



Article

Effects of Thermocycling and Staining on Optical Behavior of Light-Cured and Digital Light-Processed Dental Composites

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Abstract

This study evaluated the effects of thermocycling and staining on the optical behavior, chemical stability, and surface morphology of three commercial dental composites manufactured using different polymerization protocols: a light-cured composite (Omnichroma, OMNI) and two digital light-processed (DLP) 3D-printed composites intended for permanent (SprintRay CROWN, SPRINT) and temporary (GC Temp PRINT, TEMP) clinical applications. Disc-shaped specimens were subjected to thermocycling (0, 5000, 10,000, and 30,000 cycles) and immersion in coffee, Coca-Cola, or red wine for 1 and 7 days. Color change (ΔE_{00}) and translucency parameter (TP) were determined spectrophotometrically, while Fourier-transform infrared spectroscopy (FTIR) and atomic force microscopy (AFM) were employed to assess the polymerization efficiency (expressed as the relative conversion index) and surface morphology, respectively. Thermocycling induced material-dependent changes in color stability and translucency. After 30,000 cycles, ΔE_{00} values reached 2.77 ± 0.89 for OMNI, 0.74 ± 0.34 for SPRINT, and 3.30 ± 0.89 for TEMP. SPRINT maintained the highest TP values throughout the aging protocol, ranging from 26.09 ± 0.33 to 23.81 ± 1.93 , whereas OMNI and TEMP exhibited lower TP values. The most favorable optical performance and the highest resistance to beverage-induced coloration were detected for SPRINT, whereas TEMP showed the greatest susceptibility to aging- and staining-induced optical degradation. Red wine and coffee produced substantially greater color changes and reductions in translucency than Coca-Cola. AFM analysis demonstrated progressive surface degradation and increased roughness following prolonged thermocycling, highlighting the significant influence of material composition and manufacturing protocol on the long-term optical stability of the investigated dental composites.

Keywords: dental composites; thermocycling; staining; color; translucency



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1. Introduction

Additive manufacturing technologies are increasingly used in restorative dentistry for the fabrication of provisional and definitive restorations, crowns, bridges, implant-

supported components, and surgical guides. Among existing vat photopolymerization techniques, digital light processing (DLP) has become one of the most widely used approaches because it enables the simultaneous polymerization of entire layers, allowing for efficient fabrication of geometrically complex restorations with high dimensional accuracy and limited material waste [1–4].

The properties of DLP-printed dental resin composites are strongly influenced by the polymerization process. During fabrication, the liquid photopolymer is selectively cured layer by layer and subsequently subjected to washing and additional post-polymerization. The final degree of conversion and cross-linking density depend on several material- and process-related factors, including resin composition, photoinitiator system, filler content, light wavelength, irradiance, exposure time, layer thickness, build orientation, and post-curing conditions [5–8]. The degree of conversion (DC), defined as the percentage of reactive carbon–carbon double bonds ($C=C$) converted into single bonds ($C-C$) during polymerization, is commonly used as an indicator of polymerization quality and material performance [9]. Inadequate conversion may increase the amount of residual monomers and contribute to water sorption, solubility, hydrolytic degradation, and deterioration of the matrix–filler interface. These changes may compromise the chemical stability and surface characteristics of resin-based materials and may ultimately contribute to clinically perceptible alterations in color and translucency, especially since restorative materials are in the oral environment, continuously exposed to masticatory loading, staining agents from food and beverages, and temperature fluctuations, all of which may compromise their esthetic appearance over time [10,11]. Although conventional light-cured dental composites may exhibit variability in polymerization, potentially affecting their bulk and surface characteristics, recently introduced resin composites intended for 3D printing require further investigation to determine their long-term chemical, optical, and surface stability [12]. As these materials are increasingly used for both provisional and definitive restorations, establishing their long-term esthetic performance is essential, particularly with regard to color stability and translucency, using established perceptibility ($PT_{00} = 0.8$) and acceptability ($AT_{00} = 1.8$) thresholds for color change and translucency parameters [13].

Previous studies have largely evaluated color stability, surface roughness, or degree of conversion separately, using mostly a single aging protocol. Direct comparisons between DLP-printed restorative composites and conventional light-cured composites combining optical, chemical, and topographical analyses after sequential thermocycling and staining remain scarce.

Therefore, the aim of the present study was to determine whether dental resin composites manufactured using different polymerization protocols exhibit different patterns of optical, chemical, and surface alteration following hydrothermal aging and subsequent staining. Specifically, the study evaluated whether aging-related changes in color and translucency were accompanied by detectable alterations in chemical structure and surface topography.

The null hypotheses were that the investigated materials would not differ in their optical response to hydrothermal aging and subsequent staining and that aging would not produce detectable changes in their chemical characteristics or surface topography.

2. Materials and Methods

2.1. Specimen Preparations

Three commercially available dental composites manufactured using different polymerization protocols were investigated in this study (Table 1): a light-cured dental composite—Omnichroma, Tokuyama, Tokyo, Japan (OMNI), a DLP 3D printed dental composite—SprintRay CROWN, SprintRay, Los Angeles, CA, USA (SPRINT) and a DLP

3D-printed dental composite—GC Temp PRINT, GC Europe, Leuven, Belgium (TEMP). As the investigated materials are commercially available, their complete formulations are proprietary; however, the basic compositional information provided by the manufacturers is presented in Table 1. Disc-shaped specimens (5 mm in diameter and 2 mm in thickness) were prepared for each material according to the respective manufacturer's instructions. The thickness of the disc is selected based on clinical relevance, since it enables the maximum increment of light-cured resin composites that can be adequately polymerized on the surface as well as in deeper regions of the material.

Table 1. Photopolymerization techniques and dental composites composition used in the study. DLP—Digital Light Processing; UDMA—urethane dimethacrylate; TEGDMA—tetraethyleneglycol dimethacrylate; MMA—methyl methacrylate.

Photopolymerization Techniques	Light-Cured Dental Composite	DLP-Printed Dental Composite	DLP-Printed Dental Composite
Brand name	Omnichroma (OMNI)	Sprint Ray CROWN (SPRINT)	GC Temp PRINT (TEMP)
Resin composition	UDMA, TEGDMA	UDMA, TEGDMA, other dimethacrylates	UDMA (MMA free)
Fillers	Spherical SiO ₂ -ZrO ₂	Ceramic nanoparticles	Silica nanoparticles
Filler loading, %	79 wt. %	>50 wt. %	~20 wt. %
Producer	Tokuyama, Tokyo, Japan	SprintRay, Los Angeles, CA, USA	GC Europe, Leuven, Belgium

For the fabrication of OMNI specimens, the composite was placed into a silicone mold to a thickness of 2 mm and covered with a transparent Mylar strip (SS White, cured, Philadelphia, PA, USA) and a microscope glass slide. Gentle pressure was applied to obtain a flat surface and minimize the formation of air voids. Each specimen was then light-cured perpendicular to the specimen surface through the Mylar strip and glass slide for 20 s using an LED curing unit (Bluephase Style, Ivoclar Vivadent, Schaan, Liechtenstein) operating at an irradiance of 1000 mW cm⁻², in accordance with the manufacturer's recommendations.

Specimens of the selected DLP 3D-printed dental composites intended for permanent (SPRINT) and temporary (TEMP) clinical applications were designed using CAD software (Exocad 3.0, Darmstadt, Germany) to obtain disc-shaped specimens with dimensions of 5 mm in diameter and 2 mm in thickness. The designs were exported as standard tessellation language (STL) files to the printing software (AccuWare V3.2.0.60, Shining 3D, Hangzhou, China). Specimens were fabricated using a DLP printer (AccuFab-L4D, Shining 3D, Hangzhou, China) under the following printing parameters: 90° build orientation, 50 µm layer thickness, and a light wavelength of 405 nm. Post-processing was performed to remove residual uncured resin by immersing the printed specimens in 90% isopropyl alcohol using a cleaning system (FH-WA-01, Formlabs, Somerville, MA, USA) for 20 min, followed by drying with compressed air. Subsequently, the specimens were post-cured for 30 min at a wavelength of 405 nm using a light-curing unit (FabCure 2, Shining 3D, Hangzhou, China), according to the manufacturer's recommendations.

Following fabrication, all specimens were polished using a three-step series of Al₂O₃-impregnated polishing discs (Sof-Lex, 3M ESPE, St. Paul, MN, USA) with medium, fine, and superfine grit sizes. The discs were mounted on a handpiece and used at 20,000 rpm for 40 s per surface under continuous water irrigation and light manual pressure. All specimen preparation and polishing procedures were performed by the same operator. Specimen

dimensions were subsequently verified using a digital caliper (Lukas Tools Digital Caliper, 300 mm; Vogel, Kevelaer, Germany). Finally, specimens were stored in distilled water at 37 °C for 24 h to allow for stabilization of the polymer network and completion of post-polymerization reactions prior to hydrothermal aging and staining.

