THE EFFECT OF AGEING ON THE SURFACE ROUGHNESS OF VARSEOSMILE CROWN PLUS AND LITHIUM DISILICATE

SUHAENIH BINTI BUDIN

FACULTY OF DENTISTRY
UNIVERSITY OF MALAYA
KUALA LUMPUR

2024

THE EFFECT OF AGEING ON THE SURFACE ROUGHNESS OF VARSEOSMILE CROWN PLUS AND LITHIUM DISILICATE

SUHAENIH BINTI BUDIN

RESEARCH REPORT SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ORAL SCIENCE (PROSTHODONTICS)

FACULTY OF DENTISTRY
UNIVERSITY OF MALAYA
KUALA LUMPUR

2024

UNIVERSITY OF MALAYA
ORIGINAL LITERARY WORK DECLARATION

Name of Candidate: Suhaenih Binti Budin

Matric No : S2003478

Name of Degree : Master of Oral Science

Title of Research Report: The Effect of Ageing on the Surface Roughness of VarseoSmile Crown plus and Lithium Disilicate

Field of Study : Dental Material

I do solemnly and sincerely declare that:

(1) I am the sole author/writer of this Work;
(2) This Work is original;
(3) Any use of any work in which copyright exists was done by way of fair dealing and for permitted purposes and any excerpt or extract from, or reference to or reproduction of any copyright work has been disclosed expressly and sufficiently and the title of the Work and its authorship have been acknowledged in this Work;
(4) I do not have any actual knowledge nor do I ought reasonably to know that the making of this work constitutes an infringement of any copyright work;
(5) I hereby assign all and every rights in the copyright to this Work to the University of Malaya ("UM"), who henceforth shall be owner of the copyright in this Work and that any reproduction or use in any form or by any means whatsoever is prohibited without the written consent of UM having been first had and obtained;
(6) I am fully aware that if in the course of making this Work I have infringed any copyright whether intentionally or otherwise, I may be subject to legal action or any other action as may be determined by UM.

Candidate's Signature Date:

Subscribed and solemnly declared before,

Witness's Signature Date:

Name:

Designation:

ABSTRACT

This in-vitro study aims to investigate the effect of artificial ageing on the surface roughness of two groups: 3D printed ceramic-filled hybrid material (VarseoSmile Crown plus) (VSplus group) and milled lithium disilicate glass ceramic (IPS e.max CAD) (Emax group). A total of 20 samples with a sample size of n=10 per group were determined, accounting for a 15% dropout rate. The sample preparation involved fabricating rectangular-shaped samples of both groups with dimensions of 14×12×3 mm. For VSplus (Bego, Bremen, Germany), the samples were 3D printed using a 3D printer (Ackuretta SOL 3D printer), followed by a cleaning process with ethanol in a washing unit (Ackuretta CLEANI) and post-curing under ultraviolet light curing unit (Ackuretta CURIEplus). Meanwhile, the Emax (Ivoclar–Vivadent, Schaan, Liechtenstein) samples were fabricated using a wet dental milling machine (Aidite AMW 400) and sintered in a dental furnace (Ivoclar Vivadent Programat P310) to achieve their final properties. All samples were finished with silicon carbide abrasive papers (Beta 2 grinder-polisher machine, Buehler, Germany) and polished according to the manufacturers' guidelines to achieve a smooth surface. The artificial ageing is done by thermocycling machine (Zecttron Sdn Bhd, Malaysia) to simulate the thermal stresses experienced in the oral cavity. This process involved subjecting the samples to 5000 cycles between temperatures of 5°C and 55°C, with 20secs dwell times and 10secs transfer times. Surface roughness (*Ra* value) was measured using an optical 3D surface analyser (G4e, Alicona Imaging GmbH, Raaba, Austria) before and after ageing. A representative sample from each group was randomly selected and examined with Field Emission Scanning Electron Microscopy (FE-SEM) (Quanta FEG 450, Thermo Fisher Scientific, Brno, Czech Republic). Paired t-test was used to compare the mean *Ra* values of each group before and after ageing. Independent sample t-test was used to compare the mean *Ra* value between the two groups before and after ageing. The results showed a significant increase in surface roughness for both materials after ageing. The mean *Ra* value for VSplus significantly increased from 0.145 µm to 0.168 µm (p value<0.05), while for Emax, it increased from 0.223 µm to 0.232 µm (p value<0.05). Meanwhile, the comparison of the *Ra* value between the two groups before and after ageing shows a significant difference (p value<0.05), with the *Ra* value of VSplus being lesser than Emax, both before and after ageing. However, VSplus exhibit higher changes in mean *Ra* value before and after ageing, which is 0.023 µm changes in VSplus and only 0.009 µm changes in Emax. SEM images revealed notably increased roughness and microstructural changes in the surface topography of both groups after ageing. The findings of this study suggest that while both groups exhibit increased surface roughness due to artificial ageing, they were able to maintain their surface integrity near acceptable clinical thresholds.

Keywords: Surface roughness, 3D printed, SEM

ABSTRAK

Tujuan kajian in-vitro ini adalah untuk mengkaji kesan penuaan buatan terhadap kekasaran permukaan dua kumpulan bahan yang berbeza iaitu bahan percetakan 3D hibrid seramik (VarseoSmile Crown plus) (kumpulan VSplus) dan seramik kaca litium disilika yang melalui proses *milling* (IPS e.max CAD) (kumpulan Emax). Sebanyak 20 sampel dengan saiz sampel n=10 setiap kumpulan telah ditentukan dengan mengambil kira kadar penurunan sebanyak 15%. Penyediaan sampel berbentuk segi empat tepat untuk kedua-dua kumpulan dengan dimensi 14×12×3 mm. Bagi VSplus (Bego, Bremen, Germany), sampel dicetak dengan percetakan 3D (Ackuretta SOL 3D printer), diikuti dengan proses pembersihan menggunakan etanol (Ackuretta CLEANI) dan pendedahan di bawah cahaya ultraungu (Ackuretta CURIEplus). Sementara itu, sampel Emax (Ivoclar–Vivadent, Schaan, Liechtenstein) dihasilkan menggunakan mesin proses milling basah (Aidite AMW 400) dan disinter dalam mesin relau pergigian (Ivoclar Vivadent Programat P310) untuk mencapai sifat akhir mereka. Semua spesimen disiapkan dengan kertas pasir silikon karbida (Beta 2 grinder-polisher machine, Buehler, Germany) dan digilap mengikut panduan pengeluar untuk mencapai permukaan yang licin. Kaedah penuaan buatan menggunakan mesin *thermocycling* (Zecttron Sdn Bhd, Malaysia) untuk mensimulasikan tekanan haba yang dialami dalam rongga mulut. Proses ini melibatkan pendedahan sampel kepada 5000 kitaran antara suhu 5°C dan 55°C, dengan masa rendaman 20 saat dan masa pemindahan 10 saat. Kekasaran permukaan (nilai *Ra*) diukur menggunakan mesin penganalisa optikal 3D (G4e, Alicona Imaging GmbH, Raaba, Austria) sebelum dan selepas proses penuaan. Wakil setiap sampel dari setiap kumpulan dipilih secara rawak dan diperiksa menggunakan mesin *Field Emission Scanning Electron Microscopy* (FE-SEM) (Quanta FEG 450, Thermo Fisher Scientific, Brno, Czech Republic). Ujian *paired t-test* digunakan untuk membandingkan nilai purata *Ra* setiap kumpulan sebelum dan selepas proses penuaan.

