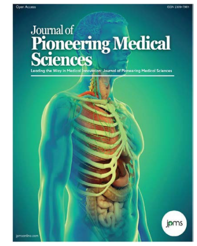
Journal of Pioneering Medical Sciences
Received: January 22, 2026 | Accepted: April 12, 2026 | Published: April 15, 2026
Volume 15, Issue S01, Pages 39-43

DOI https://doi.org/10.47310/jpms202515S0109



Comparative *In Vitro* Study of Color Stability of Nanofilled, Microhybrid and Nanohybrid Resin Composites Exposed to Commonly Consumed Beverages

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Abstract **Background:** Color stability is critical to the long-term esthetic success of resin composite restorations because visible discoloration often results in patient dissatisfaction, repolishing or restoration replacement. This in-vitro study compared the color stability of nanofilled, microhybrid and nanohybrid resin composites after immersion in commonly consumed beverages. **Methods:** Disc-shaped A2 specimens (10 mm×2 mm; final analyzed n = 150, with 10 specimens per subgroup) were prepared from Filtek Z350 XT, Filtek Z250 and Tetric N-Ceram. Specimens were randomly assigned to coffee, black tea, cola, raspberry juice or distilled water and stored at 37°C. Color was measured at baseline and after 7, 14 and 30 days using a spectrophotometer and the CIELAB system. This continuous immersion model represented accelerated staining and did not include thermocycling. **Results:** All beverage groups showed significantly greater color change than distilled water ($p<0.001$). Raspberry juice produced the highest discoloration in all materials (30-day DeltaE range: 8.94±0.87 to 12.67±1.23), followed by coffee, black tea and cola. Filtek Z350 XT showed the best overall color stability, Filtek Z250 showed the poorest performance and Tetric N-Ceram demonstrated intermediate behavior. By day 30, all beverage-exposed groups exceeded the clinical acceptability threshold (DeltaE >3.3); raspberry juice and coffee crossed this threshold earliest. **Conclusion:** Under this accelerated in-vitro model, commonly consumed beverages caused clinically unacceptable discoloration of all tested resin composites over time. Nanofilled composite demonstrated the greatest resistance to staining, whereas raspberry juice and coffee produced the most pronounced color change. These findings support material selection and patient dietary counseling but clinical extrapolation should be cautious because thermocycling, abrasion and intermittent exposure were not simulated.

Key Words Color Stability, Resin Composite, Nanofilled, Microhybrid, Nanohybrid, Beverages, Spectrophotometry, Discoloration

INTRODUCTION

Resin composites are widely used for direct esthetic restorations because they combine conservative tooth preparation with favorable handling characteristics and acceptable mechanical performance [1-3]. However, long-term color stability remains one of the most important determinants of patient satisfaction and restoration longevity, since visible discoloration may lead to repolishing, repair or replacement [4-6].

Discoloration of resin composites is multifactorial. Intrinsic factors include the composition of the resin matrix, the degree of conversion, oxidation of residual monomers and degradation at the resin-filler interface [4-10]. Extrinsic factors include adsorption and absorption of pigments from beverages, dietary agents and tobacco products [4,6-8]. Surface roughness and water sorption further modify the susceptibility of composites to staining [11-13].

The three materials evaluated in the present study differ in filler technology and matrix formulation. Filtek Z350 XT is a nanofilled composite designed to provide a smoother polishable surface, Filtek Z250 is a microhybrid composite with larger filler particles and Tetric N-Ceram is a nanohybrid composite intended to balance polish retention with mechanical strength [14-16]. These compositional differences may influence staining behavior by altering surface texture, water uptake and pigment retention [11,14,15].

Commonly consumed beverages such as coffee, tea, cola and berry-based juices can affect restorative color through different mechanisms. Coffee and tea contain chromogenic polyphenols, cola has low pH that may increase surface susceptibility to staining and raspberry juice contains anthocyanin pigments that can penetrate and discolor resin surfaces [17-22]. The clinical relevance of color change is commonly interpreted using DeltaE values, with DeltaE>3.3 generally considered clinically unacceptable [18,19].

