Evaluation of Color Stability and Micro-Hardness of New Composite Materials after Exposure to Different Beverages

Abstract

This study evaluated the color stability and microhardness of four resin composite materials after 30 days of immersion in red wine, tea, and coffee. Four materials were tested: G-aenial ACHORD (GC), Venus Pearl (Kulzer), Filtek Universal (3M), and Clearfil Majesty ES-2 (Kuraray). A total of 120 disc-shaped specimens (30 per material) were fabricated and exposed to the staining agents. Color stability was assessed using ΔE, calculated from CIE Lab values (L, a, b), and microhardness was measured using a Vickers microhardness tester. Descriptive statistics (mean ± sd) were calculated for each group. A one-way ANOVA was performed to assess significant differences in color change (ΔE) and microhardness between groups at T0 and T1. Post hoc Tukey's HSD test was used to identify specific group differences, with a significance level set at $p < 0.05$.bResults showed that red wine caused the most significant discoloration (highest ΔE), with significant differences between solutions ($p < 0.05$). Filtek Universal exhibited the highest ΔE values in both tea (ΔE = 14.46 ± 0.24) and coffee (ΔE = 14.11 ± 1.13), and the most pronounced discoloration in red wine compared to the other materials ($p < 0.05$). Clearfil Majesty ES-2 showed the lowest microhardness values in wine (Vickers hardness = 45.2 ± 2.3) and tea (Vickers hardness = 48.4 ± 3.1), while Venus Pearl exhibited the highest microhardness values (Vickers hardness = 54.8 ± 4.0 and 55.2 ± 3.8, respectively).
These findings indicate that Material C is more prone to color change, particularly in staining agents like wine, while Materials B and C demonstrate superior wear resistance. This study provides valuable insights for clinicians selecting composite materials based on aesthetic longevity and mechanical durability.

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INTRODUCTION

Composite resins are among the most commonly used direct restorative materials in modern dentistry, favored for their aesthetic qualities, strong adhesion to tooth structure, and minimal need for invasive tooth preparation (1,2). These materials are especially popular in both anterior and posterior regions due to their versatility and aesthetic appeal (3-5). Color stability is a key factor in the selection of composite materials for aesthetic restorations (6). Resin composites are prone to discoloration when exposed to common staining agents such as coffee, tea, wine, and tobacco smoke (7,8). The discoloration can result from a combination of internal and external factors, including the absorption of water, the accumulation of surface stains, and the chemical composition of the resin itself (9). Internal factors, such as oxidation of the monomers or catalysts, also contribute to changes in color (10). The discoloration of dental restorations is often a significant reason for their replacement, especially in visible, esthetically sensitive areas (11,12). The ability of a resin to maintain color stability over time is critical to the long-term success of restorations. In particular, the ability to match and maintain the appearance of natural teeth is essential for patient satisfaction (13). While both internal and external factors contribute to color changes, advancements such as the exclusion of benzoyl peroxide from light-cure formulations have reduced some forms of intrinsic discoloration (14). However, more research is needed to fully understand the in vitro effects of various staining agents, including coffee, tea, and wine, on the color stability of modern resin composites.

This study aims to evaluate the color stability and microhardness of four different composite materials, providing valuable insight into their long-term performance and suitability for esthetic restorations. Alongside color stability, the microhardness of composite resins plays a crucial role in determining their long-term clinical performance, particularly their resistance to wear and surface degradation. Microhardness refers to a material's ability to resist indentation and surface deformation under stress, which is directly related to its durability and resistance to abrasion in the oral environment. In the context of dental restorations, higher microhardness values typically correlate with greater wear resistance, an essential property for materials exposed to the mechanical stresses of chewing and grinding (15,16).

The microhardness of composite resins is influenced by several factors, including the type and amount of filler particles, the resin matrix, and the degree of polymerization achieved during curing (17). Composites with higher filler content generally exhibit increased microhardness due to the superior wear resistance of the inorganic fillers compared to the organic resin matrix (18,19). Conversely, composites with lower filler content or those that suffer from incomplete polymerization may exhibit poorer hardness values, leading to an increased likelihood of surface wear, roughness, and subsequent discoloration over time (19).

Recent advancements, such as the development of nano-composites and hybrid composites, have led to improvements in both microhardness and color stability. Nanofillers have been shown to enhance surface smoothness and abrasion resistance, thus contributing to both the aesthetic longevity and mechanical integrity of restorations (20). Furthermore, the surface hardness of a resin composite can influence its resistance to staining, as softer surfaces are more susceptible to the absorption of staining agents and bacterial plaque (21,22).