Ujian *independent t-test* digunakan untuk membandingkan nilai purata *Ra* antara dua kumpulan sebelum dan selepas proses penuaan. Keputusan menunjukkan peningkatan yang ketara dalam kekasaran permukaan bagi kedua-dua bahan selepas proses penuaan. Nilai purata *Ra* untuk VSplus meningkat daripada 0.145 µm kepada 0.168 µm (nilai $p<0.05$), manakala untuk Emax, ia meningkat dari 0.223 µm kepada 0.232 µm (nilai $p<0.05$). Sementara itu, perbandingan nilai *Ra* antara dua kumpulan sebelum dan selepas penuaan menunjukkan perbezaan yang ketara (nilai $p<0.05$), dengan nilai *Ra* VSplus adalah lebih rendah berbanding Emax, sebelum dan selepas proses penuaan. Walau bagaimanapun, VSplus menunjukkan perubahan nilai purata *Ra* yang lebih tinggi daripada sebelum proses penuaan kepada selepas proses penuaan, iaitu perubahan 0.023 µm bagi VSplus dan hanya 0.009 µm bagi Emax. Imej SEM mendedahkan peningkatan kekasaran dan perubahan mikrostruktur dalam topografi permukaan kedua-dua kumpulan selepas proses penuaan. Penemuan kajian ini mencadangkan bahawa walaupun kedua-dua kumpulan menunjukkan peningkatan kekasaran permukaan akibat proses penuaan buatan, keduanya mampu mengekalkan integriti permukaan kepada nilai kritikal kekasaran permukaan dalam situasi klinikal.

Kata kunci: Kekasaran permukaan, percetakan 3D, SEM

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful, I express my profound gratitude to Allah for His blessings throughout the completion of this research. I also seek blessings for our Prophet Muhammad S.A.W, his family, and his companions.

I wish to express my deepest appreciation to all those who have provided invaluable support and guidance throughout the progress of this research report.

My heartfelt thanks go to my main supervisor, Dr. Aeman Elkezza, for his exceptional guidance, insightful feedback, and unwavering support. His constructive critiques and advice have significantly enhanced the quality of this work.

Additionally, I extend my gratitude to both of my co-supervisors, Dr Selva Malar Munusamy and A/P Dr. Mohideen Salihu Farook, for their assistance and encouragement. Their perspectives and suggestions have been pivotal to the progress of this research.

Special acknowledgement is due to Dr. Alias Abd Aziz for his expert guidance, particularly in the statistical analyses that were crucial to this study.

I would also like to express my appreciation to the staff of the Biomaterial Lab team, specifically Mdm. Helen-Ng Lee Ching, Pn. Zarina Binti Idris and Pn. Sumiati Binti Pool for their invaluable guidance, cooperation, and assistance. In addition, I am grateful to the laboratory technicians in the ceramic and prosthodontic labs for their expertise and guidance. Furthermore, my gratitude extends to my colleagues, seniors, and friends for their support, encouragement, and valuable advice.

Last but not least, I am profoundly grateful to my family for their unwavering support and patience. A particular note of thanks goes to my spouse, Taufik Bin Ishak, whose dedicated care for our children has enabled me to focus on my research. Their understanding and encouragement have been a source of strength throughout this process.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS AND ABBREVIATIONS

3D : Three Dimensional

AM : Additive Manufacturing

CAD/CAM : Computer-Aided Design/ Computer-Aided Manufacturing

Emax : IPS e.max CAD

FDM : Fused Deposition Modelling

FESEM : Field Emission Scanning Electron Microscopy

ISO : International Standard Organization

LCD : Liquid Crystal Display

MJ : Material Jetting

Ra : Average Surface Roughness Value

SD : Standard Deviation

SEM : Scanning Electron Microscopy

SLA : Stereolithography

SLM : Selective Laser Melting

SLS : Selective Laser Sintering

SM : Subtractive Manufacturing

STL : Standard Tessellation Language

VSplus : VarseoSmile Crown plus

List of Appendices

Appendix A . **Results of surface roughness assessment**

CHAPTER 1: INTRODUCTION

1.1 Introduction

Ceramic dental materials have become essential in contemporary restorative dentistry because of their superior aesthetic and mechanical qualities. Indirect restorations should be biocompatible and should be designed to reduce plaque accumulation, which is critical in maintaining an optimal environment at the gingival-prosthesis interface (Hisbergues and Vendeville, 2009 ; Zarone et al., 2016). Ceramic dental materials have been designed to provide unique chemical and mechanical properties suited to specific clinical requirements.

Lithium disilicate has gained popularity for anterior and posterior teeth restorations. This material is highly valued for its exceptional aesthetic appearance, which closely mimics the natural translucency of tooth enamel, and its mechanical properties, such as high flexural strength and fracture toughness. Lithium disilicate restorations are known for their excellent biocompatibility, minimal plaque accumulation, and favourable interaction with soft tissues, which create an ideal gingival-prosthesis interface (Shi et al., 2022). These attributes make lithium disilicate a versatile and reliable material for various dental restorations, ensuring functional and aesthetic outcomes.

CAD/CAM (Computer-Aided Design/Computer-Aided Manufacturing) technology has revolutionised the fabrication of dental restorations. CAD/CAM technology can be broadly categorised into subtractive manufacturing (milling/grinding) and additive manufacturing (3D printing), each offering distinct advantages. Subtractive manufacturing (SM) involves the removal of material from a solid block to achieve the desired shape. It is widely used for milling crowns and metal frameworks because it produces smooth finishes and precision fit. On the other hand, additive manufacturing (AM) builds objects layer-by-layer, allowing for intricate designs and material efficiency (ISO, 2021).