A total of 132 specimens were prepared, corresponding to 44 specimens for each investigated material ($n = 44$). The specimens were randomly assigned to four independent aging intervals (T0, T1, T2, and T3), with 11 specimens allocated to each interval. For the evaluation of color change (ΔE_{00}) and translucency parameter (TP), five specimens from each material and aging interval were analyzed. Following aging, three randomly selected specimens from each material and aging interval were subjected to staining by immersion in coffee, Coca-Cola, or red wine, with one specimen assigned to each staining medium. Optical measurements were performed after 1 day of immersion, following rinsing and air-drying of the specimens. The same specimens were then returned to their respective staining solutions and re-evaluated after a total immersion period of 7 days. The purpose of such experimental design was to evaluate how progressive hydrothermal aging influences the subsequent susceptibility of resin composites to extrinsic staining. By applying staining after predefined aging intervals, the effect of changes in the polymer network induced by aging could be assessed under standardized conditions. Additionally, three independent specimens from each material and aging interval were characterized by Fourier-transform infrared spectroscopy (FTIR), while another three independent specimens were used for atomic force microscopy (AFM) analysis. The complete experimental design of the study, including specimen allocation and the number of specimens assigned to each analytical technique, is presented in Figure 1.

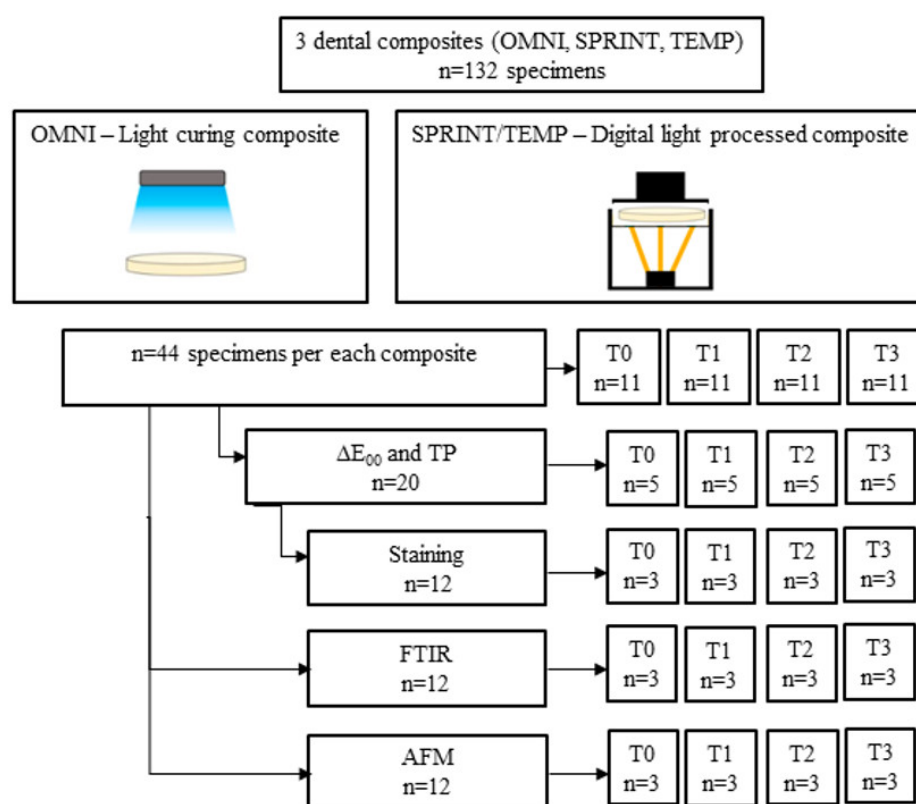


Figure 1. Workflow of the study and number of specimens used per analysis for each dental composite (OMNI, SPRINT and TEMP); T0—unaged; T1—aged for 5000 thermal cycles; T2—aged for 10,000 thermal cycles; and T3—aged for 30,000 thermal cycles.

2.2. Thermocycling

The specimens were randomly assigned to four aging groups. The first group remained unaged (T0), while specimens in the remaining groups were subjected to artificial aging using a thermocycler (THE 1200, SD Mechatronik, Feldkirchen-Westerham, Germany). Thermocycling was performed by alternating immersion in cold (5 °C) and hot (55 °C) water baths, with a dwell time of 60 s in each bath and a transfer time of 10 s between baths. The specimens were subjected to 5000 (T1), 10,000 (T2), or 30,000 (T3) cycles. These aging regimens were selected to simulate approximately 6 months (T1), 1 year (T2), and 3 years (T3) of clinical service, respectively [14]. To minimize contamination and microbial growth risks, the specimens were rinsed with distilled water and placed in freshly prepared solutions every three days during the aging procedure.

2.3. Color Change and Translucency Parameter

The color change (ΔE_{00}) and translucency parameter (TP) of the specimens were evaluated using certified white (Labsphere, NH, USA) and black (Spidercheckr 24, Lawrenceville, GA, USA) standards as background substrates. The measurement setup consisted of a D65 standard illuminant (BDS130-BWTEK, Plainsboro, NJ, USA), a TE-cooled CCD spectrometer (Glacier X, BWTEK, Plainsboro, NJ, USA), an integrating sphere (Labsphere, NH, USA), and an Optronik Digilux 9500 luxmeter (Instrument Systems GmbH, Berlin, Germany). ΔE_{00} and TP were evaluated before and after thermocycling, as well as after immersion in 10 mL of coffee (Nescafé Classic, Nestlé, Switzerland), Coca-Cola (The Coca-Cola Company, Atlanta, GA, USA) and red wine (Merlot, JP. Chenet, Les Grands Chais de France, Petersbach, GE, France). Following aging, three randomly selected specimens from each material and aging interval were subjected to staining by immersion in coffee, Coca-Cola, or red wine, with one specimen assigned to each staining medium. Optical measurements were performed after 1 day of immersion, following rinsing and air-drying of the specimens. The same specimens were then returned to their respective staining solutions and re-evaluated after a total immersion period of 7 days. The coffee solution was prepared by dissolving 3 g of instant coffee in 150 mL of freshly boiled distilled water. Coca-Cola and red wine were used as received without dilution and were refreshed daily to maintain a consistent composition. During the staining procedure, the specimens were stored at 37 °C in the dark. Following each immersion period, the specimens were rinsed with distilled water, gently air-dried, and subjected to optical characterization.

The color change was calculated according to the CIEDE2000 equation [15]:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{k_C S_C}\right) \left(\frac{\Delta H'}{k_H S_H}\right)}$$

where $\Delta L'$ —corrected lightness difference; $\Delta C'$ —corrected chroma difference; $\Delta H'$ —corrected hue difference; k_L —lightness weighting factor; k_C —chroma weighting factor; k_H —hue weighting factor; S_L —lightness compensation; S_C —chroma compensation; S_H —hue compensation; and R_T —interaction term between chroma and hue differences (accounts for blue region perceptual non-uniformity).

The translucency parameter was determined by calculating the color difference in the same specimen measured against black and white backgrounds using the following equation [16]:

$$TP = \{(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2\}^{1/2}$$

where the subscripts B and W correspond to the color coordinates measured over black and white backgrounds, respectively.

2.4. Fourier Transform Infrared (FTIR) Spectroscopy

To qualitatively evaluate changes in surface chemistry and the relative conversion index (RCI) of dental composites after aging, FTIR spectra were recorded using a Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) equipped with an attenuated total reflectance (ATR) accessory, over the spectral range of 400–4000 cm^{-1} . The polished surface of each composite specimen was placed directly in contact with the ATR crystal, and spectra were acquired without additional sample preparation or preparation of KBr pellets. FTIR spectra were acquired for unaged (T0) and aged specimens (T1, T2, and T3). The absorbance bands corresponding to the aliphatic C=C stretching vibration at 1635 cm^{-1} and the aromatic C=C bond at 1609 cm^{-1} , used as an internal reference, were identified and their intensities determined from the representative FTIR spectrum for each sample. The RCI was subsequently calculated from the ratio of these characteristic absorbance bands:

$$RCI(\%) = \left(1 - \frac{\left(\frac{C=C_{\text{aliphatic}}}{C=C_{\text{aromatic}}} \right)_{\text{aged}}}{\left(\frac{C=C_{\text{aliphatic}}}{C=C_{\text{aromatic}}} \right)_{\text{unaged}}} \right) \times 100$$

This equation was adapted from the conventional FTIR method used to determine the degree of conversion (DC) of dental resin composites [17]. However, in the present study, the unaged specimens (T0), rather than the uncured materials, served as the reference. Because FTIR spectra of the uncured materials were not recorded before polymerization, the absolute DC could not be calculated. Therefore, the resulting relative conversion index (RCI) should be regarded as a study-specific descriptive parameter reflecting changes in the aliphatic C=C-to-aromatic C=C ratio after hydrothermal aging relative to baseline. It should not be interpreted as an absolute measure of DC. For example, an RCI value of 7% indicates a 7% change in the spectral ratio relative to T0, rather than an absolute DC of 7%. The RCI was therefore used solely to describe and compare the magnitude of aging-related chemical changes among the investigated materials and was not included in inferential statistical analyses.