Although many studies have evaluated composite discoloration, fewer investigations have compared contemporary nanofilled, microhybrid and nanohybrid materials under the same staining conditions and at multiple time points [23-30]. Therefore, this in-vitro study compared the color stability of three commercially available composites after immersion in coffee, black tea, cola, raspberry juice and distilled water over 30 days. The primary objective was to compare composite categories, whereas secondary objectives were to compare beverages and immersion time. The null hypothesis was that neither composite type nor immersion medium would significantly influence color stability.

METHODS

Study Design and Sample Preparation

This laboratory-based comparative in-vitro study evaluated color stability in three resin composite categories. Three commercially available A2-shade materials were selected: nanofilled composite (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA), microhybrid composite (Filtek Z250, 3M ESPE, St. Paul, MN, USA) and nanohybrid composite (Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein). G*Power software (version 3.1.9.7) was used for sample size estimation with an effect size of 0.40, alpha of 0.05 and power of 80%, giving a minimum of 10 specimens per subgroup. A total of 150 disc-shaped specimens were prepared, yielding 50 specimens per composite and 10 specimens for each immersion subgroup. Any discrepancy in earlier counts has been corrected here for transparency.

Specimen Fabrication and Polishing

Specimens were fabricated in a standardized Teflon mold (10 mm internal diameter x 2 mm thickness). Composite was inserted in a single increment, covered with a Mylar strip and glass slide and light-cured with an LED curing unit (Elipar DeepCure-S, 3M ESPE; 1470 mW/cm2) positioned 1 mm from the specimen surface. Defective specimens were discarded and replaced before randomization. Specimens were polished sequentially with Sof-Lex aluminum oxide discs from coarse to superfine grit under water cooling according to the manufacturer instructions, then stored in distilled water at 37°C for 24 hours before baseline measurement.

Immersion Media and Allocation

Five immersion media were used: coffee, black tea, cola, raspberry juice and distilled water. Coffee was prepared by dissolving 3.6 g instant coffee in 300 mL boiling distilled water. Black tea was prepared by immersing two tea bags in 300 mL boiling distilled water for 10 minutes. Cola was used as supplied. Raspberry juice was used as the staining medium provided in the original experimental protocol; however, pH and detailed compositional analysis were not measured and this has been acknowledged as a limitation. Specimens were allocated by computer-generated randomization to five subgroups (n = 10) within each composite category and stored in 20 mL of solution at 37°C in sealed containers. Solutions were renewed every 24 hours. The continuous immersion protocol represented an accelerated staining model rather than a simulation of routine daily beverage intake.

Color Measurement and DeltaE Calculation

Color was measured at baseline, day 7, day 14 and day 30 using a reflectance spectrophotometer (VITA Easyshade V, VITA Zahnfabrik, Bad Sackingen, Germany) against a standardized white background [18]. Before each reading, specimens were rinsed with distilled water for 30 seconds and gently dried with absorbent paper to standardize surface conditions. Three readings were taken at the center of each specimen and averaged for L*, a* and b* coordinates. Color difference was calculated using the standard formula: DeltaE = Square root of [(DeltaL*)^2+(Deltaa*)^2+(Deltab*)^2] [18]. DeltaE values were interpreted as imperceptible (<1.0), clinically acceptable (1.0-3.3) or clinically unacceptable (>3.3) [18,19].

Statistical Analysis

Data were analyzed using SPSS version 26.0 (IBM Corp., Armonk, NY, USA). Shapiro-Wilk and Levene tests were used to assess normality and homogeneity of variance. Two-way ANOVA examined the effects of composite type and immersion medium on color change and repeated-measures ANOVA evaluated time-related changes. Tukey HSD was used for post-hoc comparisons. A p-value <0.05 was considered statistically significant. Effect sizes and confidence intervals were not reported in the original dataset and are therefore identified as areas for improvement in future work.