Fabricating indirect restoration using CAD/CAM technology has streamlined the production process, reducing human error, and shortening production times, contributing to the growing adoption of these technologies in dental practices. The result is a more efficient workflow that enhances the consistency and quality of dental restorations (Nassani et al., 2021). Furthermore, the variety of printable materials keeps growing, encompassing resin, metals, and ceramics-impregnated resin (Zhou et al., 2024). Ceramic AM technologies are very new and are still in the experimental stages in many fields, compared to metal and resin AM technologies, which have been available for a longer time and have many uses in several disciplines.

The newly introduced resin-matrix ceramics for 3D printed dental materials combine the advantages of organic and inorganic phases of parent ceramics and resins. It offers a shock-absorbing ability due to its modulus of elasticity, which is very close to dentine (Awada & Nathanson, 2015). The improved fracture resistance makes it advisable in areas with high occlusal loads (Matzinger et al., 2019).

VarseoSmile Crown plus (VSplus) is a ceramic-filled hybrid material designed for 3D printing of permanent restorations. This material suits single crowns on natural teeth or implant abutments. According to the manufacturer, VSplus offers easy handling, does not require mixing or shaking, and can be easily ground and polished with standard dental tools (BEGO, 2020).

A critical factor influencing the longevity and performance of dental restorations is the surface roughness of the materials used. Surface roughness can significantly affect the aesthetic quality of the restoration, as well as its susceptibility to bacterial adhesion, wear resistance, biocompatibility, and overall durability (Zafar et al., 2020). Understanding how ageing processes affect the surface characteristics of dental restorative materials is essential for ensuring the durability of prosthetic devices and improving patient satisfaction and oral health outcomes (Temizci & Bozoğulları, 2024).

This study aims to evaluate how the surface roughness of lithium disilicate and VSplus changes under artificial ageing conditions. Factors such as thermocycling, which simulates the temperature fluctuations that restorations experience in the oral cavity, will be considered to mimic the challenges these materials face over time. Surface roughness is a critical parameter that influences the aesthetic and functional aspects of dental crowns, and it is susceptible to the effects of ageing. By methodically assessing the changes in surface roughness, this study aims to provide significant insights into the long-term performance of these materials. The findings will help clinicians make well-informed decisions regarding the most suitable materials for their patients, ultimately improving the quality and longevity of dental restorations.

1.2 Aim of study

This in-vitro pilot study compares the surface roughness of 3D printed ceramic-filled hybrid material (Varseosmile Crown plus) with milled lithium disilicate glass ceramic (IPS e.max CAD) before and after inducing artificial ageing.

1.3 Objectives

1. To assess the surface roughness of the 3D printed crown material (Varseosmile Crown plus) before and after inducing artificial ageing.
2. To assess the surface roughness of the milled lithium disilicate crown material (IPS e.max CAD) before and after inducing artificial ageing.
3. To compare the surface roughness of the 3D printed (Varseosmile Crown plus) and milled lithium disilicate (IPS e.max CAD) crown material, before and after inducing artificial ageing.
4. To compare the difference between the SEM images of the 3D printed (Varseosmile Crown plus) and milled lithium disilicate (IPS e.max CAD) crown material, before and after inducing artificial ageing.

1.4 Null Hypothesis

The null hypothesis was:

There would be no difference in the surface roughness in between groups and within the same group of the 3D printed crown material (VarseoSmile Crown plus) and milled lithium disilicate crown material (IPS e.max CAD) before and after inducing artificial ageing.

CHAPTER 2: LITERATURE REVIEW

2.1 Current Trends in CAD/CAM Dental Materials and Digital Printing

CAD/CAM technology has revolutionised dentistry by customising and personalising dental prostheses to fit each patient's unique anatomical and aesthetic needs. This technological advancement ensures that prostheses are fabricated with greater precision and reliability, resulting in fewer errors than conventional methods (Nassani et al., 2021; Zhou et al., 2024) As the field continues to evolve, CAD/CAM systems are being developed to handle the milling or 3D printing of multi-material prosthetics, combining the benefits of different materials for optimal strength, aesthetics, and functionality (Ellakany et al., 2022; Grzebieluch et al., 2021).

Integrating SM and AM technologies into dental practices' digital workflow has streamlined dental restorations' fabrication process. The digital workflow starts acquiring data with intraoral scanning to capture precise digital impressions of the patient's teeth. These digital impressions are then uploaded to CAD software, which aids in designing the prosthesis from a 3D virtual model created from the digital impression. The model is subsequently manufactured using either subtractive or additive techniques (Joda et al., 2017). This workflow enhances accuracy and significantly reduces the time for prosthesis fabrication.

Clinical applications of these technologies are vast and include the production of crowns, bridges, inlays, onlays, veneers, and even full-mouth restorations. The ability to create highly accurate and aesthetically pleasing restorations with minimal manual intervention has improved patient satisfaction and clinical outcomes (Patel et al., 2022). Furthermore, the digital workflow allows for better communication between dental professionals and laboratories, ensuring the final restoration meets each patient's specific requirements.

Material science advancements have also played a critical role in the evolution of SM and AM technologies in dentistry. Innovations in ceramic and resin materials have led to the development of high-strength, aesthetically superior, and durable dental prosthetics. For example, lithium disilicate glass ceramics are widely used due to their excellent optical properties and high flexural strength (Zarone et al., 2016). Similarly, advancements in polymer chemistry have resulted in the development of high-performance composite resins that can be used in both SM and AM processes (Çakmak, et al., 2022).

2.1.1 Subtractive Manufacturing (SM) in Dentistry

SM with milling machines has been fundamental in dental prosthesis manufacture. This process involves creating prostheses with fewer porosities and a more homogenous consistency from solid materials such as blocks and discs (Abduo et al., 2014). The pioneering system in this domain was the CEREC® system (Sirona Dental Systems, Bensheim, Germany), introduced in 1985 (Duret et al., 1988). This system gained significant popularity and, depending on the restorative material, could construct restorations using two different milling techniques: soft and hard (Duret et al., 1988).

However, these subtractive methods, which utilise diamond burs at high speeds to achieve the final restoration shape, can create surface characteristics with increased roughness in dental ceramics (Fraga et al., 2017; Mota et al., 2017). Given the widespread use of CAD/CAM technology and lithium disilicate glass ceramics in dental practice, it is essential to consider the effects of ceramic processing. Lithium disilicate is highly favoured for its excellent mechanical properties and aesthetics. It is ideal for various restorations, including crowns, veneers, and bridges (Zarone et al., 2016)

Laboratory studies employing samples under these circumstances are encouraged to understand better clinical implications (Schestatsky et al., 2019; Zarone et al., 2016). It is recommended that samples be milled in a CAD/CAM system to analyse more relevant clinical scenarios even though the cost of sample fabrication increased due to equipment and software limitations (Fraga et al., 2017; Pilecco et al., 2021).

