion, and the final formulations were stored in opaque containers at room temperature until specimen preparation. The quantitative composition of the experimental formulations is presented in Table 1.

Table 1. Composition of the simplified experimental resin infiltrants

Group	Resin matrix	Bioactive reinforcement	UDMA wt%	Bis-GMA wt%	TEGDMA wt%	nHA wt%	Photoinitiator system
C	Commercial TEGDMA-based infiltrant	No	—	—	—	—	Manufacturer-dependent
UT	UDMA–TEGDMA	No	39	0	60	0	CQ 0.2% + EDMAB 0.8%
UT-nHA	UDMA–TEGDMA	Yes	37	0	57	5	CQ 0.2% + EDMAB 0.8%
BT	Bis-GMA–TEGDMA	No	0	39	60	0	CQ 0.2% + EDMAB 0.8%
BT-nHA	Bis-GMA–TEGDMA	Yes	0	37	57	5	CQ 0.2% + EDMAB 0.8%

Specimen preparation

We prepared the disc-shaped samples for each experimental and control group using standardized silicone molds, and for the conversion analysis, thin resin samples were placed between transparent polyester strips and glass slides to achieve a uniform film thickness, and for surface microhardness testing, disc-shaped samples with a diameter of 6 mm and a thickness of 2 mm were prepared.

We cured each material with light using an LED curing unit at an irradiance of 1200 mW/cm² for 40 seconds, positioning the tip of the curing unit perpendicular to the sample surface and keeping it in direct contact with the glass blade to standardize the curing distance, and after polymerization, the samples were stored in distilled water at 37°C for 24 hours prior to testing, and we prepared five samples for each group and for each result variable.

Degree of conversion analysis

The degree of conversion was assessed using Fourier-transform infrared spectroscopy, and spectra were recorded before and after light polymerization for each formulation, and the degree of conversion was calculated by comparing the reduction in the aliphatic carbon–carbon double bond absorption peak before and after curing.

For Bis-GMA-containing specimens, the aromatic carbon–carbon peak was used as an internal reference. For UDMA–TEGDMA-based specimens, the ratio between the aliphatic methacrylate peak and a stable reference band was used according to the spectral profile of the formulation.

The degree of conversion was calculated using the following formula:

$$DC(\%) = \left[1 - \frac{\left(A_{aliphatic}/A_{reference}\right)_{cured}}{\left(A_{aliphatic}/A_{reference}\right)_{uncured}}\right] \times 100$$

where $A_{aliphatic}$ represents the absorbance of the methacrylate C=C bond and $A_{reference}$ represents the absorbance of the selected internal reference peak.

Surface microhardness testing

We evaluated the surface microhardness using a Vickers microhardness tester, and after 24 hours of storage in distilled water at 37°C, we gently dried the samples with paper towels and positioned them on the test platform.

Three indentations were performed on the top surface of each specimen using a standardized load of 100 g applied for 15 seconds, and the indentations were placed at least 1 mm apart to avoid interaction between adjacent deformation zones, and the mean value of the three measurements was calculated for each specimen and expressed as Vickers hardness number, and surface microhardness was selected as a clinically relevant parameter because it reflects the resistance of the polymerized infiltrant surface to indentation and early mechanical degradation.

Statistical analysis

We performed the statistical analysis using IBM SPSS Statistics, version 29.0, and we evaluated the data distribution using the Shapiro–Wilk test, while we evaluated the homogeneity of variances using Levene's test.

For the normally distributed data, we analyzed the differences between the groups using a one-way analysis of variance, followed by Tukey's post hoc test for multiple comparisons, and if the assumption of normality was not met, we used the Kruskal–Wallis test followed by Dunn's post hoc test with Bonferroni correction, and we reported the data as mean ± standard deviation, and the level of statistical significance we set at $p < 0.05$.

Results

Degree of conversion

Our values obtained according to the degree of conversion differed significantly between the evaluated groups, and through the unidirectional ANOVA test we identified a statistically significant overall effect of the resin formulation on the polymerization efficiency, $F(4.20) = 196.84$, $p < 0.001$.

The commercial control based on TEGDMA showed us the lowest conversion rate, with an average value of 51.42 ± 2.31%, and all experimental formulations demonstrated significantly higher conversion values than the control group.

