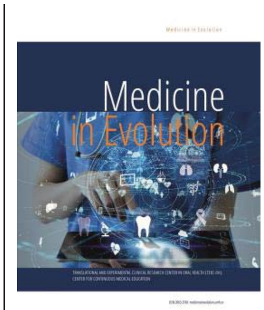
Evaluation of the Surface, Structural, And Optical Stability of a Single-Shade Universal Resin Composite After Exposure to Acidic Staining Solutions



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Ruxandra Sava-Rosianu[1,2], Ramona Dumitrescu[1,2], Lucian Floare[1,3], Vanessa Bolchis[1,2,3], Vlad Tiberiu Alexa[1,2], Berivan Laura Rebeca Buzatu[1,2,3], Daniela Jumanca[1,2], Roxana Oancea[1,2], Octavia Balean[1,2]

[1]Translational and Experimental Clinical Research Centre in Oral Health, University of Medicine and Pharmacy "Victor Babes", 300040 Timisoara, Romania;
[2]"Victor Babes" University of Medicine and Pharmacy, Clinic of Preventive, Community Dentistry and Oral Health, Eftimie Murgu Sq. no 2, 300041 Timisoara, Romania;
[3]Doctoral School, "Victor Babes", University of Medicine and Pharmacy Timisoara, Eftimie Murgu Square No 2, 300041, Timisoara, Romania

Correspondence to:
Name: Dumitrescu Ramona
E-mail address: dumitrescu.ramona@umft.ro

Name: Alexa Vlad
E-mail address: vlad.alexa@umft.ro

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Abstract

1.Background/Objectives: Advances in adhesive dentistry have led to the development of single-shade universal resin composites (SsURCs), designed to simplify shade selection and enhance aesthetic outcomes, yet their long-term stability under acidic conditions remains a concern. This study aimed to evaluate the surface properties, zeta potential, Raman spectral characteristics, and color stability of Omnichroma, a single-shade universal resin composite, before and after immersion in staining solutions (coffee, Coca-Cola, and red wine). 2.Methods: A total of 20 disc-shaped specimens were prepared and divided into four subgroups: control (artificial saliva), coffee, Coca-Cola, and red wine. Surface roughness, contact angle, and color stability were assessed before and after immersion, while Raman spectroscopy analyzed structural modifications, and zeta potential measurements determined the point of zero charge (PZC). 3.Results: Coffee caused the most significant color change ($\Delta E = 10.84 \pm 1.03$) and roughness increase ($SR = 1.53 \pm 0.38$), followed by red wine ($\Delta E = 5.33 \pm 0.27$; $SR = 1.15 \pm 0.08$) and Coca-Cola ($\Delta E = 1.28 \pm 0.44$; $SR = 1.21 \pm 0.31$). The PZC was identified at pH 3.8, indicating a predominance of anionic ionizable groups. Raman analysis revealed molecular alterations, particularly in C=O stretching and C=C aromatic ring vibrations. 4. Conclusions: These findings suggest that acidic beverages affect the structural and optical stability of Omnichroma, with coffee having the greatest impact. Further in vivo studies are needed to assess its long-term performance in clinical settings.

Keywords: Single-shade universal resin composite, contact angle, zeta potential, hardness, roughness, Raman spectroscopy, colour stability

INTRODUCTION

Direct resin composite restorations are widely used in dental clinics, benefiting from advancements in adhesive dentistry. The "Natural Layering Concept" was developed to meet patients' aesthetic expectations by replicating the appearance of natural teeth [1]. While this technique is commonly applied in clinical practice, it demands a high level of restorative skill and extended chairside time. In contrast, recently introduced single-shade universal resin composites (SsURCs) streamline the restorative process, offering a more efficient alternative [2]. Composite resins have become increasingly popular as direct restorative materials in recent decades, largely due to the growing patient preference for aesthetically pleasing restorations. These materials are highly valued by clinicians for their ability to facilitate minimally invasive procedures, their excellent aesthetic results, their strong adhesion to dental tissues, and their enhanced durability. The ability of a resin composite to seamlessly match the colour of the dental structure is a key factor in patients' perception of aesthetic treatment quality. This aspect significantly influences both their satisfaction with the final result and their assessment of the clinician's professional competence [3].