2.1.2 Additive Manufacturing (AM) in Dentistry

The advancement of CAD/CAM technology encompassing a broad spectrum of restorative materials has paved the way for dentistry's AM alongside SM. AM encompasses 3D printing technologies, where a 3D object is created by fusing liquid or powder materials layer by layer. This process involves deposition in the XY plane as a cross-sectional slice on a movable platform along the Z-axis (Naseer et al., 2021). AM technologies include digital light processing (DLP), stereolithography (SLA), material jetting (MJ), fused deposition modelling (FDM), selective laser melting (SLM), and selective laser sintering (SLS) (G. T. Kim et al., 2022; Revilla-León & Özcan, 2017; Zhou et al., 2024).

3D printing has addressed some of the limitations of CAD/CAM milling methods, allowing for more complex restorations with greater precision and less material waste (Abduo et al., 2014; Alharbi et al., 2016). However, this process has some drawbacks, including polymerisation shrinkage, higher machine and resin costs compared to conventional methods, surface roughness from the layered addition of resin, and the creation of toxic resin waste. (Ellakany et al., 2022)

Various materials have been utilised to create 3D dental prosthetics, including resin-based and metal-based materials. Resin-based materials, including those used in 3D printed dentures, offer significant advantages, including customizability, improved fit, and patient comfort (Tahayeri et al., 2018). These materials are often biocompatible and can be processed to produce highly detailed and accurate prosthetics. Moreover, using photopolymerizable resins in DLP and SLA technologies allows for rapid production and reduced material waste (Chaudhary et al., 2023). Metal-based materials have also been employed in additive manufacturing for dental applications. SLM and SLS are standard techniques used to fabricate metal dental components, such as frameworks for removable partial dentures and implant abutments (Revilla-León & Özcan, 2017).

Composite resins introduced for fixed definitive prostheses are among the latest restorative materials in additive manufacturing (Çakmak, et al., 2022). VSplus is a light-cured tooth-coloured ceramic-filled hybrid resin material based on methacrylic acid esters. Its ability to withstand high occlusal forces makes it suitable for inlays, onlays, veneers, and permanent crowns (BEGO, 2020). Introducing resin-matrix ceramics combines the advantages of organic and inorganic phases of ceramics and resins. It offers shock-absorbing ability due to its modulus of elasticity, which is very close to that of dentin and friendly to opposing enamel antagonists. The improved fracture resistance makes it advisable in areas with high occlusal loads (Albrecht, 2020; BEGO, 2020; Matzinger et al., 2019).

2.2 Surface Characterization of Dental Materials

2.2.1 Surface Roughness

The surface properties of dental restoration can affect its roughness, bonding properties, strength, and wear. A smooth surface enhances the aesthetic qualities of a dental restoration. It significantly affects its resistance to plaque accumulation, and it is essential for optimum performance and occlusal harmony (Bollen et al., 1997b; Özarslan et al., 2022; Tekçe et al., 2018). Surface roughness has been extensively studied, particularly concerning the ageing process of various dental materials and its significance in measuring material durability over time (Tekçe et al., 2018; Yuan et al., 2018).

2.2.2 Measurement of Surface Roughness (*Ra* value)

Surface roughness is typically quantified using *Ra* value (ISO, 1997). *Ra* (average roughness) is the arithmetic average of the absolute values of the profile height deviations from the mean line, recorded within the evaluation length, which provides a general description of the surface texture. Furthermore, *Ra* values provide a simple, single-value quantification of surface roughness, which is helpful for general comparisons between different materials or surfaces. Clinically acceptable Ra values for dental materials have been reported to be around 0.2 µm (Bollen et al., 1997b; Çakmak, Donmez, et al., 2022a). Higher values may lead to increased plaque retention and a higher caries incidence and periodontal inflammation (Bollen et al., 1997b).

Achieving smooth surfaces necessitates polishing or glazing (Kara et al., 2021). High-quality polishing methods can achieve surface roughness levels that meet the clinically acceptable threshold, thereby enhancing the longevity and performance of dental restorations (Brodine et al., 2021; Çakmak et al., 2023; Kara et al., 2021). *Ra* values for various ceramic-filled hybrid resin materials reported 0.2-3.0 µm following similar polishing techniques (Arafa & Ghanem, 2023; Matzinger et al., 2019). It is recommended that the surface of VSplus be polished using a pumice stone and polishing compound, ensuring that the resin is not heated during the process for optimal results (BEGO, 2019). The manufacturer also stated that VSplus could undergo high-gloss polishing with composite polishers commonly used in dental practice (BEGO, 2019).

Lithium disilicate IPS e.max is widely recognized for its superior mechanical properties and aesthetic qualities (Yuan et al., 2018; Zarone et al., 2016). The surface roughness of lithium disilicate crowns can vary based on the fabrication technique used, milled lithium disilicate crowns have been reported to have Ra values below 0.2 µm (Yuan et al., 2018). These values indicate a highly smooth surface, which is beneficial for minimizing plaque retention and enhancing the longevity of the restoration. According to the manufacturer, The IPS e.max CAD polishing techniques include self-glazing/polishing and glazing variants. The self-glazing/polishing technique is more straightforward and faster, involving manual polishing of the pre-crystallized restoration followed by crystallization, but offers limited aesthetic customization. The glazing variants, which include Speed Crystallization with glazing spray (Variant A) and Crystallization with staining/glazing paste (Variant B), provide greater aesthetic customization and higher gloss but are more complex and time-consuming. Each technique involves different steps and materials, with self-glazing/polishing being more straightforward and glazing variants requiring additional materials and steps for enhanced aesthetics (Vivadent, 2017). The choice depends on the desired aesthetic outcome and available resources. The polishing of IPS e.max CAD can also be done after the crystallization to achieve a better-polished surface or after any adjustment was made. They recommend in the manual that pre-polishing is performed using a diamond rubber polisher (e.g., OptraFine® F), followed by fine polishing with a high-gloss rubber polisher (e.g., OptraFine® P) until polished to high gloss, ensuring not to overheat the ceramic (Vivadent, 2016, 2017).

2.2.3 SEM in Surface Roughness Evaluation

The SEM is a powerful tool used to analyse the surface morphology of dental materials at high magnifications, providing detailed images that reveal surface textures and irregularities that might not be visible with other techniques. SEM scans a focused beam of electrons over a sample's surface and detects the emitted secondary or backscattered electrons to form an image. This technique is invaluable for evaluating surface roughness, as it allows for visualisation at different magnifications, typically ranging from 20x to 30,000x or more. It complements traditional profilometry techniques and provides a more comprehensive understanding of surface characteristics (J. Goldstein & C, 2003; Mohammad Ali Saghiri, 2012; Zhou & Wang, 2007).