As with color stability, achieving an optimal balance between hardness, aesthetic qualities, and ease of manipulation is essential for the success of composite resins in clinical practice. Therefore, understanding the microhardness characteristics of different composite materials is critical when selecting the most appropriate material for both functional and esthetic restorations. This study aims to evaluate the color stability and microhardness of four different composite materials, providing valuable insight into their long-term performance and suitability for esthetic restorations. The null hypothesis were as follows:

- There is not any statistically significant difference between the 4 tested composite regarding color stability after 60 days of immersion in wine, tea or coffee.
- There is not any statistically significant difference between the 4 tested composite regarding microhardness after 60 days of immersion in wine, tea or coffee.

MATERIALS AND METHODS

Samples preparation

Four types of resin composite materials were used in this study, each representing a different class of composite based on their composition and intended clinical use. The specific materials tested are reported in table 1.

Specimens were prepared by fabricating 30 disc-shaped samples (10 mm in diameter, 2 mm in thickness) for each resin composite. The preparation protocol for each composite was as follows. Round molds with a diameter of 10 mm and height of 2 mm were fabricated using heavy body silicone (3M ESPE, Minnesota, USA), ensuring uniform size and shape for all specimens. A bulk-fill technique was used to fill each mold with the corresponding resin composite. The material was gently pressed into the mold with a cement spatula to avoid air entrapment and excess material. After insertion, each specimen was light-cured for 20 seconds using a Mectron LED light-curing unit (Carasco, Italy) with an intensity of 1500 mW/cm^2, ensuring consistent light intensity

Restorative material	Organic content	Inorganic content	Filler rate vol %
G-aenial ACHORD, GC (Tokyo-Japan)	UDMA, dimethacrylates	Pre-polymerized fillers, silica, strontium and lan- thanide fluoride	63
Venus Pearl, Kulzer (Weheheim-Germany)	TCD-DI-HEA, UDMA	Ba-Al-F-glass, prepolymerized filler, SiO2 nanofiller	58
Filtek universal, 3M (Minnesota, USA)	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA	Silica, zirconium	63.3
Clearfil Majesty ES-2, Kuraray (Tokyo-Japan)	Bis-GMA, hydrophobic aromatic dimethacrylates	Pre-polymerized filler, silanated barium glass	66

Tab. 1 Materials tested in the present study.

across all groups, monitored by a digital radiometer. After curing, specimens were polished using 3M ESPE Soflex polishing discs and the Dentsply Enhance PoGo polishing system (Milford, USA), followed by a goat hairbrush to ensure a smooth, standardized surface.
After polishing, a total of 120 disc-shaped specimens were prepared and divided into four groups based on the resin type:

- Group A: G-aenial ACHORD (30 discs)
- Group B: Venus Pearl (30 discs)
- Group C: Filtek Universal (30 discs)
- Group D: Clearfil Majesty ES-2 (30 discs)

Color stability test

To evaluate the color stability of the composites, each group was exposed to one of three commonly consumed beverages: red wine, tea, and coffee. The specimens were randomly assigned to one of the three beverage groups as follows:

- Group 1 (Red Wine): Submerged for 30 days in red wine (Ascheri BAROLO 2017, Piedmont, Italy) with a pH of approximately 3.0 and an alcohol content of 14%.
- Group 2 (Tea): Submerged for 30 days in tea made by simmering 15 g of loose tea in 1 L of boiling water for 5 minutes.
- Group 3 (Coffee): Submerged for 30 days in coffee (Lavazza Espresso, Torino, Italy), made by infusing 15 g of ground coffee in 1 L of boiling water for 3 minutes.

The specimens were placed in sterilized ice molds, with each mold holding one group of 10 discs per beverage. All samples were kept at 37°C in an incubator for the entire duration of the staining period, with the beverages being replaced every 2 weeks to maintain the staining efficacy. Color stability was assessed by measuring the color change (ΔE) of each specimen at baseline (T0) and after 60 days of immersion (T1). Color measurements were recorded using a VITA Easyshade spectrophotometer (VITA Zahnfabrik, Germany), which uses the CIE Lab color system* to quantify color shifts.
The total color change (ΔE) was calculated using the formula reported in figure 1.

Microhardness test

Microhardness was evaluated before (T0) and after 60 days of staining (T1) using a Vickers microhardness tester (Qualitest QV-1000, Florida, USA). Each specimen was tested at three random locations on its surface, with the following procedure:

- *Indentation Process:* A diamond pyramid indenter was used to apply a 5 N load for 20 seconds.
- *Measurement:* The mean of three indentations was calculated to represent the surface microhardness for each specimen.
- *Pre- and Post-Staining Testing:* Microhardness values were recorded both at baseline (T0) and after the staining period (T1), to compare the effects of staining on the hardness of each composite material.