Over time, significant advancements in dental composite technology have improved their physical, chemical, and aesthetic properties, enabling their use in a wide range of clinical scenarios. However, the modern diet poses new challenges to these materials. Increased consumption of acidic beverages such as carbonated drinks, coffee, black tea, and alcohol introduces potential risks to resin-based restorations. The acidic nature of these substances, with their low pH, can compromise the structural integrity of the composite, alter the properties of its fillers, and weaken the bond between the fillers and the resin matrix, ultimately reducing the longevity of restorations. Clinicians must carefully evaluate the structural composition of composite resins and their resistance to chemical and mechanical stresses when selecting materials for restorative treatments. Within the oral cavity, restorations are continuously exposed to environmental changes, whether through acidic foods and beverages or the mechanical forces of mastication and, in certain cases, excessive occlusal forces. These challenges can significantly influence the performance and durability of resin-based materials.

Single-shade universal resin composites (SsURCs) are designed to adapt to a wide range of tooth shades, eliminating the need for shade selection. Their ability to seamlessly integrate with surrounding tooth structures relies on the blending effect, which occurs through light reflection and scattering. When light interacts with the composite material, it disperses across the surface of the filler particles and scatters in various directions. This optical phenomenon is influenced by factors such as restoration size and the material's translucency. The terms "chameleon effect" and "colour adjustment potential" are also commonly used to describe this blending capability [4].

Omnichroma is a single-shade, structurally coloured universal composite designed for a wide range of direct restorative applications. Its extensive colour-matching capability eliminates the need for shade selection, streamlines the restorative process, and reduces composite inventory. This allows clinicians to minimize chair time, decrease material waste, and lessen dependence on traditional shade-matching techniques [5].

The available literature on Omnichroma provides limited data, primarily focusing on its color adjustment potential [6, 7], shade-matching ability [8], optical behavior [9], and color stability [1]. Some studies also explore its cytotoxicity [10], as well as its flexural strength (FS) and elastic modulus (EM) [11, 12]. However, comprehensive research on its mechanical, spectral, and structural properties remains scarce, highlighting the need for further investigations to fully understand its clinical performance.

Aim and objectives

The aim of this study is to evaluate the surface properties (contact angle, zeta potential and roughness) and Raman spectral characteristics of a single-shade composite, as well as its colour stability before and after immersion in staining solutions (coffee and acidic carbonated beverage).

MATERIALS AND METHODS

A disc-shaped specimen was prepared using a custom-made plexiglass mold with an internal diameter of 10 mm and a height of 2 mm. Omnichroma, a light-cured, radiopaque, single-shade universal composite (Tokuyama Dental, Tokyo, Japan; Batch No. 123E83), was used as the restorative material. Omnichroma is a single-shade universal resin composite that utilizes structural color technology to match a wide range of tooth shades without the need for additional tints or pigments. Its color-matching ability is based on the uniform size and arrangement of filler particles, which enable it to blend seamlessly with surrounding tooth structures. Its filler system consists of 79% by weight (68% by volume) of spherical silica-zirconia filler, with a mean particle size of 0.3 µm (ranging from 0.2 to 0.6 µm), along with a composite filler. The resin system comprises urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA), with additional components including Mequinol, dibutyl hydroxyl toluene, and a UV absorber. The composite was carefully compacted into the mold using a Teflon-coated plastic filling instrument, and excess material was removed with an explorer. A celluloid strip was placed over the compacted resin, followed by a 1 mm thick glass slide to flatten the surface and extrude any remaining excess material. The resin was then light-cured for 40 seconds through the Mylar strip and glass slide using an LED curing unit (3M Elipar DeepCure-S LED, USA) with a light intensity of 400 mW/cm^2 in a uniform continuous curing mode. The light intensity was verified after every five samples.

