

**“VICTOR BABEȘ” UNIVERSITY OF MEDICINE AND PHARMACY  
FROM TIMIȘOARA**

**FACULTY OF DENTAL MEDICINE**

**DEPARTMENT 1**

**HAJDU ADRIAN IOAN**



# **PhD THESIS**

**CONSIDERATIONS REGARDING THE PERFORMANCE  
OF DENTAL COMPOSITE RESINS (DCR) IN THE ORAL  
ENVIRONMENT**

**– A B S T R A C T –**

Scientific Coordinator:

**PROF. UNIV. HABIL. DR. GĂLUȘCAN ATENA**

**T i m i ș o a r a**

**2 0 2 4**



Despite advances in oral healthcare, dental caries remains a significant global public health issue, affecting a large portion of the population, especially among disadvantaged groups. The chosen research topic focuses on dental caries management, emphasizing the dynamic process of enamel demineralization and remineralization. Modern caries management aims to diagnose caries risk early and implement preventive strategies to delay restorative treatments. This research is driven by the clinical need to improve dental restorative materials and adapt to global shifts in dental practices. Direct composite resins (DCRs) have gained popularity due to their aesthetic appeal and improved mechanical properties. However, these materials still face challenges like polymerization shrinkage and reduced abrasion resistance, leading to a failure rate of 3-11% annually. The study of DCRs is crucial for enhancing their longevity and performance in the oral environment, particularly against common beverages that patients consume. The research aligns with international efforts, such as the Minamata Convention, which advocates reducing amalgam use in dentistry, leading to a greater reliance on composite resins.

The primary objective of this thesis is to evaluate the properties of DCRs in varied oral environments, particularly focusing on the impact of dietary habits using in vitro models. The study aims to answer how resin-based composite materials change their initial properties when exposed to common beverages, thereby providing insights into their long-term performance.

The thesis is divided into two parts: a **General part**, which includes a literature review, and a **Special part**, which details personal contributions through three studies. These studies evaluate the surface characteristics, color stability, hydrophobicity, and microscopic surface changes of different dental composites when exposed to beverages like red wine, black coffee, and Coca-Cola.

The research employed a comprehensive in vitro methodology to assess the effects of acidic beverages on dental resin composites. The findings showed significant variations in the performance of the materials, highlighting the importance of considering patient dietary habits in material selection. The research provides valuable insights for improving the durability and aesthetic quality of dental restorations.

## **General Part:**

### **Chapter 1: History of Composite Resins**

RBCs are favored for their aesthetics, cost-effectiveness, and clinical performance. However, early materials like silicate cements had limitations. Dr. Rafael Bowen's development of the Bis-GMA monomer in 1962 revolutionized RBCs, enhancing their strength and aesthetics. The evolution from gold and amalgam to modern RBCs reflects advancements in aesthetics, safety, and performance. Concerns over mercury in amalgam have driven research toward safer alternatives. Research in the 1960s and 1990s led to significant improvements in RBCs, focusing on enhancing mechanical properties and adhesion. Fillers have evolved to improve strength, aesthetics, and radiopacity. Innovations in RBCs include reducing polymerization shrinkage, enhancing photo initiators, and developing bioactive materials to interact with the oral environment.

### **Chapter 2: The Mimicry of Composite Diacrylic Resins**

Biomimetics in dentistry aims to replicate the natural tooth's biomechanics, structure, and aesthetics. Modern dentistry focuses on minimally invasive techniques using bioinspired materials for remineralization and regeneration. RBCs must balance dimensional stability, stress distribution, aesthetic appearance, and biocompatibility. Inorganic fillers in RBCs enhance strength, while resin composites mimic natural dentin. The biomimetic approach focuses on preserving natural tooth structure and function while using materials that mimic natural biomechanics. RDCs are preferred for their minimal preparation requirements and strengthening potential. The elastic modulus (EM) and surface hardness (SH) of restorative materials are key factors in predicting clinical performance. RDCs with similar EM to dentin are preferred for stress distribution and reducing fracture risk. RDCs are ideal for addressing aesthetic concerns due to their ability to match the color and morphology of natural teeth. Long-term studies show high survival rates for RDC restorations.

### **Chapter 3: Color Measurement and Perception in Dentistry: CIELAB and CIEDE2000 Systems**

Accurate color measurement is crucial for achieving aesthetically pleasing dental restorations. The CIELAB and CIEDE2000 systems offer objective, standardized methods for

color matching, enhancing consistency and precision in dental practice. CIELAB Color Space describes color using three coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ), widely used for color specification and quality control in dentistry. CIEDE2000 Formula provides a more accurate assessment of color differences, aligning better with human perception and improving clinical outcomes in color matching and acceptability.