2.5. Atomic Force Microscopy (AFM)

Surface topography was characterized using an atomic force microscope (NTEGRA PRIMA, NT-MDT, Moscow, Russia) operating in semi-contact mode. Topography images were acquired simultaneously using NSG01 silicon probes (Tallinn, Estonia) with a nominal resonant frequency of approximately 150 kHz and a tip radius of approximately 10 nm. To improve the representativeness of the measurements, five randomly selected surface regions (while avoiding visible defects and edge regions) were analyzed for each specimen under identical scanning conditions, and the corresponding roughness values were averaged. Because the specimens were polished using a standardized protocol, no predefined orientation relative to the polishing direction was maintained during AFM acquisition. The acquired height images were processed using the manufacturer's software, and the root mean square roughness (Rq) was calculated from the surface height distribution.

2.6. Statistical Analysis

Color change (ΔE_{00}) and translucency parameter (TP) measurements were performed on replicate specimens for each aging group. Statistical analyses were carried out using SPSS Statistics software (version 22.0, IBM Corp., Armonk, NY, USA). The results are presented as mean values $\pm$ standard deviation (SD). Given the relatively small sample size and the non-normal distribution of the data, a non-parametric statistical approach was considered the most appropriate. Therefore, the Mann–Whitney U test was used to

evaluate differences between groups. Specifically, each group of sample replicates at the different aging intervals was compared separately with the corresponding baseline (T0) group. Statistical significance was established at $p < 0.05$ (*). FTIR-derived RCI values and AFM findings were evaluated descriptively and were not subjected to inferential statistical analysis because they were obtained from representative specimens rather than replicate measurements.

3. Results

3.1. Effects of Thermocycling on Color Change and Translucency Parameter

Color differences (ΔE_{00}) and translucency parameter (TP) values after thermocycling are presented in Figure 2. For clinical interpretation, ΔE_{00} values were compared with the established perceptibility ($PT_{00} = 0.8$) and acceptability ($AT_{00} = 1.8$) thresholds. Asterisks indicate statistically significant differences relative to baseline ($p < 0.05$).

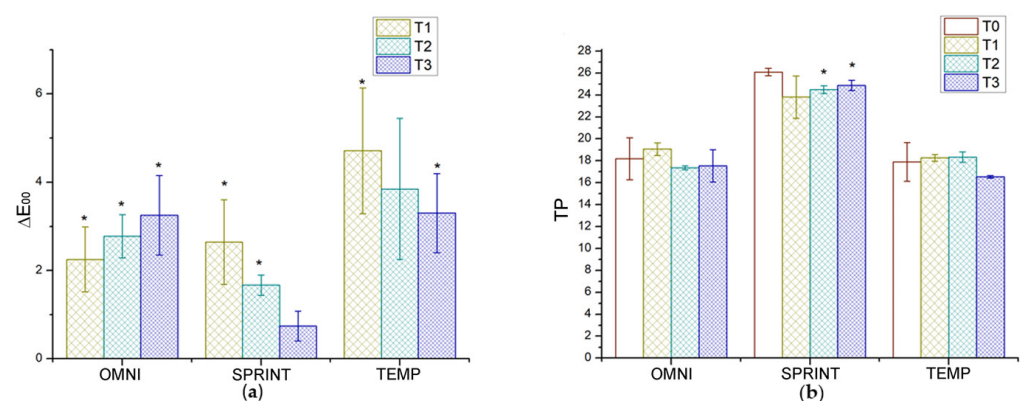


Figure 2. Color change, ΔE_{00} (a) and translucency parameter, TP (b) of the investigated dental composites after thermocycling. Data are presented as mean $\pm$ standard deviation (SD) of replicate measurements. ΔE_{00} values are expressed relative to the color of the unaged specimens (T0), corresponding to a value of 0 on the graph. TP values obtained after aging are compared with the corresponding initial values (T0). Statistically significant differences are indicated by * ($p < 0.05$).

Following 5000 thermocycles (T1), all investigated materials exhibited ΔE_{00} values above the acceptability threshold. TEMP showed the greatest color difference ($\Delta E_{00} = 4.71$), followed by SPRINT ($\Delta E_{00} = 2.64$) and OMNI ($\Delta E_{00} = 2.25$) (Figure 2a).

For OMNI, ΔE_{00} values increased progressively across the evaluated aging intervals, and significant differences relative to baseline were observed at T1, T2, and T3 ($p < 0.05$). All recorded values remained above the acceptability threshold.

SPRINT showed a different pattern, with a progressive reduction in ΔE_{00} as the number of thermocycles increased. Its ΔE_{00} value decreased from 2.64 at T1 to 1.67 at T2 and 0.74 at T3. Significant differences relative to baseline were observed at T1 and T2 ($p < 0.05$), whereas no significant difference was detected at T3 ($p > 0.05$). The color difference was above the acceptability threshold at T1, below the acceptability threshold at T2, and below the perceptibility threshold at T3.

TEMP exhibited the highest ΔE_{00} values among the investigated materials at T1 and T2. Significant differences relative to baseline were observed at T1 and T3 ($p < 0.05$), whereas the difference at T2 was not statistically significant ($p > 0.05$). Despite a gradual decrease after T1, the ΔE_{00} values of TEMP remained above the acceptability threshold throughout the thermocycling procedure.

The TP values differed markedly among the investigated materials (Figure 2b). At baseline, SPRINT exhibited the highest translucency (26.09 ± 0.33), whereas OMNI and TEMP showed lower and comparable values of 18.19 ± 1.91 and 17.90 ± 1.77 , respectively.

OMNI demonstrated only minor fluctuations in TP throughout thermocycling, with no statistically significant differences relative to baseline at any aging interval ($p > 0.05$). SPRINT exhibited a gradual reduction in TP with increasing thermocycling duration. The TP values at T2 and T3 were significantly lower than the baseline value ($p < 0.05$), whereas the change at T1 was not statistically significant ($p > 0.05$). Despite this reduction, SPRINT remained the most translucent material throughout all evaluated aging intervals. TEMP showed relatively stable TP values during the initial aging intervals, followed by a slight reduction at T3; none of these changes was statistically significant relative to baseline ($p > 0.05$).

3.2. Effects of Staining on Color Change and Translucency Parameter

Color differences following immersion in coffee, Coca-Cola, and red wine are presented in Figure 3. The magnitude of discoloration varied according to the restorative material, preceding thermocycling interval, staining medium, and immersion duration. Because only one specimen was evaluated for each combination of material, aging interval, and staining condition, the staining results were interpreted descriptively.

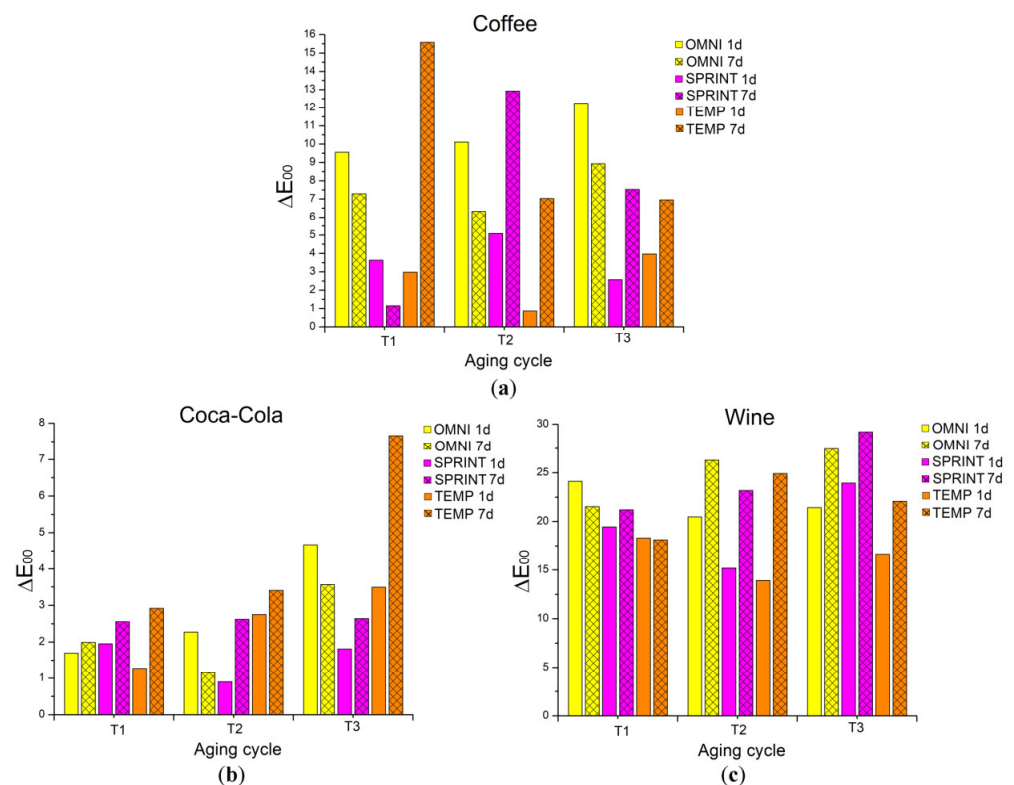


Figure 3. Color change (ΔE_{00}) after immersion in staining beverages: coffee (a), Coca-Cola (b) and wine (c).