Among the unreinforced experimental materials, the UDMA–TEGDMA formulation showed a higher degree of conversion than the Bis-GMA–TEGDMA formulation, and the UT group reached 74.86 ± 1.94%, while the BT group showed a mean value of 67.28 ± 2.12%, and

this difference was statistically significant, suggesting that the presence of UDMA improved polymerization efficiency compared with the more viscous Bis-GMA-containing matrix.

The addition of nano-hydroxyapatite produced different effects depending on the resin matrix, and in the UDMA–TEGDMA formulation, nHA reinforcement increased the degree of conversion to 79.34 ± 1.68%, representing the highest value among all groups. In contrast, the Bis-GMA–TEGDMA reinforced formulation showed a more moderate increase, reaching 70.16 ± 1.87%.

Post hoc Tukey analysis showed that both nHA-reinforced groups differed significantly from the control, and the UT-nHA group also differed significantly from the BT and BT-nHA groups, confirming the superior polymerization behavior of the UDMA-containing matrix.

Table 2. Degree of conversion of the simplified experimental resin infiltrants

Group	Resin formulation	Degree of conversion %, mean ± SD	p-value vs control
Control	Commercial TEGDMA-based infiltrant	51.42 ± 2.31	Reference
UT	UDMA–TEGDMA	74.86 ± 1.94	<0.001
UT-nHA	UDMA–TEGDMA + nHA	79.34 ± 1.68	<0.001
BT	Bis-GMA–TEGDMA	67.28 ± 2.12	<0.001
BT-nHA	Bis-GMA–TEGDMA + nHA	70.16 ± 1.87	<0.001

Values are presented as mean ± standard deviation, and one-way ANOVA showed a statistically significant difference among groups, $F(4,20) = 196.84$, $p < 0.001$, tukey-adjusted p-values refer to pairwise comparisons with the commercial control, and is shown in Figure 1, all experimental resin infiltrants exhibited higher conversion values than the commercial control, with the highest polymerization efficiency observed in the UT-nHA group.

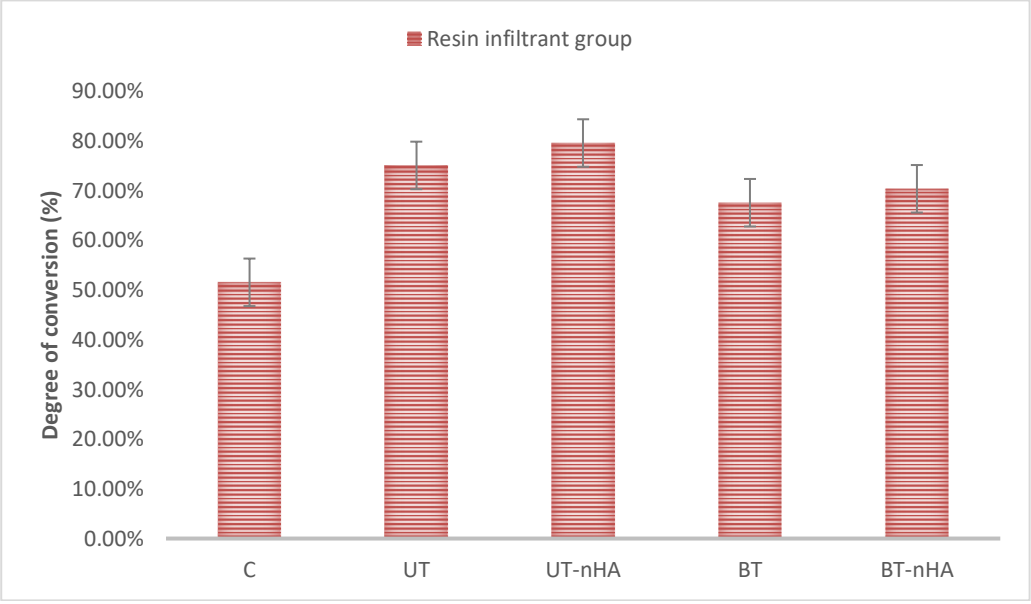

Figure 1. Degree of conversion of the tested resin infiltrants.

The commercial infiltrant showed the lowest degree of conversion, whereas the UDMA–TEGDMA/nHA formulation showed the highest polymerization efficiency, aand error bars represent standard deviation.