### **Special Part**

The primary objective of this PhD thesis is to evaluate the performance and characteristics of direct dental restoration resins (DCRs) under varied oral conditions influenced by patients' dietary habits using in vitro models. The central research question guiding this study is: "How do resin-based composite materials used in direct dental restorations change their initial properties in response to the oral environment, particularly due to the high consumption of certain beverages?" To address this question, the research focuses on three specific objectives: assessing the color changes of DCRs exposed to different beverages, examining surface changes in DCRs when exposed to acidic drinks, and evaluating the microscopic alterations in DCRs under these conditions.

The research consists of three studies:

- Study 1: Evaluates the color changes of three DCRs (Herculite Ultra XRV, G-ænial A'CHORD, and Omnicroma) in contact with commercial beverages (Red Wine, Black Coffee, and Coca-Cola) using spectrophotometric analysis.
- Study 2: Assesses the hydrophobicity and surface roughness of these dental composites after immersion in acidic beverages through an in vitro study.
- Study 3: Examines the microscopic surface changes of these DCRs when exposed to beverages using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS).

A total of 60 disk-shaped specimens were prepared from three different resin composites. Each composite material had 20 specimens, divided into control groups and groups immersed in coffee, wine, and Coca-Cola. The specimens were created using metallic molds and polymerized with an LED curing light, followed by polishing and ultrasonic cleaning. The composites tested were:

- Herculite Ultra XRV: A nanohybrid resin composite with barium glass fillers and silica nanoparticles.
- G-ænial A'CHORD: A hybrid composite with pre-polymerized resin fillers.

- Omnicroma: A supra-nanohybrid resin composite with silica-zirconia fillers.

The study involved immersing composite resin samples in black coffee, red wine, and Coca-Cola for 20 minutes daily over 10 consecutive days, with a control group maintained in similar conditions without beverage exposure. Specimens were kept in an incubator at 37°C to mimic the moist oral environment, with the solutions refreshed daily to maintain consistency.

***Study 1: Color Changes of DCR (Herculite Ultra XRV Ultra XRV, G-ænial A'CHORD, and Omnicroma) in contact with commercial beverages (Red Wine, Black Coffee and Coca-Cola)-a Spectrophotometric analysis***

Dental composite resins (DCRs) are popular for their aesthetic properties and strong adhesive bonding. Despite technological advancements, the degradation of composite materials remains a concern, particularly when exposed to various beverages that can cause surface erosion and discoloration. This study aimed to evaluate the color changes in three recent composite resins (Herculite Ultra XRV, G-ænial A'CHORD, and Omnicroma) after exposure to common beverages such as coffee, red wine, and Coca-Cola using spectrophotometric analysis. The study used a spectrophotometer to measure color changes in the composites, employing the CIELAB color system to calculate the color difference ( $\Delta E$ ). The measurements were taken on days 4 and 10 of the immersion period. Statistical analysis, including MANOVA and Student's t-test, was used to compare the mean data and determine the significance of the differences observed.

The results indicated that all tested composites experienced significant color changes after immersion in the beverages. Key findings include: Herculite Ultra XRV showed the highest  $\Delta E$  in Coca-Cola after 4 days and in red wine after 10 days, G-ænial A'CHORD exhibited the most significant discoloration in coffee and red wine, particularly after 10 days and Omnicroma displayed the highest color change in coffee, with Coca-Cola causing the least discoloration across all materials.

The statistical analysis confirmed significant differences in color stability among the composites, with each material showing varying sensitivity to different staining solutions. The study highlights the importance of understanding the impact of common dietary beverages on the long-term aesthetic stability of dental restorations. The findings align with previous research, demonstrating that beverages like coffee, red wine, and Coca-Cola can cause substantial discoloration in composite resins. The study also emphasizes the need for careful material selection based on patient lifestyle and consumption habits to maintain the aesthetic

quality of dental restorations. All tested composites experienced significant color changes after exposure to staining solutions, with coffee and red wine causing the most pronounced effects. Herculite Ultra XRV showed moderate color changes across all solutions, with Coca-Cola and red wine having the most significant impact. G-ænial A'CHORD was most affected by coffee and red wine, indicating the highest level of discoloration among the tested materials. Omnichroma displayed the highest color change in coffee, with Coca-Cola resulting in the least discoloration. The study confirms the susceptibility of dental composites to discoloration from acidic and staining substances, which can compromise clinical outcomes. The results underscore the importance of considering the effects of common beverages on the aesthetic durability of dental composites, highlighting the need for ongoing research to improve material performance in diverse oral environments.