Following coffee immersion, most ΔE_{00} values exceeded the clinical acceptability threshold, although the response varied considerably among the materials and aging intervals (Figure 3a). OMNI exhibited consistently high color changes after 1 day of immersion, with ΔE_{00} values increasing from approximately 9.6 at T1 to 12.2 at T3. After 7 days, its ΔE_{00} values were lower than the corresponding 1-day values at all aging intervals but remained above the acceptability threshold. SPRINT showed relatively low discoloration at T1, particularly after 7 days, but exhibited a marked increase after 7 days of immersion at T2, reaching approximately 12.9. At T3, its ΔE_{00} values were approximately 3.2 and 7.8 after 1 and 7 days, respectively. TEMP displayed relatively low 1-day ΔE_{00} values, ranging from below the acceptability threshold at T2 to approximately 4.0 at T3. However,

7-day immersion produced substantially greater discoloration, particularly at T1, where the highest coffee-induced color change was observed ($\Delta E_{00} \approx 15.6$).

Coca-Cola produced lower color changes than coffee and red wine (Figure 3b). At T1, ΔE_{00} values ranged from approximately 1.3 to 2.9, with TEMP exhibiting the lowest 1-day value. At T2 and T3, SPRINT showed the lowest 1-day discoloration, with ΔE_{00} values of approximately 0.9 and 1.8, respectively. OMNI exhibited an increase in its 1-day ΔE_{00} value with progressive thermocycling, reaching approximately 4.7 at T3. However, its 7-day values were lower than the corresponding 1-day values at T2 and T3. For SPRINT and TEMP, prolonged immersion generally resulted in greater discoloration. The highest Coca-Cola-induced color change was recorded for TEMP after 7 days of immersion at T3 ($\Delta E_{00} \approx 7.7$).

Red wine produced the greatest discoloration among the investigated staining media, with all ΔE_{00} values markedly exceeding the clinical acceptability threshold (Figure 3c). At T1, the values ranged from approximately 18.0 for TEMP to 24.1 for OMNI. At T2, 1-day immersion resulted in ΔE_{00} values of approximately 20.5, 15.2, and 13.9 for OMNI, SPRINT, and TEMP, respectively. After 7 days, these values increased to approximately 26.3 for OMNI, 23.4 for SPRINT, and 24.9 for TEMP. At T3, SPRINT exhibited the highest wine-induced color change after 7 days of immersion ($\Delta E_{00} \approx 29.0$), followed by OMNI ($\Delta E_{00} \approx 27.5$) and TEMP ($\Delta E_{00} \approx 22.0$). TEMP generally showed the lowest 1-day discoloration, whereas the differences among the materials became less consistent after prolonged immersion.

Immersion in coffee affected the translucency parameter (TP) of all investigated composites across all aging stages (Figure 4a). OMNI exhibited relatively high TP values after 1 day of immersion (16.11, T0), followed by an initial increase after the first aging interval and a gradual decline with further aging (12.54, T3). SPRINT maintained the highest TP values throughout the investigated aging period, with only a temporary reduction observed after the second aging stage. TEMP showed relatively stable TP values following 1 day of coffee immersion, with only minor variations among aging intervals (16.75-T1; 17.6-T2; 15.6-T3). Comparison of TP values after 1 and 7 days of immersion indicated an initial increase in translucency followed by a gradual decline with increasing aging severity. After 7 days of coffee immersion, SPRINT remained the most translucent material, followed by OMNI, whereas TEMP consistently exhibited the lowest TP values.

Immersion in Coca-Cola also altered the translucency of all investigated composites (Figure 4b). For OMNI, TP values generally increased with aging after 1 day of immersion, reaching the highest values after the most severe aging treatment (22.27-T3). However, prolonged immersion resulted in a partial reduction in translucency, particularly at advanced aging stages (17.15-T2; 19.28-T3). SPRINT exhibited consistently high TP values throughout all aging intervals and showed a tendency toward increased translucency with aging, while maintaining the highest TP values among the investigated materials after both 1 and 7 days of immersion. In contrast, TEMP exhibited the lowest TP values throughout the entire experiment, with translucency progressively decreasing as aging severity and immersion duration increased.

The translucency parameter (TP) values following immersion in coffee, Coca-Cola, and red wine are presented in Figure 4. The observed changes varied according to the material, aging interval, staining medium, and immersion duration. Because only one specimen was evaluated for each combination of material, aging interval, and staining condition, the results were interpreted descriptively.

Following coffee immersion, SPRINT generally exhibited the highest TP values, whereas OMNI and TEMP showed lower translucency (Figure 4a). For OMNI, the TP value after 1 day of immersion increased from approximately 16.1 at T0 to 19.1 at T1, followed by a decline at T2 and T3. After 7 days, a similar overall pattern was observed, although the

T3 value was slightly higher than the corresponding 1-day value. SPRINT maintained high TP values at T0 and T1, showed a marked reduction at T2, and increased again at T3 after 1 day of immersion. Its 7-day values were generally slightly lower than the corresponding 1-day values. TEMP exhibited moderate fluctuations, with 1-day TP values increasing from T0 to T2 and decreasing slightly at T3. After 7 days, TEMP generally showed lower TP values than after 1 day, particularly at T0 and T1.

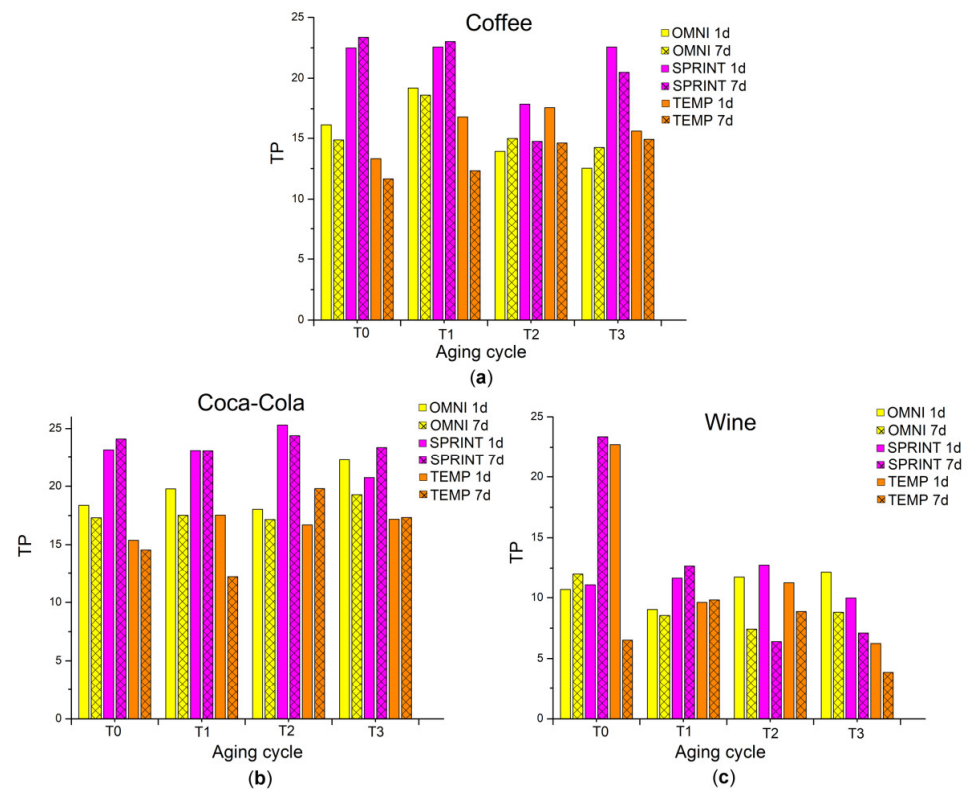


Figure 4. Translucency parameter (TP) after immersion in staining beverages: coffee (a), Coca-Cola (b) and wine (c).

Coca-Cola produced comparatively smaller changes in TP (Figure 4b). OMNI showed moderate fluctuations, with the highest 1-day TP value observed at T3. Its 7-day values were lower than the corresponding 1-day values at T1, T2, and T3. SPRINT maintained the highest translucency among the investigated materials at most aging intervals and reached its maximum TP value at T2 after 1 day of immersion. A slight reduction was observed at T3, particularly after 7 days. TEMP generally exhibited lower TP values than OMNI and SPRINT. Its lowest value was observed at T1 after 7 days of immersion, followed by higher values at T2 and T3. Thus, TEMP did not show a progressive reduction in translucency with increasing aging severity.