A total of 20 Omnichroma composite disc-shaped specimens were prepared. The specimens were divided into four subgroups (n = 5 each), with one group serving as a control and the other three immersed in different staining solutions: artificial saliva (control), coffee, Coca-Cola, and red wine. The solutions were prepared as follows:

- Control Group
- Coffee Group: Specimens were immersed in a Nespresso coffee solution, prepared by dissolving one capsule of black coffee (Nespresso, Switzerland) in 100 ml of boiling distilled water and allowing it to cool to room temperature.
- Coca-Cola Group: Specimens were stored in Coca-Cola (Coca-Cola Co., USA), a carbonated soft drink composed of carbonated water, high fructose corn syrup, caramel color, phosphoric acid, and caffeine. To maintain carbonation, tightly sealed containers were used, and a fresh bottle was opened daily.
- Red Wine Group: Specimens were immersed in 150 ml of red wine (Feteasca Neagra, Purcari, Romania). The wine was poured into sealed glass containers to prevent oxidation, and a fresh bottle was used daily.

All specimens were stored at 37°C in an incubator to simulate oral conditions, and the solutions were replaced daily to ensure consistency. The pH of the solutions was measured and stabilized using a calibrated digital pH meter (Hanna Instruments HI 5221, Romania), with an accuracy of ±0.01 pH. Measurements were taken before specimen immersion to ensure consistency in the storage environment.

The primary color of Omnichroma was recorded using a digital spectrophotometer (Vita Easyshade®V, Compact, Vita Zahnfabrik, Bad Säckingen, Germany). To ensure accurate measurements and minimize absorption effects, each specimen was placed on a white background. The probe tip of the spectrophotometer was positioned perpendicularly at the center of the specimen and brought into direct contact with its surface.

To assess its hydrophobicity and surface interaction properties, the water contact angle was measured on two distinct surfaces of the material: a smooth, polished area (Surface 1) and a rough, uneven area (Surface 2). These measurements provide insight into the wettability and potential influence of surface texture on the composite's clinical performance. Water contact angle measurements were performed using the Drop Shape Analyzer-DSA25 (KRÜSS GmbH, Germany). The Double Sessile Drop method was employed with distilled water at a drop volume of 1 µL. The temperature was maintained at 20°C throughout the experiment. Each composite material was subjected to six to seven measurements per surface to ensure statistical reliability. Water served as the drop phase, with air as the surrounding phase. The system recorded parameters such as contact angle (CA) at right (r), left (l), and mean (m) positions, surface free tension (SFT), and volume of the applied liquid droplets. The mean contact angle and standard deviation were calculated for each surface of the samples. The instrument's software (KRÜSS ADVANCE 1.14.1.16701) automatically analyzed the drop profiles. The experimental process was designed to ensure replicability and eliminate variability by maintaining identical environmental and procedural settings for both sets of measurements. Additional SFE (surface free energy) results were calculated using the OWRK model, focusing on polar and dispersive contributions.

The zeta potential was measured using a Particle Charge Detector Mutek PCD-03 (Mütek GmbH, Neckartailfingen, Germany). This device is designed to assess the concentration of water-soluble ionic polymer solutions and determine the zero-charge point of composite micro- and nanoparticles. For the analysis, 10-20 mg of solid powder was dispersed in 10 mL of PBS solution, ensuring a uniform suspension. After an equilibration period of 10 minutes, the streaming potential was recorded directly from the instrument's display (Figure 1).

Figure 1. Streaming Potential Analysis of a Dental Composite: Powderization and Measurement Using the PCD Device

The vibrational properties of Omnichroma composite resin were analyzed before and after immersion in coffee, red wine, and Coca-Cola using the LabRAM Soleil™ Raman Microscope (HORIBA Scientific). Raman spectra were acquired with a 532 nm laser, utilizing QScan™ lightsheet confocal imaging for enhanced spatial resolution and SmartSampling™ technology for rapid spectral acquisition. Measurements were performed with four accumulations to improve signal clarity and minimize noise. A 5× magnification objective lens was used to precisely focus on the sample surface, covering a spectral range of 200 to 3200 cm^{-1}. To prevent thermal damage and ensure optimal spectral quality, a neutral density (ND) filter at 10% (8.9 mW) was applied. Data collection and spectral analysis were conducted using LabSpec 6 software for precise evaluation of structural changes.