Data from both the color change and microhardness tests were analyzed using SPSS (software Version 26, IBM, Armonk, NY, USA). Descriptive statistics (mean ± standard deviation) were calculated for each material group. A one-way ANOVA was used to determine any significant differences between the groups in

$$\Delta E=(L1-L0)2+(a1-a0)2+(b1-b0)2\Delta E=(L1-L0)2+(a1-a0)2+(b1-b0)2$$

L represents lightness (black to white),
a represents the red-green axis,
b represents the yellow-blue axis.

L0, a0, b0 are the baseline color values, and
L1, a1, b1 are the color values at the 00-day measurement point (T1)

Fig. 1

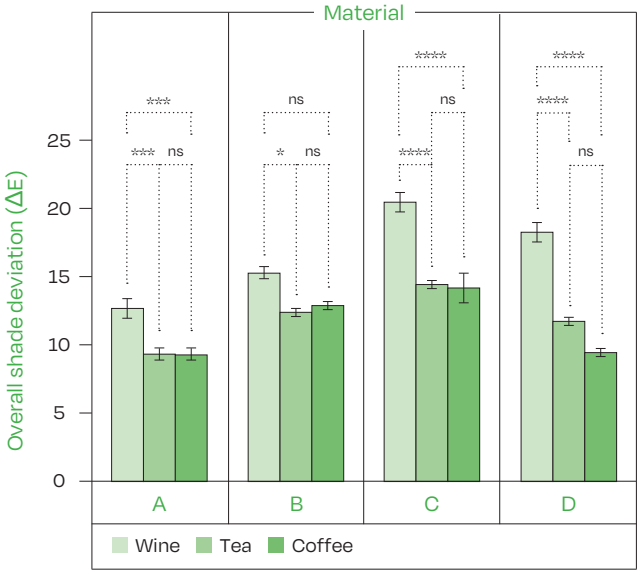

Fig. 2 Graph showing ΔE after immersion in different solutions for 2 months for four tested materials. Data are presented as the mean ± SE (n=10). *P< 0.05, **P< 0.01, ***P< 0.001, ****P< 0.0001.

Material	Wine	Tea	Coffee
A	12.74 ± 0.61a	9.25 ± 0.44 a	9.22 ± 0.5 a
B	15.25 ± 0.39 a	12.37 ± 0.20 b	12.98 ± 0.17 b
C	20.44 ± 0.73 b	14.46 ± 0.24bc	14.11 ± 1.13 bc
D	18.44 ± 0.67 b	11.64 ± 0.41b	9.49 ± 0.29 a

Tab. 2 Means and standard errors of overall shade deviation (ΔE) after immersion in different solutions for 2 months.

terms of color change (ΔE) and microhardness values at T0 and T1. If significant differences were found, post hoc Tukey's HSD test was applied to determine which specific groups differed from each other. The significance level was set at $p < 0.05$.

RESULTS

Color Change (ΔE)

Results from mixed model repeated measure ANOVA showed that time and solutions statistically affect each material's overall shade deviation (ΔE). Across all groups, wine was the solution that caused the most discoloration as shown in Figure 2. The wine was significantly different from tea and coffee in other materials in the overall shade deviation (ΔE), deviation of lightness (ΔL), and deviation of hue (Δh). However, there was no discernible distinction between coffee and tea. Multiple pairwise comparisons of the material effect revealed that material C related to high ΔE values in the tea and coffee solution as shown in Table 2. (ΔE Tea = 14.46 ± 0.24; ΔE Coffee= 14.11 ± 1.13) Material A registered statistically lower ΔE values after immersion in the 3 different solutions compared to the other materials. Material C and D registered statistically higher values after immersion in wine, while materials B and C registered statistically higher values after immersion in Coffee.