Red wine resulted in the lowest TP values among the investigated staining media, particularly after thermocycling and prolonged immersion (Figure 4c). At T0, SPRINT exhibited markedly higher translucency than OMNI and TEMP. Following aging, SPRINT showed a substantial reduction in TP, with further decreases observed after 7 days at T2 and T3. OMNI also showed an overall reduction in translucency, especially after prolonged immersion, although the 1-day TP values varied across aging intervals. TEMP generally exhibited the lowest TP values after aging. The lowest value recorded in the entire experiment was observed for TEMP after 7 days of red wine immersion at T3, with a TP of approximately 3.9.

Overall, red wine was associated with substantially lower TP values than coffee or Coca-Cola. However, the effect of prolonged immersion was not uniform and depended on the material and preceding aging condition.

3.3. Fourier Transform Infrared (FTIR) Spectroscopy Analysis

FTIR spectra of the investigated composites at baseline and after hydrothermal aging are presented in Figure 5. All materials exhibited characteristic absorption bands at approximately 2930 cm^{-1} , assigned to C–H stretching vibrations; 1716 cm^{-1} , assigned to C=O stretching; 1637 cm^{-1} , associated with aliphatic C=C stretching; 1609 cm^{-1} , associated with aromatic C=C stretching; and approximately $990\text{--}1000\text{ cm}^{-1}$, attributed to Si–O vibrations.

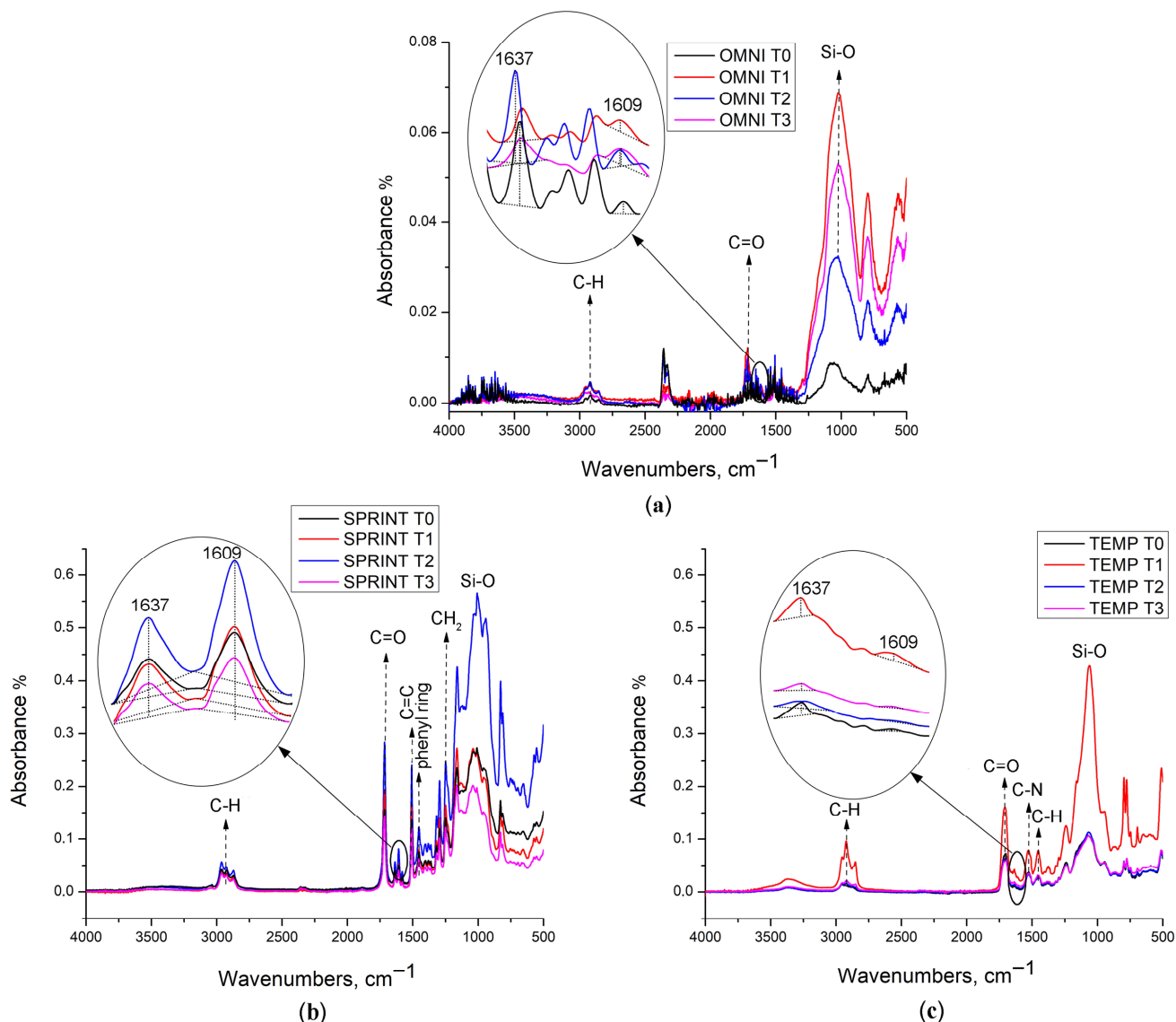


Figure 5. Fourier Transform Infrared (FTIR) spectra of investigated composites.

In addition to these common bands, the SPRINT spectra showed characteristic signals at approximately 1505 , 1436 , and 1245 cm^{-1} (Figure 5b). These bands were assigned to aromatic ring vibrations, phenyl-ring-related stretching or in-plane deformation modes, and CH_2 wagging or twisting vibrations, respectively. The band near 1245 cm^{-1} may also be associated with Si– CH_2 – CH_2 -containing structures. The TEMP spectra exhibited additional bands at approximately 1530 and 1453 cm^{-1} , assigned to C–N stretching and C–H bending vibrations, respectively (Figure 5c).

The regions corresponding to the aliphatic and aromatic C=C bands are shown at higher magnification in the insets in Figure 5. The integrated intensities of these bands, their aliphatic C=C-to-aromatic C=C ratios at each aging interval, and the corresponding relative conversion index (RCI) values are presented in Table 2.

Table 2. Integrated intensities of aliphatic and aromatic C=C peaks in FTIR spectra, their ratio, and relative conversion index (RCI) after each aging period.

Material	Aging	(C=C) _{aliphatic}	(C=C) _{aromatic}	$\frac{(C=C)_{aliphatic}}{(C=C)_{aromatic}}$	RCI, %
OMNI	T0	0.02131	0.00471	4.53	
	T1	0.03598	0.03229	1.11	75
	T2	0.03727	0.01884	2.06	54
	T3	0.03645	0.02926	1.24	72
SPRINT	T0	0.78091	0.97469	0.80	
	T1	0.67573	0.89935	0.75	6
	T2	1.12472	1.49715	0.75	6
	T3	0.52385	0.70042	0.74	7
TEMP	T0	0.13182	0.04313	3.06	
	T1	0.47214	0.36181	1.30	57
	T2	0.20586	0.08130	2.53	17
	T3	0.19955	0.08473	2.35	23

Only minor aging-related changes in the aliphatic C=C/aromatic C=C ratio were observed for SPRINT, as indicated by its relatively low RCI values. Because the unaged specimens rather than the uncured materials served as the reference, these changes should not be interpreted as additional polymerization or as absolute changes in the degree of conversion. Instead, they may reflect the combined effects of post-polymerization reactions, leaching of residual constituents, hydrolytic alterations, and other aging-related chemical processes.

The relationship between color change (ΔE_{00}) and the relative conversion index (RCI) following hydrothermal aging is presented in Figure 6. Distinct aging-related trends were observed among the investigated materials.

TEMP exhibited the highest RCI values at all aging intervals, indicating the greatest relative changes in the aliphatic C=C/aromatic C=C ratio. The RCI gradually decreased with increasing thermocycling, whereas the corresponding ΔE_{00} values remained relatively low and showed only minor variations after the initial aging interval.

In contrast, SPRINT demonstrated the lowest RCI values throughout the investigated aging period, suggesting only limited changes in the FTIR spectral ratio following hydrothermal aging. Despite these minimal chemical changes, the corresponding ΔE_{00} values progressively decreased with increasing aging, reaching the lowest value after 30,000 thermocycles.

OMNI exhibited intermediate RCI values that increased gradually from T1 to T3. In contrast, ΔE_{00} values decreased after 10,000 thermocycles and subsequently increased again after 30,000 cycles, resulting in the highest color difference among the investigated materials at the final aging interval.

Overall, no consistent relationship was observed between the magnitude of color change and the corresponding RCI values. Materials exhibiting larger relative changes in the FTIR spectral ratio did not necessarily demonstrate greater color changes, indicating

that alterations in the aliphatic C=C/aromatic C=C ratio alone cannot fully explain the optical changes induced by hydrothermal aging.