The surface characterization of the dental resin composite samples was conducted using a Mitutoyo SJ-201 Roughness Tester (Mitutoyo Europe GmbH, Germany). To determine the arithmetic average roughness (Ra), each specimen was analyzed through three-line measurements. The profilometer, featuring a 5 µm radius diamond tip, recorded two-dimensional surface profiles at a scanning speed of 0.5 mm/s, with a cut-off value of 0.8 mm and a total measuring length of 4 mm. Measurements were performed in three distinct areas on each sample, and the arithmetic mean of these readings was calculated to represent the Ra (peak-valley roughness) value.

Color measurements were performed using the VITA Easyshade V digital spectrophotometer (Vita Zahnfabrik, Bad Säckingen, Germany), designed for accurate and reliable shade determination of teeth and restorations. The color parameters L*, a*, and b* were analyzed according to the Commission Internationale de l'Eclairage (CIE) standards, while hue angle (°) and relative color saturation (C*) were derived from these values. Specimens were immersed daily for 20 minutes over 10 days in red wine (Fetească Neagră), black coffee (Nespresso), or Coca-Cola, followed by air drying. Color changes were recorded before and after immersion, and the total color difference (ΔE) was calculated using the formula:

$$\Delta E = ([\Delta a^*]2 + [\Delta b^*]2 + [\Delta L^*]2)1/2$$

A $\Delta E > 2.7$ indicated very distinct changes, $1.2 \leq \Delta E \leq 2.7$ denoted distinct changes, and $\Delta E < 1.2$ was considered non-distinct.

Statistical analysis of surface roughness, color change, and hardness of the Omnichroma composite before and after immersion in coffee (Nespresso), Coca-Cola, and red wine (Fetească Neagră) was performed using SPSS 23.0 software (SPSS, Chicago, IL, USA). One-way ANOVA and one-sample t-tests were used to compare the variations in these properties across the different staining solutions, with statistical significance set at $p < 0.05$.

RESULTS

The pH measurements of the immersion solutions revealed variations in acidity levels, which could influence the discoloration of the composite specimens. The coffee solution (Nespresso) exhibited a mildly acidic pH of 5.0, while the red wine (Fetească Neagră, Purcari, Romania) showed a more pronounced acidity with a pH of 3.5. Coca-Cola had the lowest pH value of 2.5, indicating a highly acidic environment.

For Omnichroma, the average diameter of droplets was recorded as 1.58 mm ± 0.05 mm for the first measurement set and 1.67 mm ± 0.03 mm for the second. The mean contact angle was 64.94° ± 4.82° in the first test and 76.97° ± 3.08° in the second, indicating variations in wettability under consistent conditions. The average droplet volume was 0.564 µL ± 0.079 µL in the first test and 0.877 µL ± 0.049 µL in the second. Corresponding three-phase points (r) and (l) were recorded as 3.6 mm ± 0.1 mm and 2.0 mm ± 0.1 mm for the first set and 3.7 mm ± 0.0 mm and 2.1 mm ± 0.0 mm for the second (Figure 2). The SFE analysis revealed the total surface free energy and its polar and dispersive contributions, aligning with the expected hydrophilic characteristics of the material.