Microhardness of the Surfaces

Results that were taken from mixed model 2-way

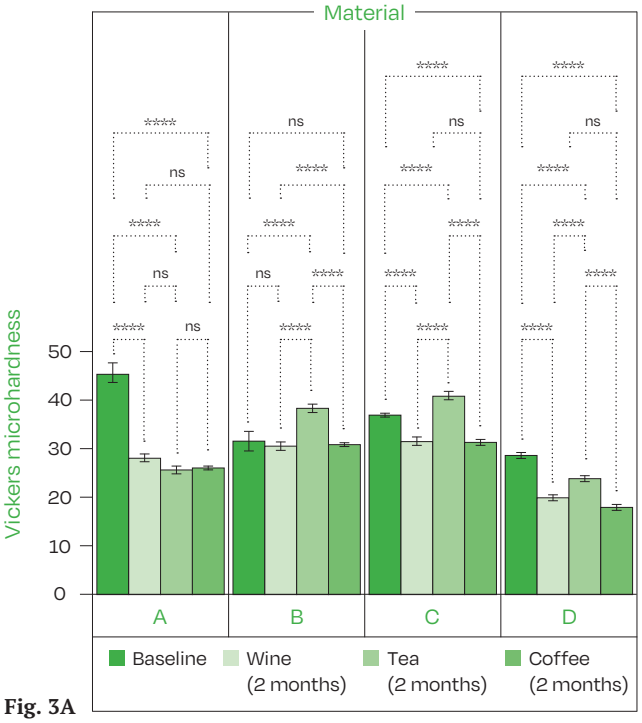

Fig. 3A

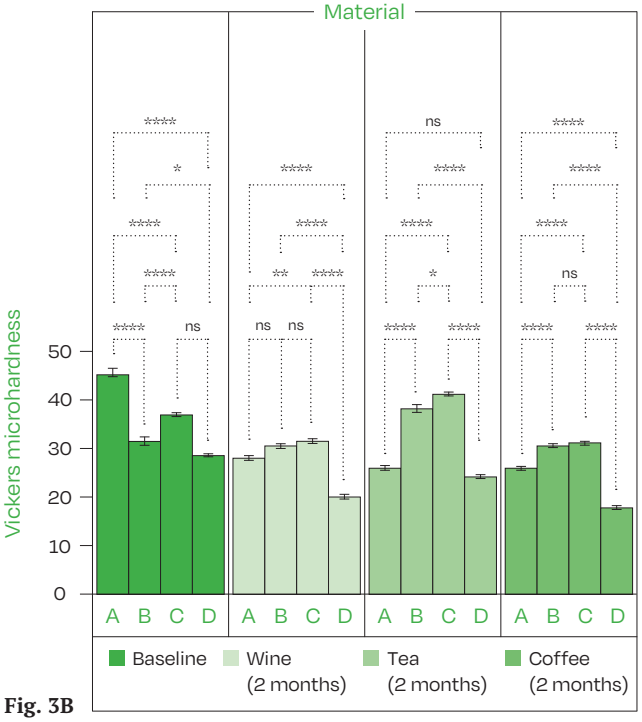

Fig. 3B

Fig. 3A, 3B Vickers microhardness values of different materials at baseline and after immersion in different solutions for 2 months. Data are presented as the mean ± SE (n=10). *P< 0.05, **P< 0.01, ***P< 0.001, ****P< 0.0001.

Material	Baseline	Tea	Coffee	Wine
A	45.46 ± 1.08 a	28.46 ± 0.19 b	26.22 ± 0.31 c	26.43 ± 0.18 b
B	31.79 ± 0.63 c	30.68 ± 0.68 a	38.36 ± 0.73 b	30.99 ± 0.28 a
C	37.04 ± 0.32 b	31.67 ± 0.63 a	41.05 ± 0.52 a	31.42 ± 0.31a
D	28.89 ± 0.26 c	20.43 ± 0.17 c	24.37 ± 0.45 c	18.43 ± 0.19 bc

Tab. 3 Means and standard errors of Vickers microhardness values of different materials at baseline and after immersion in different solutions for 2 months.

ANOVA showed that "material" and "solution" in the microhardness test had a significant effect ($P < 0.001$) (fig. 3A, 3B). Materials showed statistically different microhardness values at baseline. Materials B and C obtained statistically significant higher values of microhardness compared to materials A and D after immersion in all the different solutions as shown in Table 3.