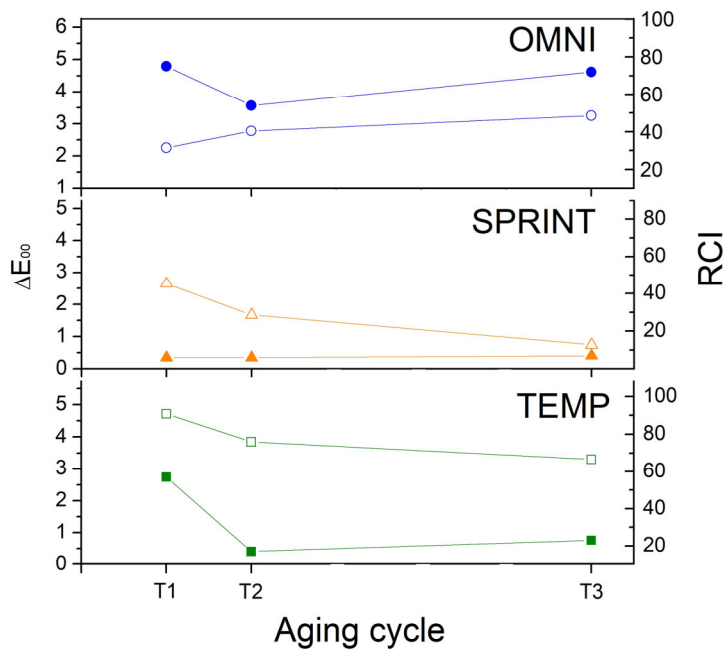


Figure 6. Correlation plots between the Relative Conversion Index (RCI) and color change (ΔE_{00}) after each aging stage. Lines connecting consecutive data points are shown only to illustrate the temporal evolution during aging and do not represent fitted regression curves. Open symbols represent ΔE_{00} values, whereas filled symbols represent RCI values in %.

3.4. AFM

Representative two-dimensional and three-dimensional AFM topographies of the investigated composites at baseline and after thermocycling are presented in Figure 7.

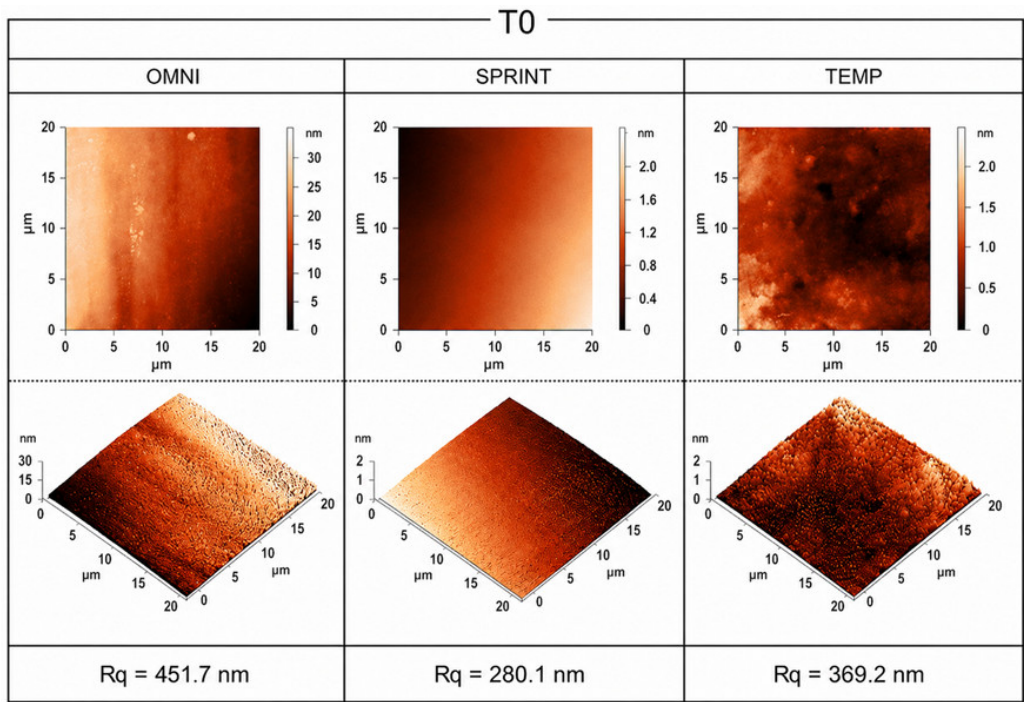


Figure 7. Cont.

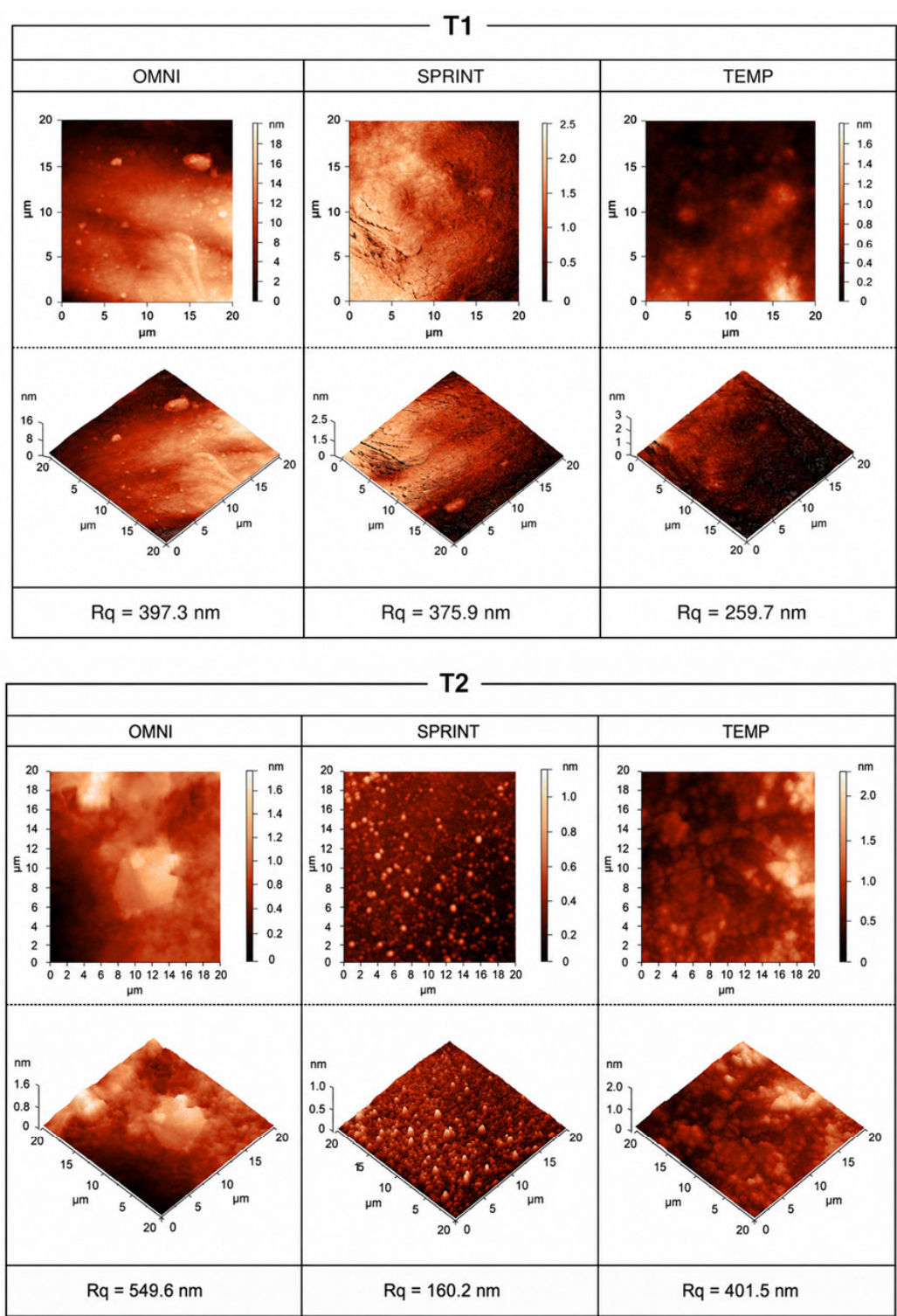


Figure 7. Cont.