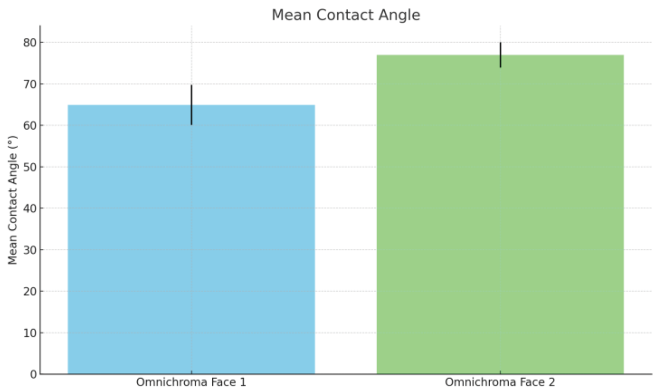

Figure 2. Mean Water Contact Angle of Omnichroma on Different Surface Textures

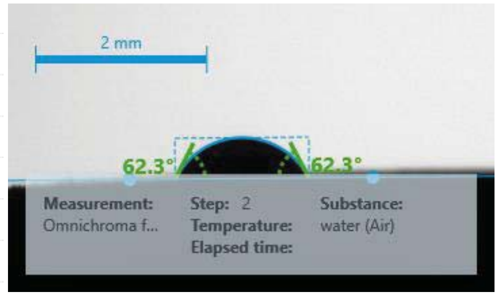

Figure 3. Example of Contact Angle Measurement for Omnichroma Composite

To determine the point of zero charge (PZC)—the pH at which the streaming potential reaches zero—a pH titration was conducted on the Omnichroma sample (Figure 3). The titration process began in an acidic medium using a 0.5% HNO_3 solution and gradually transitioned toward the basic region by incrementally adding NaOH solution. The results indicated that the PZC was observed at pH 3.8, suggesting that the composite material contains a significantly higher number of anionic ionized/ionizable groups compared to positively charged ones (Figure 4).

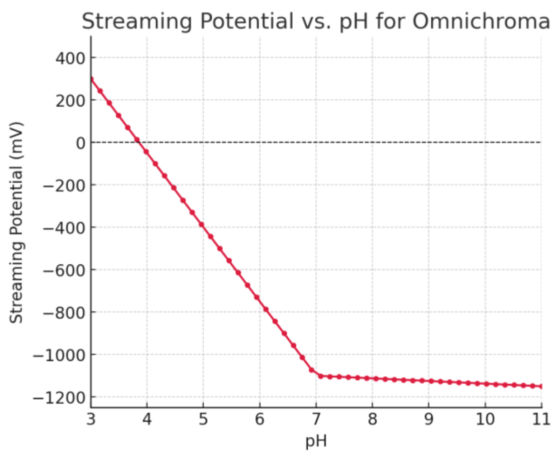

Figure 4. Streaming Potential vs pH for Omnichroma

Initially, Omnichroma exhibited a surface roughness (SR) of 1.06 ± 0.09. After 10 days of immersion, the roughness increased to varying degrees depending on the solution. Coffee immersion had the most pronounced effect, increasing the roughness to 1.53 ± 0.38, indicating a moderate abrasive impact. Red wine led to a slight increase, with a final SR of 1.15 ± 0.08, while Coca-Cola caused a minor increase to 1.21 ± 0.31, showing a less significant effect than coffee but comparable to red wine (Figure 5).

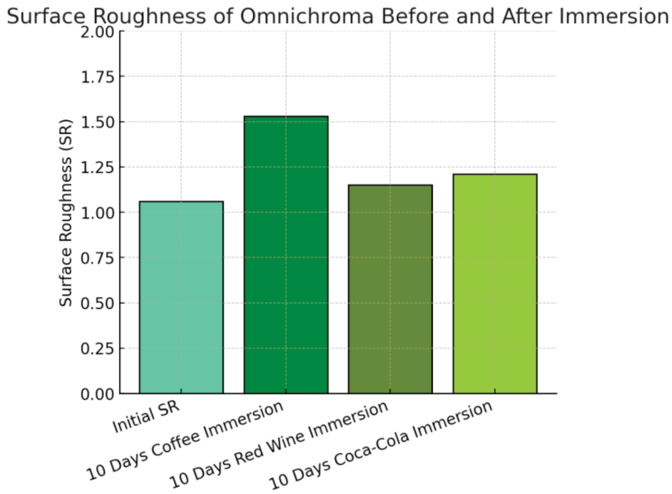

Figure 5. Surface Roughness of Omnichroma before and after immersion

Omnichroma exhibited the most significant color change in coffee (ΔE = 10.84 ± 1.03), a moderate color alteration in red wine (ΔE = 5.33 ± 0.27), and the least noticeable change in Coca-Cola (ΔE = 1.28 ± 0.44).