In previous studies, SEM has been used to observe the surface roughness of dental materials at various magnifications. For example, several studies used SEM at magnifications of 1000x to 6000x to analyse the surface characteristics of dental ceramics, providing detailed images that highlighted surface irregularities and texture (Özarslan et al., 2022; Vasiliu et al., 2020). In addition, Munusamy et al. (2020) also used SEM at magnifications of 5000x to analyse the detailed surface topography and the microstructural changes of CAD/CAM composite after being conditioned in different types of dietary solvent (Munusamy, 2020). Meanwhile, Tekçe et al. (2018) utilised SEM at 2000x magnification to assess the effects of different polishing protocols on composite resins, enabling a comparative analysis of surface smoothness (Tekçe et al., 2018). Similarly, Arafa and Ghanem (2023) employed SEM at 1000x and 3000x to examine the microstructure and changes of the filler distribution of hybrid resins after thermal cycling and chewing simulation, revealing fine details of surface wear and roughness (Arafa & Ghanem, 2023).

High magnifications such as 5000x provide an in-depth view of the microstructure and nanostructure of the material surface, revealing fine details such as nanometre-scale roughness and the presence of micro-cracks or voids. This level of magnification is essential for analysing intricate surface topographies and finer features that are not visible at lower magnifications. This is important for understanding the mechanical and physical properties of materials, and ultimately, this level of analysis is crucial for advanced research into the durability and performance of dental materials (J. Goldstein & C, 2003; Mota et al., 2017; Munusamy, 2020; Stokes, 2008; Vasiliu et al., 2020; Zhou & Wang, 2007). Hence, for this study, 5000x magnification was used for a better understanding of the microstructural changes of VSplus and Emax group after artificial ageing.

2.2.4 Surface Roughness on Clinical Implication

The surface properties of restorative materials used in the oral cavity should prevent the production of biofilms and bacterial adherence. Understanding the relationship between surface roughness and biofilm formation in different materials is crucial when planning for the long-term success of dental restorations (Bremer et al., 2011; Matalon et al., 2021). Biofilms are structured communities of bacteria that adhere to surfaces, and the surface texture of dental materials influences their formation. Rough surfaces facilitate the initial attachment of bacteria, which can develop mature biofilms resistant to mechanical and chemical cleaning methods (Bremer et al., 2011). Therefore, materials and techniques that minimise surface roughness are essential for preventing biofilm-related complications (Bremer et al., 2011; Vo et al., 2015).

Quirynen and Bollen (1995) established that minor variations in surface roughness could significantly influence bacterial colonisation even in the same material. Rough surfaces provide niches for bacteria to colonise. For example, subgingival roughness, in particular, promotes the retention of microorganisms, which can lead to periodontitis and implant failure (Bollen et al., 1997b; Brecx et al., 1983; Vo et al., 2015). Studies have shown that materials with smoother surfaces are less prone to plaque accumulation, reducing the risk of periodontal inflammation and infection. In addition, Bollen et al. (1997) found a significant positive correlation between surface roughness above 0.2 µm and plaque retention, emphasising the importance of maintaining smooth surfaces to minimise the risk of oral diseases (Bollen et al., 1997b).

Customisation of restorative materials is a key strategy to minimise surface roughness and its associated risks. By tailoring the composition and properties of the materials, it is possible to achieve smoother surfaces that resist bacterial adherence and plaque accumulation (Özarslan et al., 2022; Vo et al., 2015). This approach can be particularly beneficial in subgingival areas where roughness increases the retention of microorganisms and the risk of periodontal disease. Surface roughness of restorations is significant for patients with suboptimal oral hygiene, with a higher plaque accumulation tendency (Matalon et al., 2021). This will promote periodontal inflammation and infection and eventually cause pulp sensitivity, gingival recession, and tissue inflammation. Furthermore, inflamed tissue will further complicate the restorative rehabilitation process.

Recent research trends in surface roughness focus on developing materials and techniques that minimise roughness and enhance the longevity of dental restorations. Digital technologies such as CAD/CAM systems allow for precise control over the surface texture of restorations, further reducing the risk of roughness-related complications (Joda et al., 2017). Advanced materials like lithium disilicate, such IPS e.max CAD and ceramic-filled hybrid resins, such as VSplus, demonstrate excellent surface smoothness, making them suitable for long-term dental applications (Brodine et al., 2021; Çakmak et al., 2023). Future research should focus on developing materials that maintain smoothness over time and under varying oral conditions.

2.3 Artificial Ageing: Thermocycling and its Significance

The long-term success of dental restorations is limited by their durability in the oral environment. Longevity and efficiency are the ideal characteristics of dental restorations (Mjor et al., 1990). Therefore, restorative materials must withstand a harsh environment, which varies from patient to patient. Factors that include mastication forces, occlusal habits, dietary factors, humidity, and temperature fluctuations may affect material longevity (Rezaie et al., 2020; Sakaguchi & Powers, 2011). In-vitro simulations can help predict the longevity of dental materials by assessing their mechanical and structural decay characteristics during clinical ageing. Although laboratory evaluation and in vitro studies cannot precisely simulate conditions in the oral cavity, such as the clinical environment, to some extent, they can simulate the oral cavity environment through an artificial ageing procedure for teeth and restorations (Morresi et al., 2014; Szczesio-Wlodarczyk et al., 2022).

Artificial ageing is a regulated procedure intended to simulate the effects of ageing naturally on dental materials over a shorter period. This procedure typically involves exposure to environmental factors such as temperature variations, humidity, and chemical agents. Artificial ageing methods, such as thermocycling, are crucial in dental research. These methods help researchers evaluate how materials behave under conditions that mimic the dynamic oral environment, thus providing insights into their durability and performance over time. For instance, thermocycling involves subjecting dental materials to repeated cycles of hot and cold temperatures to simulate the thermal stresses experienced in the mouth (Morresi et al., 2014)

2.3.1 Different Types of Artificial Ageing Methods

(a) Thermocycling

Thermocycling is the laboratory method that simulates the ageing of dental restorative materials by subjecting them to the combined effects of repeated temperature fluctuations and moisture changes. The process involves cycling between hot and cold temperatures for specific durations, with a set number of cycles to mimic the thermal fluctuations experienced in the oral environment. This method facilitates the assessment of a material's hydrolytic stability and its capacity to withstand degradation under extreme temperature conditions. This makes thermocycling a comprehensive and effective method for predicting dental materials' clinical performance and longevity. (Morresi et al., 2014; Szczesio-Wlodarczyk et al., 2022)

Thermocycling is often employed in conjunction with other ageing systems, such as mechanical loading and toothbrushing, to replicate the conditions that restorative materials face in the oral environment but the choice of intervention often depends on the specific objectives of the study and the materials being tested (Morresi et al., 2014). Yuan et al. conducted a study to examine the effects of different intervention groups, including brushing alone, thermocycling alone, and combination brushing and thermocycling. They discovered that whereas brushing alone with zirconia resulted in a smoother surface, thermocycling alone and brushing combined with thermocycling increased the surface roughness value. This highlights that thermocycling alone has a distinct impact on surface roughness (Yuan et al., 2018).