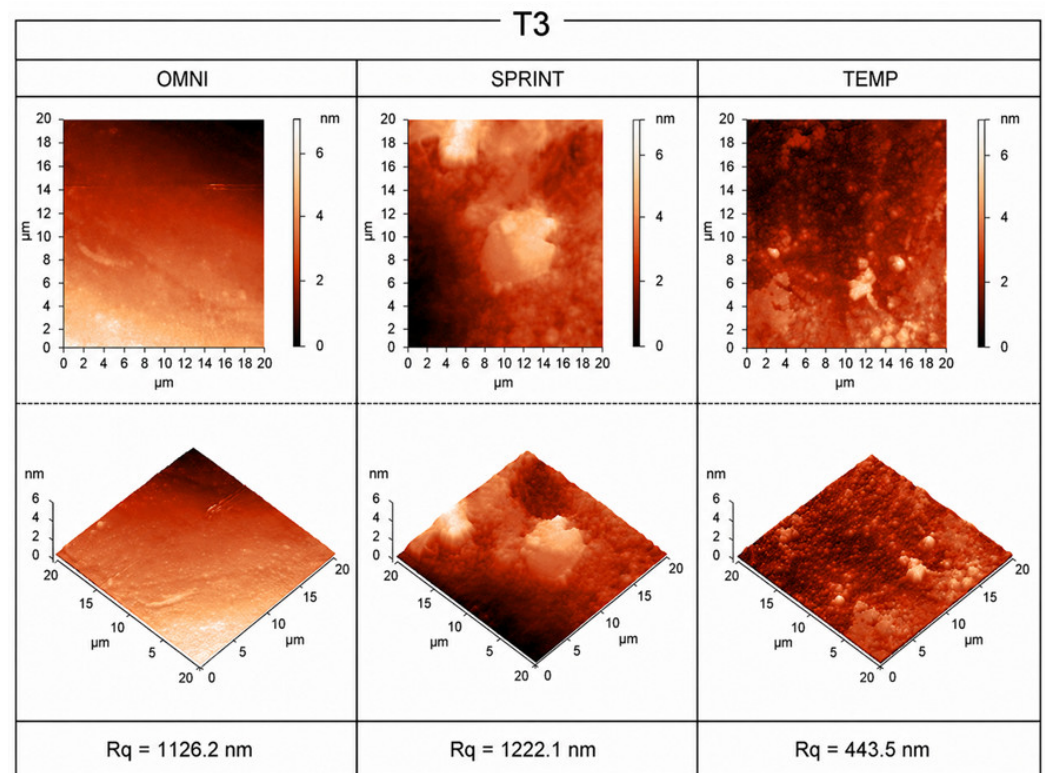


Figure 7. AFM topographs of composites before (T0) and after thermocycling (T1, T2, and T3).

For OMNI, the unaged specimen (T0) exhibited a relatively heterogeneous surface with moderate roughness ($R_q = 451.7$ nm). After 5000 thermocycles (T1), the roughness slightly decreased to 397.3 nm, and the surface appeared somewhat more uniform. Following 10,000 cycles (T2), R_q increased to 549.6 nm, accompanied by greater topographical heterogeneity and more pronounced elevated and depressed regions. The largest change was observed after 30,000 cycles (T3), when R_q increased markedly to 1126.2 nm. At this interval, the AFM images showed substantial height variation and a highly irregular surface topography.

SPRINT exhibited the lowest baseline roughness among the investigated materials ($R_q = 280.1$ nm), with a relatively uniform surface at T0. After 5000 cycles, R_q increased to 375.9 nm, and localized surface irregularities became more evident. At T2, roughness decreased to 160.2 nm, representing the lowest R_q value recorded in the study. The corresponding AFM image showed a comparatively uniform fine-granular topography. After 30,000 cycles, R_q increased sharply to 1222.1 nm, the highest value observed among all materials and aging intervals. The surface at T3 was characterized by pronounced topographical heterogeneity and large differences between elevated and depressed regions.

For TEMP, the baseline specimen exhibited a moderately heterogeneous surface with an R_q value of 369.2 nm. After 5000 cycles, roughness decreased to 259.7 nm and the surface appeared more uniform. At T2, R_q increased to 401.5 nm, together with greater variation in surface height and a more heterogeneous topography. After 30,000 cycles, roughness further increased to 443.5 nm. The corresponding AFM images showed irregularly distributed elevations and depressions, although the magnitude of the surface changes was less pronounced than that observed for OMNI and SPRINT at T3.

Overall, thermocycling produced material-dependent and non-linear changes in surface roughness. At T3, OMNI and SPRINT showed marked increases in R_q , whereas TEMP exhibited only a moderate increase. Among all conditions, SPRINT at T2 had the lowest R_q value, while SPRINT at T3 had the highest.

The material-dependent changes observed in the representative AFM topographies were further illustrated by plotting Rq as a function of thermocycling interval (Figure 8). The three materials exhibited distinct, non-linear responses to hydrothermal aging. OMNI showed a slight initial decrease in Rq, followed by an increase at T2 and a pronounced rise at T3. SPRINT demonstrated the greatest variability, with an increase at T1, a marked decrease at T2, and a sharp rise at T3, where it reached the highest Rq value among the investigated materials. TEMP exhibited comparatively smaller changes, with an initial decrease followed by moderate increases at T2 and T3. Overall, prolonged thermocycling resulted in substantially greater increases in surface roughness for OMNI and SPRINT than for TEMP.

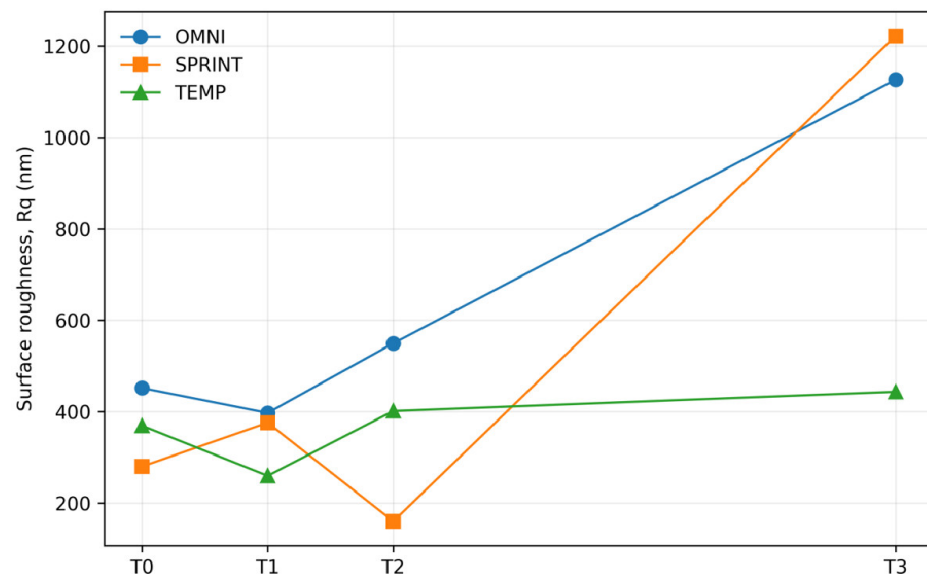


Figure 8. Surface roughness (Rq) of the investigated materials as a function of thermocycling interval (T0–T3). Lines connecting consecutive data points are included solely to illustrate aging-related trends and do not represent fitted regression curves.

Compared with OMNI and SPRINT, TEMP exhibited less severe surface degradation after prolonged thermocycling, suggesting greater resistance to thermally induced surface deterioration.

4. Discussion

The findings of the present study showed that thermocycling affected the optical properties, chemical characteristics, and surface topography of the investigated dental resin composites, while exposure to staining media produced material and staining medium dependent changes in color and translucency. The magnitude and direction of these changes varied according to the investigated material, aging interval, and staining medium, indicating a strongly material dependent response to hydrothermal and chromogenic challenges. Accordingly, the null hypotheses were partially rejected. Statistically significant differences were identified for the outcomes subjected to inferential analysis, whereas the staining related findings were interpreted descriptively. The clinical relevance of the observed color changes was assessed using established perceptibility and acceptability thresholds, and several ΔE_{00} values exceeded the clinical acceptability threshold. These findings suggest that prolonged hydrothermal aging and exposure to commonly consumed chromogenic beverages may compromise the esthetic stability of investigated dental resin composites, although the extent of these changes depends on the specific material and exposure conditions.

Thermocycling was selected as an established *in vitro* aging model that provides insight into the behavior of dental materials under simulated oral conditions [18]. All three structural components of resin composites—the resin matrix, filler particles, and filler–matrix interface may be affected by hydrolytic degradation. Although the relative contribution of these components remains debated, a recent study suggested that hydrothermal degradation may be predominantly associated with changes within the polymer matrix [19]. Hydrothermal aging can promote water sorption, plasticization, swelling, and hydrolytic changes within the resin matrix, whereas deterioration of the filler–matrix interface may further facilitate water penetration and contribute to changes in optical properties [20]. In addition, hydrolysis of the silane coupling agent may weaken the filler–matrix interface and promote the development of interfacial defects, thereby potentially increasing susceptibility to chromogenic agents [21]. The extent of hydrothermal degradation may also be influenced by filler loading, as highly filled composites contain a lower relative volume of resin matrix available for water diffusion and have therefore been reported to exhibit greater resistance to hydrothermal aging [22].

Following hydrothermal aging, all investigated composites exhibited changes in color, although the magnitude and temporal pattern of these changes were material dependent. After the first thermocycling interval, SPRINT showed the lowest ΔE_{00} value and therefore the greatest initial color stability, followed by OMNI, whereas TEMP exhibited the largest color change. With continued aging, ΔE_{00} decreased for TEMP and SPRINT, while OMNI also showed comparatively favorable color stability at the later aging intervals. These non-linear responses indicate that prolonged thermocycling did not uniformly increase discoloration in the investigated materials.