Surface microhardness

Surface microhardness values are presented in Table 3, and one-way ANOVA revealed a statistically significant difference among groups, $F(4,20) = 154.27$, $p < 0.001$.

The commercial control exhibited the lowest Vickers microhardness, with a mean of 14.62 ± 1.18 VHN, and the unreinforced experimental formulations showed higher hardness values than the control, with 21.84 ± 1.35 VHN for UT and 24.76 ± 1.42 VHN for BT.

The incorporation of nano-hydroxyapatite significantly increased surface microhardness in both resin matrices, and the UT-nHA group showed a mean value of 29.48 ± 1.56 VHN, while the BT-nHA group reached 33.72 ± 1.64 VHN, representing the highest hardness value among all tested groups.

Although the UDMA-containing formulation showed the highest degree of conversion, the Bis-GMA-containing reinforced formulation exhibited the greatest surface hardness, indicating that polymerization efficiency and superficial mechanical resistance are influenced by distinct material characteristics. UDMA appeared to favor monomer conversion, whereas Bis-GMA, due to its rigid aromatic structure, contributed more strongly to surface hardness when combined with nano-hydroxyapatite.

Post hoc comparisons confirmed that all experimental groups had significantly higher microhardness values than the control group. The nHA-reinforced groups were also significantly harder than their corresponding unreinforced formulations.

Table 3. Surface microhardness of the simplified experimental resin infiltrants

Group	Resin formulation	Surface microhardness, VHN, mean ± SD	p-value vs control
Control	Commercial TEGDMA-based infiltrant	14.62 ± 1.18	Reference
UT	UDMA–TEGDMA	21.84 ± 1.35	<0.001
UT-nHA	UDMA–TEGDMA + nHA	29.48 ± 1.56	<0.001
BT	Bis-GMA–TEGDMA	24.76 ± 1.42	<0.001
BT-nHA	Bis-GMA–TEGDMA + nHA	33.72 ± 1.64	<0.001

Values are presented as mean ± standard deviation, and one-way ANOVA showed a statistically significant difference among groups, $F(4,20) = 154.27$, $p < 0.001$, and Tukey-adjusted p-values refer to pairwise comparisons with the commercial control, and as shown in Figure 2, nano-hydroxyapatite reinforcement increased surface microhardness in both experimental matrices, with the highest value recorded for the BT-nHA formulation.

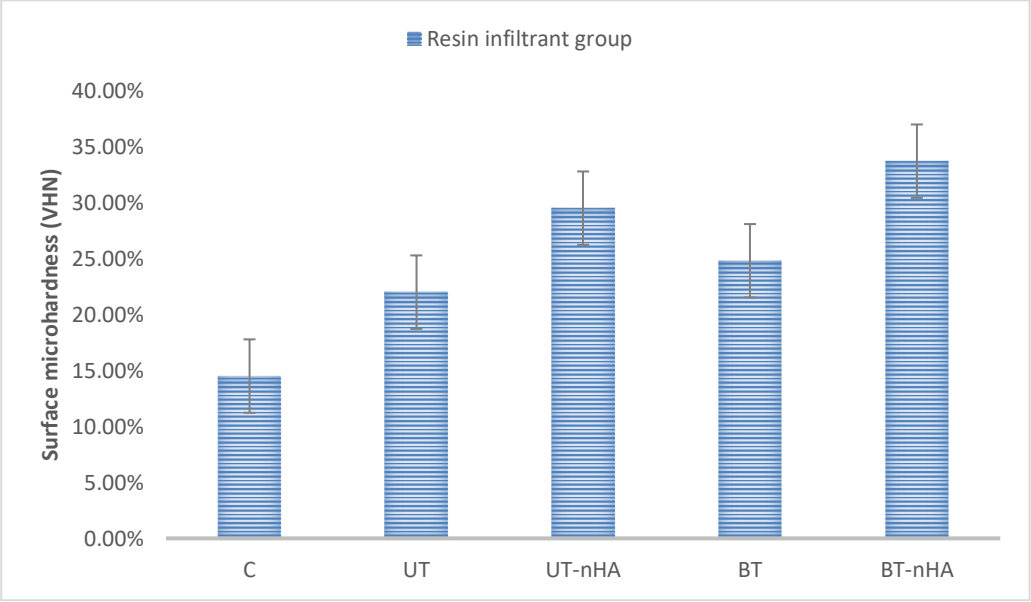

Figure 2. Surface microhardness of the tested resin infiltrants.