(b) ***pH Cycling:***

pH cycling is a laboratory method that simulates the fluctuating *pH* levels in the oral environment by exposing dental restorative materials to alternating acidic and neutral conditions. This process typically involves immersing the samples in acidic solutions (around *pH* 4.0 to 5.0) for a set duration, followed by immersion in neutral solutions (around *pH* 7.0) to mimic daily dietary effects. After pH cycling, the materials are evaluated for properties such as microleakage and bond strength to assess their durability and performance under conditions similar to those in the mouth. (Francisconi et al., 2008; Honório et al., 2008)

(c) ***Mechanical Loading:***

Mechanical loading as artificial ageing refers to applying controlled forces or stresses to dental materials to simulate the effects of the masticatory forces. This process is designed to accelerate the degradation and wear of dental materials, allowing researchers to evaluate how these materials will perform under realistic conditions in the oral environment. By subjecting dental materials to mechanical loading in a laboratory setting, researchers can assess changes in properties such as strength, hardness, and structural integrity, providing insights into the materials' durability and potential lifespan in clinical applications. It is often used in conjunction with thermocycling to assess the mechanical durability of materials (Elshiyab et al., 2018; Schestatsky et al., 2020).

2.3.2 Thermocycling Protocols

Thermocycling is one of the most widely used procedures in laboratory research to reproduce dynamic stresses. That is also widely accepted in international literature and has been commonly employed in dental research since 1952 (Morresi et al., 2014). Many experimental studies have been published that use thermocycling regimes to test dental materials characteristics (Bedran-de-Castro et al., 2004; D'Amario et al., 2010; Doerr et al., 1996; Schuckar & Geurtsen, 1997; Wegner et al., 2002), following the publication of Gale and Darvell's review over ten years ago (Gale & Darvell, 1999). This system is conventionally used to simulate the in vivo ageing of restorative materials by subjecting them to repeated cyclic exposures to hot and cold temperatures in water baths, aiming to reproduce thermal changes occurring in the oral cavity (Catalbas et al., 2010; El-Araby & Talic, 2007; Özel Bektas et al., 2012).

Natural teeth and dental restoratives are subjected to constant and extreme changes in the oral environment brought about by fluctuations in temperature and pH (Morresi et al., 2014; Wahab et al., 2003). Thermocycling, through temperature parameter, simulates the entrance of hot and cold substances in the oral cavity and shows the relationship of linear coefficient of thermal expansion between tooth and restorative material (Cenci et al., 2008)

Although the International Organization for Standardization (ISO) considered a protocol of 500 cycles as appropriate in simulating the ageing of biomaterials, many studies suggest that 500 cycles is a limited number to represent an adequate ageing time (Amaral et al., 2007; Gale & Darvell, 1999; Stewardson et al., 2010). Five hundred cycles would only correspond to cycles estimated to occur in less than 2 months in the mouth (Stewardson et al., 2010). Instead, Gale & Darvell, 1999 postulated that 10,000 thermal cycles correspond to one year of clinical function (Gale & Darvell, 1999). This estimation is based on the hypothesis that such cycles might occur 20 to 50 times daily. Many authors have accepted this concept, and they have followed the principle (Amaral et al., 2007; De Munck et al., 2005; Hatanaka et al., 2006; Özel Bektas et al., 2012; Saboia et al., 2009; Stewardson et al., 2010; Ulker et al., 2010; Xie et al., 2010). A total number of 5000 cycles was chosen for this study to balance the need for rigorous testing with practical considerations and limitations such as time, cost, and resource availability. It is a manageable number that can still yield significant data on the durability and performance of materials. This number of cycles is also similar to number of cycles to previous studies (Catalbas et al., 2010; Özel Bektas et al., 2012; Xie et al., 2010).

The choice of temperatures and the number of cycles can vary depending on the specific goals of the study. However, the ISO standard proposed in the ISO 11405 recommendations reported temperatures of 5 C and 55 C to test dental materials, considering these values as the closest to the physiological thermal fluctuations of the oral cavity (ISO, 1994). Many authors have followed the same choice of temperature fluctuations (De Munck et al., 2005; Hatanaka et al., 2006; Özel Bektas et al., 2012; Saboia et al., 2009; Stewardson et al., 2010; Xie et al., 2010).

Dwell time is the time that the sample is immersed in a bath of a particular temperature (Schmid-Schwap et al., 2011). It corresponds to a latency period, during which the oral capacity is required to reach its normal temperature again after consuming hot or cold food and drink. Unfortunately, the choice of dwell times in experimental studies appears arbitrary, showing significant variability in the different studies, and no effect of dwell time on results has been established (Helvatjoglu-Antoniades et al., 2004a; Kenshima et al., 2004; De Munck et al., 2005; Cavalcanti et al., 2007). However, the recommended dwell time suggested by ISO 11405, 1994, in the last thirty years is ≥20 seconds for each temperature environment, and the transfer time between baths is 5-10 seconds (ISO, 1994).

The effects of thermocycling on dental materials are typically evaluated using a combination of surface roughness measurements, mechanical testing, and microscopic analysis. Surface roughness can be assessed using profilometers to measure parameters such as *Ra* value, providing quantitative data on the changes in surface texture (Yuan et al., 2018). Mechanical properties such as flexural strength and fracture toughness can be tested to determine any changes in the material's structural integrity (Yuan et al., 2018). Microscopic techniques, particularly Scanning Electron Microscopy (SEM), play a crucial role in evaluating the microstructural changes induced by thermocycling. SEM allows for high-resolution imaging of the material's surface, revealing microcracks, phase transformations, and surface wear (Goldstein et al., 2003; Zhou et al., 2007). This detailed analysis helps researchers understand the mechanisms behind the material's response to thermal stresses and guides the development of more resilient restorative materials (Goldstein et al., 2003; Zhou et al., 2007).

2.3.3 Clinical Relevance of Thermocycling

The cyclic expansion and contraction induce stress on the material, potentially leading to microcracks, phase transformations, and changes in surface characteristics. Dental materials must withstand the stresses and strains imposed by temperature changes without exhibiting significant changes in properties, such as surface roughness, colour stability, mechanical strength, or marginal integrity. This information is crucial for developing and improving materials used in dental restorations, ensuring they meet the necessary standards for longevity and performance in the dynamic oral environment. (Morresi et al., 2014) (Cenci et al., 2008).