Ethics, Funding and Reporting

As an in-vitro materials study, formal human-subject ethics approval was not required; however, the manuscript now clarifies this point for transparency. No external funding or manufacturer support was reported. The author declares no conflict of interest. A brief data availability statement has been added below.

RESULTS

All specimens demonstrated measurable color change after immersion, with beverage-exposed groups showing greater discoloration than the distilled water control. Overall, the 30 day ranking for color stability was Filtek Z350 XT (best), Tetric N-Ceram (intermediate) and Filtek Z250 (worst). Across beverages, the staining potential ranked raspberry juice>coffee>black tea>cola>distilled water.

At day 30, raspberry juice produced the highest mean DeltaE values in all three materials (8.94±0.87 for nanofilled, 12.67±1.23 for microhybrid and 11.34±1.08 for

Table 1: Mean Color Change (DeltaE±SD) Values at 30 Days by Composite Type and Immersion Medium

Immersion Medium	Nanofilled (Z350 XT)	Microhybrid (Z250)	Nanohybrid (N-Ceram)	p-value
Distilled Water	0.87±0.21	1.12±0.28	1.03±0.24	0.082
Cola	3.45±0.43	4.78±0.56	4.23±0.51	<0.001*
Black Tea	5.67±0.62	7.34±0.78	6.89±0.69	<0.001*
Coffee	6.23±0.56	9.45±0.92	8.12±0.84	<0.001*
Raspberry juice	8.94±0.87	12.67±1.23	11.34±1.08	<0.001*
p-value	<0.001*	<0.001*	<0.001*	-

Table 2: Mean Color Change (DeltaE±SD) Values by Composite Type and Immersion Time

Composite Type	Immersion Medium	Day 7	Day 14	Day 30	p-value (Time)
Nanofilled	Distilled Water	0.42±0.11	0.61±0.15	0.87±0.21	0.023*
	Cola	1.89±0.34	2.67±0.38	3.45±0.43	<0.001*
	Black Tea	2.78±0.41	4.12±0.52	5.67±0.62	<0.001*
	Coffee	3.12±0.45	4.89±0.51	6.23±0.56	<0.001*
	Raspberry juice	4.56±0.58	6.78±0.72	8.94±0.87	<0.001*
Microhybrid	Distilled Water	0.56±0.14	0.78±0.19	1.12±0.28	0.018*
	Cola	2.56±0.42	3.67±0.48	4.78±0.56	<0.001*
	Black Tea	3.89±0.54	5.56±0.63	7.34±0.78	<0.001*
	Coffee	4.67±0.62	7.12±0.78	9.45±0.92	<0.001*
	Raspberry juice	6.78±0.89	9.56±1.02	12.67±1.23	<0.001*
Nanohybrid	Distilled Water	0.48±0.12	0.72±0.18	1.03±0.24	0.021*
	Cola	2.23±0.38	3.23±0.45	4.23±0.51	<0.001*
	Black Tea	3.45±0.48	5.12±0.58	6.89±0.69	<0.001*
	Coffee	4.12±0.56	6.23±0.71	8.12±0.84	<0.001*
	Raspberry juice	5.89±0.74	8.45±0.91	11.34±1.08	<0.001*

Table 3: Percentage of Specimens Exceeding the Clinical Acceptability Threshold (DeltaE>3.3)

Composite Type	Immersion Medium	Day 7 (%)	Day 14 (%)	Day 30 (%)
Nanofilled	Distilled Water	0	0	0
	Cola	0	0	60
	Black Tea	0	80	100
	Coffee	0	100	100
	Raspberry juice	100	100	100
Microhybrid	Distilled Water	0	0	0
	Cola	0	70	100
	Black Tea	80	100	100
	Coffee	100	100	100
	Raspberry juice	100	100	100
Nanohybrid	Distilled Water	0	0	0
	Cola	0	40	90
	Black Tea	60	100	100
	Coffee	90	100	100
	Raspberry juice	100	100	100

nanohybrid), whereas cola produced the lowest beverage-related staining values. Distilled water remained within clinically acceptable limits throughout the study.