***Study 2: Assessment of the hydrophobicity and examination of the surface roughness of dental composite resins after immersion in three acidic beverages (red wine, black coffee, and Coca-Cola) in an in vitro study.***

The oral cavity is a complex ecosystem where dental caries often results from the buildup of polymicrobial biofilms on dental surfaces. The attachment of bacteria to both natural teeth and dental restorative materials is a significant factor in the development of caries and periodontal diseases. Despite technological advancements, recurrent caries remains a primary reason for restoration failure, often linked to biofilm formation on dental restorations. Resin composites, widely used for cavity restoration due to their aesthetic appeal, low toxicity, and enhanced performance, are susceptible to surface changes when exposed to various oral conditions, including acidic beverages. This study aims to evaluate the surface roughness and hydrophobicity of three dental composite materials—Herculite Ultra XRV, G-ænial A'CHORD, and Omnichroma—after immersion in acidic beverages (red wine, black coffee, and Coca-Cola). The null hypothesis posits that no significant differences will be observed in the surface characteristics of these materials after exposure to the acidic beverages.

The study evaluated three commercial dental composite resins: Herculite Ultra XRV, a nanohybrid resin composite known for its high aesthetic properties and mechanical strength, G-ænial A'CHORD, a hybrid resin composite recognized for ease of handling and superior polishability and Omnichroma, a monocolour composite that adapts to surrounding tooth colors. A total of 60 disk-shaped samples (20 for each material) were fabricated. These specimens were polished and immersed in red wine, black coffee, or Coca-Cola for 10 days to simulate real-life conditions. The polishing process was standardized to ensure uniformity across all samples. To assess hydrophobicity, contact angles were measured using a Drop Shape

Analyzer. Lower contact angles indicated more hydrophilic surfaces. The surface charge of the composites was measured to determine their electrokinetic properties. Surface roughness was measured using a profilometer, with roughness values recorded before and after immersion. The acidity of the beverages was measured using a pH meter to evaluate the potential for surface degradation. The results indicate that Herculite Ultra XRV showed the highest mean contact angle (79.46°), indicating a more hydrophobic surface. G-ænial A'CHORD had a mean contact angle of 73.22°, suggesting moderate hydrophobicity. Omnichroma displayed the lowest mean contact angle (64.94°), indicating a more hydrophilic surface. The materials exhibited negative surface charges, with Omnichroma having the most negative streaming potential, suggesting a stronger negative charge compared to the others.

Herculite Ultra XRV showed varied changes in roughness after immersion, with red wine causing the most significant increase. G-ænial A'CHORD exhibited a substantial increase in roughness after immersion in Coca-Cola, indicating significant surface degradation. Omnichroma experienced moderate increases in roughness, with coffee having the most pronounced effect. The beverages had varying acidity levels, with Coca-Cola being the most acidic (pH 2.4), followed by red wine (pH 3.5), and black coffee (pH 5.6). The acidic nature of these beverages likely contributed to the observed surface changes.

The study highlights the importance of surface characteristics, such as roughness and hydrophobicity, in the clinical success of dental restorations. Smooth and polished surfaces are essential for aesthetics, hygiene, and the prevention of plaque accumulation, which can lead to secondary caries. The study found that different composites respond differently to acidic environments, with Herculite Ultra XRV demonstrating the best resistance to staining and surface degradation due to its higher hydrophobicity. G-ænial A'CHORD was most susceptible to surface roughness changes, particularly after exposure to Coca-Cola, while Omnichroma was more prone to staining due to its lower hydrophobicity. The study also emphasizes the significance of polishing techniques in determining the surface quality of composites. Proper polishing can enhance the material's resistance to discoloration and bacterial adhesion, thereby improving its longevity. In conclusion, surface characteristics significantly impact the performance and durability of dental restorative materials. Exposure to acidic beverages alters the surface roughness and color stability of dental composites, with each material responding differently. Herculite Ultra XRV exhibited superior hydrophobicity, making it more resistant to staining. G-ænial A'CHORD showed the highest increase in surface roughness after exposure to Coca-Cola, indicating its vulnerability to surface degradation. Omnichroma was the most prone to staining, especially when exposed to coffee. The study underscores the need for selecting materials that can withstand acidic environments to maintain aesthetic and functional integrity over time. Future research should explore the



incorporation of antibacterial agents into composites and investigate the effects of various foods and beverages on dental materials using more realistic in vitro models that mimic oral conditions.