Understanding the effects of thermocycling on dental materials has significant implications for clinical practice. Materials that maintain their properties under thermal stress will likely perform better in the oral environment, offering longer-lasting restorations with fewer complications (Temizci & Bozoğulları, 2024). For instance, the resilience of CAD/CAM milled lithium disilicate and 3D printed VSplus under thermocycling conditions suggests that these materials can withstand the daily thermal fluctuations in the mouth without significant degradation (Yuan et al., 2018; Arafa & Ghanem, 2023). Moreover, the ability to predict the long-term performance of dental materials through artificial ageing and thermocycling studies helps dentists make informed decisions about material selection. This knowledge is crucial for providing patients with restorations that meet their aesthetic and functional needs and ensure durability and minimal maintenance over time (Szczesio-Wlodarczyk et al., 2022).

Artificial ageing and thermocycling are essential dental research procedures for evaluating restorative materials' long-term performance. These methods simulate the dynamic conditions of the oral environment, providing valuable insights into how materials like CAD/CAM milled lithium disilicate and 3D printed VSplus behave under thermal stress. Using standardized protocols, such as those proposed by ISO 11405, ensures the reliability and relevance of these studies, ultimately guiding the development and selection of durable, high-performance dental restorations.

2.4 Studies on VarseoSmile Crown plus (VSplus)

VSplus is the world's first hybrid material for 3D printing of permanent single crowns, inlays, onlays, and veneers. It is a newly developed tooth-coloured, ceramic-filled hybrid light-cured resin material based on methacrylic acid esters (BEGO, 2020). The manufacturer claims easy handling, no mixing or shaking necessary with regular use, and easy finishing and polishing using standard tools (BEGO, 2020).

The mechanical characteristics of VSplus achieve breaking loads two times higher than the maximum average human masticatory forces of 720 N (Gibbs et al., 2002) and almost four times as high as the maximum average masticatory force of approximately 490 N (Fischer et al., 2003), both initially and after a 10-year chewing simulation. Furthermore, another study demonstrated no substantial reduction in breaking load after artificial ageing (BEGO, 2020; Eva Jerman, 2020). This result implies that the restorations are thus preserved for an extended period, and there is a low risk that a crown could fracture in the patient's mouth. Regarding the abrasion resistance, VSplus demonstrated less material loss (higher resistance to wear) after the chewing simulation than the material Sinfony (Albrecht, 2020; BEGO, 2020).

3D printed dental materials drawbacks include toxic resin waste production, exhibit shrinkage during polymerization, and surface roughness from the layering addition of resin (Ellakany et al., 2022). However, in the solubility study of VSplus 3D printed restorations, VSplus yielded no substances in the detectable area. Therefore, conceivable risk to patients, e.g., through allergies, can be classified as exceedingly low (BEGO, 2020). In addition, crowns made of VSplus do not tend to decementation, i.e., a loss of composite material or marginal gap formation, when using commercially available, dual-curing luting composites (BEGO, 2020).

Limited studies have focused on the response of surface roughness of VSplus to artificial ageing. Preliminary research suggests that this material exhibits favourable mechanical properties. However, a comprehensive analysis of its surface roughness behaviour under artificial ageing conditions is yet to be thoroughly explored. Understanding how VSplus withstands simulated ageing is essential for providing insights into its long-term clinical performance. Even though previous studies have investigated additively manufactured composite resins indicated for definitive prostheses, the knowledge of their *Ra* value is limited (Çakmak, Donmez, et al., 2022a). There is a lack of available data and different variations in the results of the studies on the surface roughness of newly introduced 3D printed. However, *Ra* values obtained from previous papers using identical polishing techniques for various other kinds of ceramic-filled hybrid resin materials were found to be *Ra* 0.2-3.0 µm (Arafa & Ghanem, 2023).

2.5 Studies on Lithium Disilicate

Nowadays, glass ceramics have improved mechanical properties like increased fracture resistance, resistance to erosion, and thermal shock resistance. Increased strength in glassy ceramics is achieved by adding appropriate fillers that are uniformly dispersed throughout the glass, such as aluminium, magnesium, zirconia, leucite, and lithium disilicate (Flury et al., 2010; Zarone et al., 2016). The ceramics reinforced by lithium disilicate are true glass ceramics, with the crystal content increased to approximately 70% and the crystal size refined to improve flexural strength (Sakaguchi & Powers, 2011). Filler particles are added to the base glass composition to enhance the mechanical properties and optical effects such as opalescence, colour, and opacity (Amaya-Pajares et al., 2016). One of the problems with filler particle reinforced crowns has been sintering shrinkage and colour control, which have only been overcome in the last 30 years through the advent of CAD/CAM dentistry. (Fu et al., 2020)

CAD/CAM technology has revolutionized the fabrication of dental restorations, offering high precision and consistency. Lithium disilicate, particularly in the form of IPS e.max CAD, is widely recognized and renowned for its superior mechanical properties and aesthetic qualities (Dolev et al., 2019; Sailer et al., 2017). The milled lithium disilicate crowns (IPS e.max CAD) have been reported to have *Ra* values below 0.2 µm (Yuan et al., 2018). Meanwhile, another study reported the *Ra* value ranging from 0.2 to 0.3 µm (Brodine et al., 2021). In addition, a previous study found that ceramic polishing systems created smoother and more uniform surfaces in terms of quantity and quality of roughness compared to glazing and reglazing (Mohammadibassir et al., 2019). These values indicate a highly smooth surface, which is beneficial for minimizing plaque retention and enhancing the longevity of the restoration.

Studies have shown that CAD/CAM milled lithium disilicate maintains its surface roughness within clinically acceptable limits even after extensive thermocycling, indicating its durability under thermal stress (Yuan et al., 2018). These demonstrate the material's ability to maintain a smooth surface with minimal surface degradation under clinical conditions. This finding underscores the material's ability to withstand the thermal fluctuations in the oral cavity without compromising its surface integrity, which is crucial for maintaining the restoration's aesthetic appearance and clinical performance over time (Yuan et al., 2018).

2.6 Research Gap and Rationale

The existing literature lacks comprehensive studies on the long-term performance of VSplus, particularly regarding its durability under artificial ageing conditions. While numerous studies have examined the effects of artificial ageing on dental materials, a thorough evaluation of the artificial ageing of VSplus compared to lithium disilicate is needed. Several previous studies have primarily focused on 3D printed composite resin-based materials used for provisional crown (Ellakany et al., 2022; Tahayeri et al., 2018).