The Raman analysis revealed structural modifications in the Omnichroma composite resin after immersion in staining solutions. Compared to the control sample, notable variations in vibrational intensities were observed, particularly in the C=O stretching (1700 cm^{-1}) and C=C aromatic ring vibrations (1600 cm^{-1}). The coffee-immersed sample (A) exhibited the most pronounced spectral changes, indicating polymer network alterations. The Coca-Cola (B) and red wine (C) samples showed moderate shifts, suggesting minor interactions with the resin matrix. The control sample (D) maintained a stable vibrational profile, confirming the integrity of the unexposed composite (Figure 6).

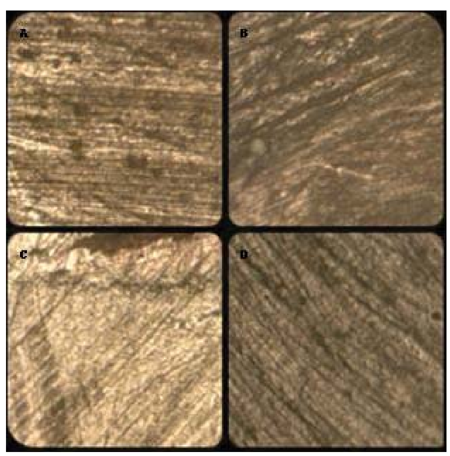

Figure 6. Surface Morphology of Omnichroma After Immersion in Staining Solutions: (A) Coffee, (B) Coca-Cola, (C) Red Wine, (D) Control

DISCUSSIONS

The pursuit of natural-looking restorations has recently led to the development of restorative systems that incorporate multiple shades and varying translucency levels, including enamel shade resin composites available in high, medium, and low value [13].

Single-shade universal resin composites are increasingly used in clinical practice due to their ability to simplify shade selection, provide good aesthetics, and minimize material waste from expired products [14]. For long-term success, resin-based composite restorations must exhibit optimal physical, mechanical, and biological properties to withstand the erosive and abrasive conditions of the oral environment [15]. While most existing studies primarily focus on the aesthetic properties of SsURCs, limited research has been conducted on their surface characteristics and stability. Therefore, this study aims to evaluate key properties of SsURCs, including contact angle, surface roughness, microhardness, zeta potential, Raman spectral characteristics, and colour stability before and after immersion in staining solutions (coffee and acidic carbonated beverage), providing a comprehensive analysis of their performance in clinical applications.

According to the manufacturer, Omnichroma does not contain pigments; instead, its colour properties rely on structural colour, a smart chromatic technology designed to control the optical characteristics of the resin composite. This innovation enables the composite to interact with light waves at specific frequencies, reflecting precise wavelengths within the tooth colour spectrum. To achieve this structural colour effect, the composite's filler must consist exclusively of uniformly sized spherical particles. Tokuyama's research has demonstrated that 260 nm spherical fillers generate the necessary a and b colour parameters to match natural teeth. Any variations in the size or shape of the filler material can disrupt or hinder the structural colour phenomenon, ultimately affecting the composite's shade-matching capability. Therefore, Omnichroma exclusively incorporates 260 nm spherical filler (Omnichroma Filler) to ensure optimal colour adaptation [16].

Our study results indicate that the varying pH levels of the immersion solutions may play a significant role in the staining susceptibility of the composite material, with more acidic environments potentially enhancing discoloration over time.

Several studies [17, 18] have reported an average critical surface roughness threshold of 0.2 µm, though no universally accepted value for surface roughness assessment currently exists. A clinical study by Jones et al. [19] found that patients could perceive surface irregularities when the mean roughness (Ra) reached 0.3 µm. In the present study, all tested composite resins exhibited a significant increase in mean surface roughness following immersion in staining solutions, suggesting potential implications for both aesthetic durability and patient-perceived texture changes. This increase in surface roughness may be attributed to the acidity of the beverages used, as well as the surface irregularities that develop during the finishing process.