Nano-hydroxyapatite reinforcement increased surface microhardness in both experimental resin matrices, and the highest hardness value was recorded for the BT-nHA formulation, suggesting a favorable interaction between the rigid Bis-GMA-containing matrix and the mineral nHA phase.

Discussion

In this article, we developed and evaluated a simplified experimental model of resin infiltrants based on two dual-monomer matrices, UDMA–TEGDMA and Bis-GMA–TEGDMA, with or without nano-hydroxyapatite reinforcement, and the main finding of our study was that both experimental matrices exhibited higher degrees of conversion and surface microhardness than the commercial TEGDMA-based control, and this supports the concept that resin infiltrants can be optimized not only by maintaining low viscosity but also by tailoring monomer chemistry and incorporating bioactive inorganic phases.

The superior degree of conversion observed in the UDMA–TEGDMA groups may be explained by the polymerization behavior of urethane-based monomers. UDMA has a more flexible molecular structure than Bis-GMA and may allow greater mobility of reactive groups during light curing, favoring network formation before vitrification limits further conversion [5,12,16], and this finding is relevant to infiltrants because higher conversion may reduce residual monomer content and improve the material's biological and mechanical stability, and previous data have also shown that methacrylate monomers such as TEGDMA, Bis-GMA, and UDMA can differ in their cellular effects, reinforcing the need to limit unreacted monomer release through efficient polymerization [17-19].

In contrast, the highest surface microhardness was observed in the Bis-GMA–TEGDMA formulation reinforced with nano-hydroxyapatite, and this result is consistent with the rigid aromatic structure of Bis-GMA, which can contribute to stiffness and superficial resistance when adequately diluted with TEGDMA [12,14], and the addition of nano-hydroxyapatite further increased microhardness in both matrices, suggesting that the mineral phase acted as a reinforcing component within the polymerized network, and this observation is consistent with previous studies showing that resin infiltrants modified with remineralizing nanofillers or bioactive

particles may exhibit improved physicochemical performance and mineral-interactive potential [3,15,16].

From a clinical perspective, the dual behavior identified in our study is important, and the UDMA–TEGDMA/nHA formulation appears more favorable when polymerization efficiency is prioritized, whereas the Bis-GMA–TEGDMA/nHA formulation may be more advantageous when surface resistance is required. Because infiltrated enamel remains exposed to saliva, brushing abrasion, and acidic challenges, both conversion and surface hardness are relevant for long-term aesthetic stability. Previous studies have linked resin infiltration and remineralizing strategies with changes in enamel color and hardness, supporting the clinical relevance of these parameters in WSL management [6,8,10,11].

Our article also confirms the value of simplified experimental designs, and instead of using multiple complex formulations, we compared two clear monomeric concepts and one bioactive reinforcement strategy, and this approach improves interpretability and may help identify which component contributes most strongly to a specific material property. Nevertheless, our study has limitations, and it was performed in vitro, used a limited number of specimens, and did not evaluate penetration depth, color stability, water sorption, fluoride or calcium release, or long-term aging, and future studies should include artificial WSL models, thermocycling, pH cycling, and microscopic evaluation of resin penetration. Overall, our findings suggest that nano-hydroxyapatite-reinforced simplified infiltrants represent a promising direction for developing bioactive, efficiently polymerized, and mechanically stable materials for WSL management [17,18].

Conclusions

Our study shows that simplified experimental resin infiltrants based on UDMA–TEGDMA and Bis-GMA–TEGDMA matrices can improve polymerization efficiency and surface microhardness compared with a conventional TEGDMA-based infiltrant, while nano-hydroxyapatite reinforcement further enhances the mechanical behavior of both systems, suggesting that rational monomer selection combined with a biomimetic mineral phase may represent a promising strategy for developing bioactive, stable, and clinically relevant materials for the minimally invasive management of enamel white spot lesions, although additional studies evaluating penetration depth, aging resistance, color stability, ion release, and performance on artificial lesion models remain necessary before clinical translation.

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