Color change increased significantly with time in all beverage groups. In the accelerated continuous immersion model, some groups exceeded the clinical acceptability threshold as early as day 7 or day 14, whereas by day 30 all beverage-exposed groups had become clinically unacceptable (Table 1-3).

Two-way ANOVA demonstrated significant main effects of composite type, immersion medium and time, as well as a significant interaction effect, indicating that the degree of staining depended on the combination of restorative material and beverage exposure. Post-hoc comparisons showed that the nanofilled composite had significantly lower DeltaE values than the microhybrid and nanohybrid materials in most staining media.

DISCUSSION

The present study showed that both the restorative material and the immersion medium significantly influenced color stability. Nanofilled composite demonstrated the greatest resistance to discoloration, whereas microhybrid composite showed the greatest susceptibility. These findings support the view that filler architecture, resin matrix composition and polish retention influence the uptake and retention of staining pigments [14,15,23,24,29].

Raspberry juice produced the greatest color change in all materials. A plausible explanation is the combined effect of highly chromogenic anthocyanin pigments and acidic conditions, which may facilitate pigment adsorption and absorption [17]. Coffee and black tea also caused substantial staining, consistent with their known chromogen content [20-22,30]. Cola produced the lowest beverage-related color change but even cola resulted in clinically unacceptable discoloration after prolonged continuous immersion [21,22,30].

The superior behavior of Filtek Z350 XT may be related to its nanofilled architecture, which is associated with improved surface smoothness and better polish retention [14,15]. In contrast, the higher staining values seen with Filtek Z250 may reflect the behavior of larger filler particles and a matrix more prone to water sorption and pigment retention [6,8,9,27]. Tetric N-Ceram showed intermediate performance, suggesting partial benefit from nanohybrid formulation without matching the behavior of the nanofilled composite [14,16].

The time-dependent increase in DeltaE across all beverages indicates progressive cumulative staining [19-26,28-30]. However, these findings should be interpreted within the limits of the study design. Continuous immersion at 37°C represents accelerated exposure rather than normal clinical consumption. In daily life, staining is influenced by intermittent beverage contact, saliva, pellicle formation, oral hygiene, brushing abrasion and thermal fluctuation [11,12,21,26]. Because thermocycling and mechanical aging were not included, the present model does not fully reproduce oral conditions [26].

This study provides clinically relevant guidance for restorative material selection and patient counseling. Patients with frequent intake of berry juices, coffee or tea may benefit from selection of materials with better stain resistance and from reinforcement of maintenance measures such as periodic polishing and dietary advice [4,21-23,26,29,30]. Nevertheless, the study did not evaluate surface roughness, stain removability or repolishing efficacy, so mechanistic interpretation remains limited [4,11,12].

The main limitations were the in-vitro disc model, absence of thermocycling and toothbrushing abrasion, lack of pH and chemical characterization of the raspberry juice, restriction to shade A2 and lack of surface roughness or microscopic analysis [12,18,26]. Future studies should combine intermittent immersion with artificial saliva, thermal cycling, abrasion and surface analysis to improve clinical simulation [11,21,26].

CONCLUSIONS

Within the limitations of this accelerated in-vitro immersion model, commonly consumed beverages adversely affected the color stability of all tested resin composites.

Raspberry juice showed the highest staining potential, followed by coffee, black tea and cola. Filtek Z350 XT demonstrated the best color stability, Filtek Z250 the worst and Tetric N-Ceram intermediate behavior.

By day 30, all beverage-exposed groups exceeded the clinical acceptability threshold for color change, whereas distilled water remained acceptable.

These findings support patient dietary counseling and material selection based on staining risk but clinical extrapolation should be cautious because thermocycling, abrasion and restoration anatomy were not simulated. Future studies should evaluate stain removal and more realistic oral aging models.

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