***Study 3: Evaluation of microscopic surface changes DCR (Herculite Ultra XRV Ultra XRV, G-ænial A'CHORD, and Omnichroma) when exposed to common beverages such as coffee, red wine, and Coca-Cola. Using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)***

Despite advances in dental composite materials, degradation remains a significant issue, particularly when these materials are exposed to various acidic beverages. Such exposure can lead to surface erosion, compromising the long-term integrity and aesthetic appeal of restorative materials. The oral environment, with its varying pH levels and exposure to different substances, poses challenges to the durability of these materials. This study aimed to evaluate the microscopic surface changes and elemental composition of three dental composites—Herculite Ultra XRV, G-ænial A'CHORD, and Omnichroma—after immersion in commonly consumed beverages: coffee, red wine, and Coca-Cola. SEM and EDS analyses were used to assess the extent of degradation and changes in surface morphology.

The study focused on three types of dental composites: Herculite Ultra XRV, a nanohybrid resin composite, G-ænial A'CHORD: A hybrid resin composite and Omnichroma, a supra-nanohybrid resin composite. SEM Analysis was used to evaluate the surface morphology of the composites after immersion in the beverages. EDS Analysis employed to determine the elemental composition of the composites before and after exposure to the beverages. The study found significant changes in both the surface morphology and elemental composition of the dental composites after 10 days of immersion in the beverages. In case of Herculite Ultra XRV SEM images revealed increased surface roughness and degradation, particularly after immersion in Coca-Cola. Coffee and red wine also caused surface damage, though to a lesser extent. As Regarding the elemental Composition, EDS analysis showed a significant decrease in carbon and oxygen content after immersion in all three beverages, with a notable increase in sulfur and barium, especially after exposure to Coca-Cola. In case of G-ænial A'CHORD, the surface morphology for this composite showed significant surface roughness and porosity after exposure, particularly to red wine and coffee. Coca-Cola caused substantial surface deposits. The composite experienced a marked decrease in oxygen and aluminum content, with an increase in carbon, especially in samples exposed to Coca-Cola. In case of Omnichroma's surface morphology, Coca-Cola caused the most pronounced surface degradation, with increased roughness and erosion. Coffee also caused notable

surface changes. EDS analysis revealed a significant increase in carbon content after Coca-Cola immersion, with a sharp decrease in silicon, indicating severe compositional changes. Among the three composites, each material exhibited distinct susceptibilities to the beverages: Herculite Ultra XRV was most affected by red wine, showing the greatest reduction in carbon and oxygen, G-ænial A'CHORD showed the most significant degradation when exposed to coffee and Omnichroma was most vulnerable to Coca-Cola, with severe changes in elemental composition and surface morphology.

The study highlights the critical impact of acidic beverages on the degradation and discoloration of dental composites. The SEM and EDS analyses provided detailed insights into how different beverages interact with composite materials, leading to significant surface and compositional changes. These findings emphasize the importance of selecting dental materials based on patient dietary habits to enhance the longevity and aesthetics of restorations. The study also acknowledges its limitations, including the short immersion period and the fact that in vitro conditions may not fully replicate the complex oral environment. Future research should explore longer-term effects, include a wider range of composite materials, and consider additional factors such as oral hygiene practices.

In conclusion, the study demonstrated that coffee, red wine, and Coca-Cola significantly degrade and discolor dental composites. Each composite material showed varying degrees of susceptibility to the beverages, with Herculite Ultra XRV most affected by red wine, G-ænial A'CHORD by coffee, and Omnichroma by Coca-Cola. SEM revealed extensive surface roughness and degradation, especially after Coca-Cola immersion, while EDS showed significant changes in elemental composition, particularly in carbon and oxygen content. The findings underscore the need for personalized material selection based on patient lifestyle to improve the longevity and aesthetics of dental restorations. The in vitro conditions and short immersion period limit the study's applicability to real-world scenarios. Future research should address these limitations to better understand the long-term performance of dental composites.

The thesis contributes to dental material science by exploring how common dietary substances impact the durability and aesthetic reliability of dental composites. The findings align with global initiatives advocating for safer dental materials, such as those promoted by the Minamata Convention. The research also opens avenues for future studies, including the need for longer-term and more comprehensive evaluations of dental composites under varied conditions.

The thesis introduces innovative methodologies, including the integration of spectrophotometric analysis and contact angle measurements, to assess the effects of acidic beverages on dental composites. This approach provides a comprehensive evaluation of how these materials react to common dietary challenges. The research also suggests new avenues for developing dental materials with enhanced resistance to acidic environments and improved longevity.

The research confirmed that commonly consumed beverages significantly impact dental composites, with material-specific susceptibilities. SEM and EDS analyses revealed significant surface degradation and changes in elemental composition, highlighting the importance of careful material selection in clinical practice. The findings suggest the need for personalized material choices in dentistry and recommend further research to explore the long-term effects of various beverages on a broader range of dental composites under real-world conditions.