The long-term color stability of commercial resin-based composite materials used in aesthetic restorative procedures remains a concern, as discoloration and poor color matching are among the primary reasons for restoration replacement [20]. The methodology employed in this study aligns with previous research utilizing spectrophotometry and the CIE Lab coordinate system*, which is widely recommended for dental applications. This system is particularly suited for detecting subtle color variations, offering advantages such as high sensitivity, repeatability, and objective measurement, making it an effective tool for assessing color stability in dental materials. The findings of this study align with previous research indicating that coffee induces greater discoloration in composite materials compared to other beverages [21, 22]. Bagheri et al. [23] reported that while cola compromises the surface integrity of composites due to its low pH, it does not cause as much discoloration as coffee and tea, as it lacks yellow dye pigments. Similarly, Sirin Karaarslan et al. [24] observed a decrease in L-values across all samples after an aging process, signifying darker shades in the tested composites. In the present study, values decreased in all Omnichroma samples following immersion in coffee, Coca-Cola, and red wine, confirming the darkening effect of these beverages. The results are consistent with previous studies [25, 26] analyzing color changes in composite resins exposed to staining solutions, further supporting the observation that discoloration leads to negative ΔL values, indicating a shift toward darker shades over time.

The color stability of resin-based composite materials is influenced by multiple factors, including the degree of monomer conversion and the chemical composition of the material. A higher degree of conversion results in fewer unreacted monomers, reduced solubility, and enhanced resistance to discoloration. In contrast, unconverted double carbon bonds trap residual monomers within the composite, making it more prone to staining. Additionally, hydrophilic organic matrices, which promote water absorption, can accelerate the degradation of the polymeric network, leading to the release of by-products such as formaldehyde and methacrylic acid, both of which contribute to discoloration. Increased water sorption further compromises color stability by expanding the free volume within the polymer, allowing greater diffusion of water molecules, which first degrades the material and subsequently leads to staining [6].

The Raman spectroscopy findings highlight the varying degrees of chemical interactions between Omnichroma and acidic staining solutions, influencing its surface composition and structural stability. Due to Omnichroma's structural color technology, which relies on diffraction and light scattering from its microscopic structure, the small color changes observed in this study may be even less perceptible in a clinical setting. Additionally, Omnichroma's resin matrix is primarily composed of urethane dimethacrylate (UDMA), a hydrophobic monomer known for its ability to enhance the water resistance of the material. This increased hydric stability reduces the likelihood of polymer degradation and minimizes further color alterations, contributing to the long-term aesthetic durability of the restoration [6, 27].

A key limitation of this study is the in vitro design, as laboratory conditions cannot fully replicate the complexities of the intraoral environment. Additionally, the research focused solely on single-shade universal resin composites, without including other universal composite types, such as multi-shade or bulk-fill composites. Another constraint was the absence of an aging process for the tested SsURC samples, which limits insights into their long-term performance. To gain a more comprehensive understanding, further in vivo studies are needed to evaluate the clinical effectiveness of SsURCs, particularly in terms of discoloration, wear resistance (including erosive, abrasive, and mechanical factors), bacterial adhesion, plaque accumulation, and potential toxicity.

CONCLUSIONS

This study demonstrated that acidic staining solutions significantly influence the surface properties and color stability of Omnichroma composite resin. Among the tested solutions, coffee had the most pronounced effect, causing the greatest increase in surface roughness and color change, followed by red wine and Coca-Cola, which exhibited lesser but still notable alterations. Raman spectral analysis indicated structural modifications in the composite's chemical matrix, while zeta potential measurements reflect the composite's surface charge behavior in acidic environments. Although Omnichroma's structural color technology enhances shade matching, its long-term stability in acidic conditions remains a concern. The findings highlight the need for further in vivo research to evaluate the composite's performance under real intraoral conditions, considering factors such as aging, wear resistance, bacterial adhesion, and mechanical degradation.

Conflicts of Interest

The authors declare no conflict of interest.

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