DISCUSSION

In this study, we assessed the color stability and microhardness of four composite materials exposed to common staining beverages—wine, tea, and coffee.
From the results obtained in the present study both the null hypothesis are rejected. In fact,
G-aenial ACHORD, GC (Tokyo, Japan) registered statistically lower ΔE values after immersion in the 3 different solutions compared to the other materials. While Venus Pearl, Kulzer (Wehrheim, Germany) and Filtek universal, 3M (Minnesota, USA) obtained statistically significant higher values of microhardness compared to G-aenial ACHORD, GC (Tokyo, Japan) and Clearfil Majesty ES-2, Kuraray (Tokyo, Japan).
Color stability is a critical factor in ensuring long-term aesthetic success in restorative materials. A composite restoration must match the natural tooth color not only at placement but also throughout the lifetime of the restoration (23). In this study, immersion of composite materials in wine, tea, and coffee for two months led to significant discoloration across all materials tested. Wine was identified as the solution that caused the most pronounced discoloration (ΔE), consistent with previous studies showing that red wine contributes significantly to staining due to its acidic nature and high chromogenic potential (24). Filtek universal, 3M (Minnesota, USA) exhibited the highest ΔE values in both tea (ΔE Tea = 14.46 ± 0.24) and coffee (ΔE Coffee = 14.11 ± 1.13), suggesting that certain composite materials are more prone to discoloration in these beverages. This finding supports previous research indicating that some materials are more susceptible to staining based on their chemical structure and matrix composition (25,26). In fact, 3M Filtek Universal contains zirconium particles that exhibited greater values in the microhardness test, which may be attributable to the zirconia filler. Additionally, the distribution of the filler or its dimensions could influence the findings obtained for the hardness (27,28). Interestingly, tea and coffee did not result in significantly different color changes, which aligns with earlier studies suggesting these beverages cause less discoloration than wine. However, Filtek universal, 3M (Minnesota, USA) and Clearfil Majesty ES-2, Kuraray (Tokyo-Japan) showed significantly greater chroma (ΔC) shifts in wine, suggesting that acidic environments not only induce overall color shifts but also affect the intensity of the color, particularly in composites with certain filler compositions. Wine's acidic nature, combined with its chromogenic potential, likely exacerbates staining by degrading the composite surface and allowing pigments to penetrate the material (29). Beyond aesthetics, the mechanical properties of restorative materials, particularly microhardness, are crucial for the long-term durability of dental restorations. Microhardness reflects a material's resistance to wear, scratching, and indentation, which is essential for maintaining both functional and aesthetic integrity over time (24,30). Our study showed significant reductions in microhardness values for all materials after immersion in wine, tea, and coffee solutions. Material D exhibited the lowest microhardness values across all solutions, particularly in tea, suggesting it is more prone to degradation in acidic environments, which could reduce its wear resistance over time. This result is in line with previous studies showing that acidic beverages can lead to a decrease in composite hardness due to erosion of the resin matrix and dissolution of fillers (31,32).
Conversely, Filtek universal, 3M (Minnesota, USA) and VenusPearl, Kulzer (Wehrheim, Germany) exhibited higher microhardness values in both wine and coffee, indicating better retention of mechanical strength and surface integrity. Despite showing the highest ΔE values, Filtek universal, 3M (Minnesota, USA) demonstrated superior mechanical properties in these beverages, particularly in acidic environments. This suggests that filler content and particle distribution play a key role in both color stability and microhardness (33,34,35).
The significant reduction in microhardness across all materials after exposure to staining solutions highlights the potential for long-term degradation of resin composites when exposed to common oral staining agents. These findings underscore the need to consider both aesthetic performance and functional durability when selecting composite materials for patients at risk

of exposure to acidic or staining beverages.
From a clinical perspective, the findings suggest that Filtek universal, 3M (Minnesota, USA), which exhibited the highest ΔE values in both tea and coffee, may not be the ideal choice for patients who consume large amounts of these beverages. G-aenial ACHORD, GC (Tokyo, Japan) performed statistically better than other materials regarding color changes and can be considered preferable for use in anterior regions. In contrast, Venus Pearl, Kulzer (Wehrheim, Germany) and Filtek universal, 3M (Minnesota, USA), which demonstrated higher microhardness values in wine, may be better suited for patients exposed to acidic beverages or posterior areas, as they retained both mechanical properties better over time. Given that microhardness is closely linked to wear resistance and the longevity of restorations, clinicians should prioritize materials that maintain both color stability and mechanical integrity in the presence of common dietary agents.
These findings can be used to guide material selection for different dental and composite applications, where resistance to discoloration and durability (microhardness) are critical factors. Future studies could further investigate the long-term effects of these solutions on composite materials and their potential for use in clinical practice.

CONCLUSION

Within the limitations of this in vitro study, it can be concluded:

- Wine caused the most discoloration (highest ΔE) across all materials.
- G-aenial ACHORD, GC (Tokyo, Japan) registered statistically lower ΔE values after immersion in the 3 different solutions compared to the other materials.
- Venus Pearl, Kulzer (Wehrheim, Germany) and Filtek universal, 3M (Minnesota, USA) obtained statistically significant higher values. of microhardness compared to G-aenial ACHORD and Clearfil Majesty ES-2, Kuraray (Tokyo, Japan) after immersion in all the different solutions.

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