Differences in filler loading, filler size and distribution, resin composition, photoinitiator system, and filler–matrix coupling may have contributed to the material-dependent optical responses. This interpretation is consistent with previous studies indicating that the color stability of resin composites is influenced by several compositional and polymerization-related factors [23,24]. In particular, the comparatively lower filler content of TEMP may have contributed to its greater initial susceptibility to hydrothermal aging by increasing the relative proportion of resin matrix available for water sorption. However, because the complete material formulations were proprietary, the contribution of individual compositional factors could not be determined.

The FTIR findings also demonstrated material-dependent changes in the aliphatic C=C/aromatic C=C ratio during aging. SPRINT showed comparatively small changes in the study-specific relative conversion index, indicating that the measured spectral ratio remained relatively stable throughout thermocycling. This observation should not, however, be interpreted as evidence of a higher or lower absolute degree of conversion, because spectra of the uncured materials were unavailable and the RCI was used only as a descriptive index. Moreover, the relationship between the observed spectral changes and color stability cannot be established directly from the present data.

The reduction in ΔE_{00} observed for SPRINT and TEMP during prolonged thermocycling further indicates that hydrothermal aging does not necessarily produce a progressive increase in color change. Continued post-curing reactions, polymer-network relaxation, leaching of residual constituents, or changes in surface characteristics could potentially influence the optical response during aging. Nevertheless, the present experimental design does not permit identification of the mechanisms responsible for this material-specific behavior. Differences in proprietary resin formulations, filler characteristics, photoinitiator systems, and other undisclosed constituents may have contributed, but these possibilities remain speculative.

Immersion in chromogenic beverages produced material-, medium-, and time-dependent changes in the color of the investigated resin composites. Coffee and red wine generally

produced more pronounced discoloration than Coca-Cola, although the magnitude and temporal pattern of the changes varied among the materials. The staining potential of red wine has been attributed to the combined effects of its alcohol content, acidity, and intense chromogenic compounds. Alcohol may promote softening of the resin matrix and increase susceptibility to pigment uptake, whereas the yellow-brown chromogens present in coffee may be adsorbed onto or absorbed into the polymer matrix depending on their polarity and affinity for the material [25–27]. These mechanisms, however, were not directly investigated in the present study.

SPRINT generally exhibited favorable color stability under several staining conditions, although it was not consistently the least affected material at every immersion interval. Its behavior may be related to its comparatively high ceramic filler content and lower relative proportion of resin matrix. Nevertheless, the contribution of the DLP fabrication process itself cannot be isolated because the investigated materials differed simultaneously in filler loading, resin composition, photoinitiator system, and other proprietary constituents. The present findings are broadly consistent with previous studies of permanent 3D-printed restorative resin composites, although direct comparison is limited by differences in material formulations and experimental protocols [28]. TEMP, which contains a considerably lower reported amount of silica-based filler, showed greater color changes under some staining conditions. Its lower filler content may have increased the relative volume of resin matrix available for water and pigment uptake, but this interpretation remains tentative.

Changes in color were accompanied by alterations in the translucency parameter, another relevant indicator of esthetic performance. Hydrothermal aging may modify translucency through water sorption, plasticization of the resin matrix, and changes in the optical relationship between the resin and filler phases. Alterations at the filler–matrix interface may also affect light scattering, particularly when differences in refractive index develop during aging [29,30]. However, these mechanisms were not directly measured in the present study.

Red wine produced marked translucency changes in several material–time combinations, which may be associated with the combined effects of acidity, alcohol, and intense chromogenic compounds. TEMP appeared comparatively susceptible to wine-induced alterations in translucency, whereas SPRINT showed more stable TP values under several exposure conditions. Nevertheless, neither material exhibited a uniformly consistent response throughout the entire staining protocol. These observations are broadly comparable with those of our previous study on another 3D-printed resin composite intended for long-term clinical use [31]. Coffee also produced noticeable, material-dependent changes in translucency, potentially because of chromogen uptake within the resin matrix. Coca-Cola generally caused less pronounced alterations than coffee and red wine, although measurable changes were still observed in some conditions and may be related to its acidic composition. OMNI exhibited an intermediate and variable response rather than a uniformly balanced resistance across all staining media.

AFM analysis demonstrated that thermocycling produced material-dependent alterations in the surface topography of all investigated resin composites. Repeated temperature changes may generate thermomechanical stresses because of differences in the coefficients of thermal expansion of the resin matrix and inorganic fillers. Water uptake may additionally promote matrix plasticization and hydrolytic changes at the filler–matrix interface, potentially affecting surface integrity and roughness [32,33]. However, these mechanisms were not directly evaluated in the present study.

OMNI exhibited a relatively smooth surface before aging, followed by increasing topographical heterogeneity and a pronounced increase in Rq after prolonged thermocycling. The aged surfaces showed more prominent protrusions, depressions, and height variation

than the unaged surface. Such changes may influence light scattering and the retention of chromogenic substances, although the present study did not establish a direct relationship between surface topography and optical stability [34–36].

SPRINT showed the most variable and non-linear roughness response. Rq increased after the first aging interval, decreased at T2, and subsequently rose sharply at T3, reaching the highest final value among the investigated materials. These fluctuations may reflect competing aging-related processes within the resin-rich surface and filler–matrix system; nevertheless, the AFM findings do not permit identification of specific mechanisms such as filler dislodgement, polymer-network deterioration, or surface reorganization [37–39].

TEMP exhibited comparatively smaller changes in surface roughness, with an initial decrease in Rq followed by moderate increases during subsequent thermocycling intervals. Although its lower reported filler content may influence its response to hydrothermal aging, the formation of the observed surface depressions cannot be attributed solely to filler loading. Resin chemistry, filler characteristics, polymerization conditions, and filler–matrix coupling may also have contributed [40].

Although increased surface roughness may facilitate pigment retention and discoloration, the present findings indicate that roughness was not the sole determinant of optical stability. Color change is also influenced by intrinsic factors such as resin matrix composition, water sorption, pigment affinity, filler characteristics, photoinitiator system, and changes occurring within the bulk of the material. In addition, AFM evaluates nanoscale topography within a limited surface area, whereas color measurements reflect the overall optical response of the specimen. These differences in measurement scale and the multifactorial nature of discoloration may explain why the material with the highest final roughness did not necessarily exhibit the greatest color change. Because no direct correlation analysis was performed, this interpretation should remain cautious.

The present study may provide valuable insight into the effects of thermocycling and staining on commercially available dental resin composites, when several limitations are acknowledged. First, staining-induced color change (ΔE_{00}) and translucency (TP), the FTIR-based relative conversion index (RCI), and AFM surface topography were evaluated descriptively using representative specimens or measurements. Consequently, these findings should be interpreted as descriptive indicators rather than statistically validated quantitative comparisons.

Second, the investigated materials were commercially available products with proprietary formulations. Because the complete compositions were not disclosed and uncured materials were unavailable for comprehensive characterization, the contribution of individual constituents to the observed optical, chemical, and surface changes could not be determined.

Finally, the study was conducted under controlled *in vitro* conditions, which cannot fully reproduce the complexity of the oral environment. Water sorption, solubility, and leachate release were not evaluated, and complementary techniques such as scanning electron microscopy were not employed. Future studies incorporating these analyses, together with replicate measurements and longer-term aging protocols, would provide a more comprehensive understanding of the mechanisms underlying the observed material behavior.

5. Conclusions

Within the limitations of this *in vitro* study, it was shown that thermocycling significantly affected the optical properties of all investigated dental composites, producing material-dependent changes in color stability and translucency. Exposure to staining beverages further compromised the esthetic performance of the composites, with red wine and coffee producing substantially greater color changes than Coca-Cola. The permanent DLP-printed composite SPRINT exhibited the most favorable optical behavior, characterized by

the highest resistance to coloration and the most stable translucency after aging and staining. The temporary DLP-printed composite TEMP showed the greatest susceptibility to aging- and staining-induced optical degradation, resulting in the least favorable long-term esthetic performance. The conventionally light-cured composite OMNI exhibited moderate changes in color and translucency following thermocycling.

Although thermocycling increased surface roughness in all materials, the relationship between roughness and optical stability was not direct. The superior performance of permanent DLP-printed composite SPRINT despite pronounced surface degradation suggests that intrinsic material characteristics, including resin matrix composition, filler architecture, and manufacturing protocol, play a more important role in preserving esthetic properties than surface roughness alone.

From a clinical perspective, maintaining color stability and translucency over time is essential for preserving the esthetic appearance of restorations. The superior optical and chemical stability observed for SPRINT suggests greater potential for long-term esthetic performance, whereas materials exhibiting greater coloration may require earlier replacement, particularly in clinical situations with high esthetic requirements.

Taking into account that this study involves a limited number of commercially available composites, the obtained results should be interpreted with caution. Future investigations should be focused on evaluating a broader range of commercially available light-cured and 3D-printed dental composites to determine whether the observed results are material-specific or applicable across different formulations. Therefore, further studies incorporating a broader range of material formulations and more clinically relevant experimental conditions are needed to validate and expand upon the findings of the present study.

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Conflicts of Interest: The authors declare no conflicts of interest. Dental M Press is a dental laboratory and helped in producing the material specimens but had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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