VSplus represents a hybrid material designed for 3D printing permanent dental prosthetics. Currently, there is limited information related to its surface roughness following prolonged usage. These factors are integral to the overall performance and longevity of dental restorations. In contrast, lithium disilicate has been widely studied and is known for its excellent mechanical properties, aesthetic qualities, and durability (Zarone et al., 2016). There is a gap in the literature regarding the performance of this novel hybrid ceramic compared to the well-established glass ceramic. Thus, further investigation is needed to understand how artificial ageing impacts the surface roughness of VSplus and how it compares to lithium disilicate.

This research aims to compare the surface roughness of VSplus with lithium disilicate under artificial ageing conditions.

CHAPTER 3: MATERIALS AND METHOD

3.1 Materials

This in-vitro study assessed and compared the surface roughness of the 3D printed crown material VSplus and milled lithium disilicate before and after inducing artificial ageing. Table 3.1 lists the details of the composition of test materials and the filler content utilized in this study.

Table 3.1 Materials used in this study

Trade Name	Manufacturer	Composition
VarseoSmile Crown plus (VSplus group) CAD/CAM Additively manufactured composite resin	Bego, Bremen, Germany	Esterification products of 4,4′-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanised dental glass, methyl benzoylformate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide, 30–50 wt%—inorganic fillers (particle size 0.7 µm)
IPS e.max CAD (Emax group) CAD/CAM Milling lithium disilicate glass ceramic	Ivoclar–Vivadent, Schaan, Liechtenstein	SiO2, Li2O, K2O, P2O5, ZrO2, ZnO, other and colouring oxides

3.2 Sample Size Calculation

The sample size was estimated using G*Power 3.1.9.4 statistical software. Since there is a lack of studies comparing VSplus and Emax, the baseline mean of average roughness (*Ra)* and standard deviation (SD) for VSplus and Emax were obtained from previous studies to estimate the expected effect size. For VSplus group, the mean *Ra* and SD were 0.2596 ± 0.0045 (Arafa & Ghanem, 2023), and for Emax group, they were 0.47 ± 0.15 (Mohammadibassir et al., 2019). The sample size calculation was conducted with the level of significance, $\alpha = 0.05$, and power of study $(1 - \beta) = 98\%$. The minimally accepted sample size was 8 samples for each group when the response was normally distributed. The sample size was adjusted to 10 per group to compensate for the 15% dropout to ensure a sufficient number of samples, resulting in a total of 20 samples. A brief outline of the methodology is illustrated in Figure 3.1.

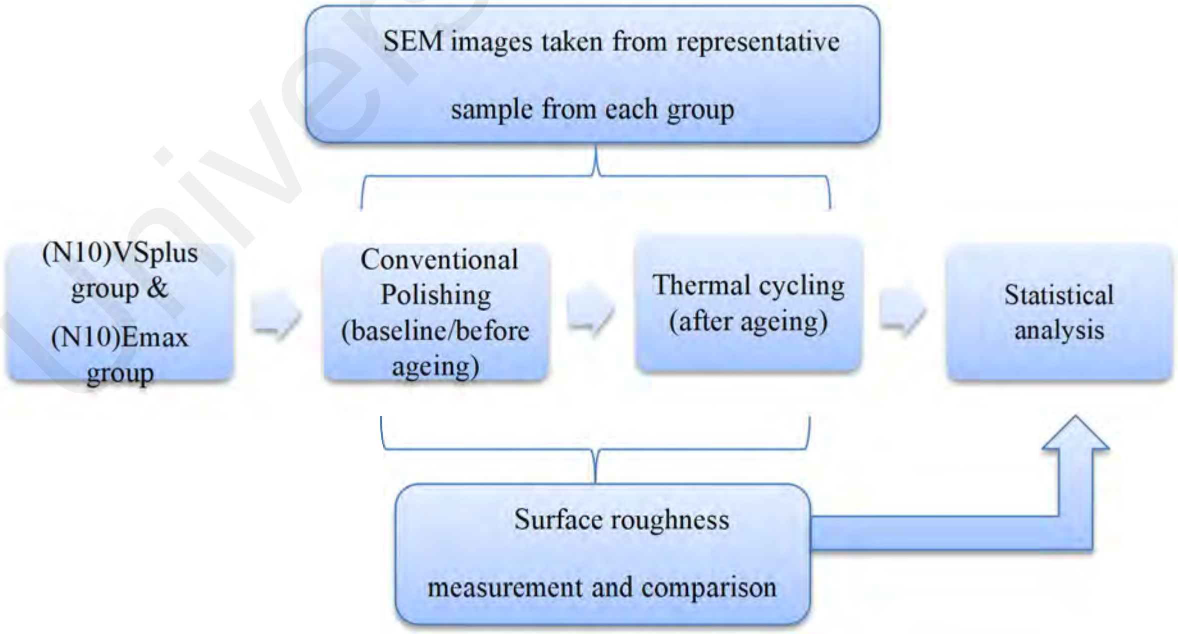

Figure 3.1: Diagram of The Methodology

3.3 CAD/CAM 3D printed VarseoSmile Crown plus Samples Fabrication

A standard tessellation language (STL) format file of rectangular-shaped samples with desired dimensions (14×12×3 mm) was designed using a software. The finished STL file was printed using LCD (Liquid Crystal Display) technology utilising a 3D printer (Ackuretta SOL 3D printer, Taiwan) (Figure 3.2). The composite resin samples were printed vertically from a liquid material of VSplus on the build platform. After the supports were automatically generated, the support arrangement was repeated 10 times for standardization. The layer thickness was set to 50 μm, and the samples were printed using the digital 3D printer.

On completion of printing, all the 3D printed samples were separated from the build platform using the spatula. Then, they were cleaned following the manufacturing recommendation in two steps with ethanol (96 %) using an ultrasonic bath (Ackuretta CLEANI Washing Unit, Taiwan) (Figure 3.3). First, for 3 min in a reusable ethanol solution (96 %), the samples were cleaned carefully for another 2 min in a freshly used (96 %) solution. Finally, the 3D printed samples were removed from the ethanol bath and sprayed with additional ethanol (96 %) to remove any remaining resin residue. After cleaning, the 3D printed samples were dried using compressed air under an extraction unit.

The printed samples were post-cured two times for 20 minutes and then left to cool for 3-5 min in an ultraviolet light curing device (Ackuretta CURIEplus Curing Unit, Taiwan) (Figure 3.4), which provides a consistent cure due to its uniform 360° light distribution. It is suitable for post-curing 3D printed composite resin materials to ensure complete polymer conversion, reduce residual monomer, and obtain the highest mechanical properties. Then, all the supporting structures (Figure 3.5) of the final printed end product were cut with a cutting wheel.

Figure 3.2: Ackuretta SOL 3D Printer

Figure 3.3: Ackuretta CLEANI Washing Unit

Figure 3.4: Ackuretta CURIEplus Curing Unit

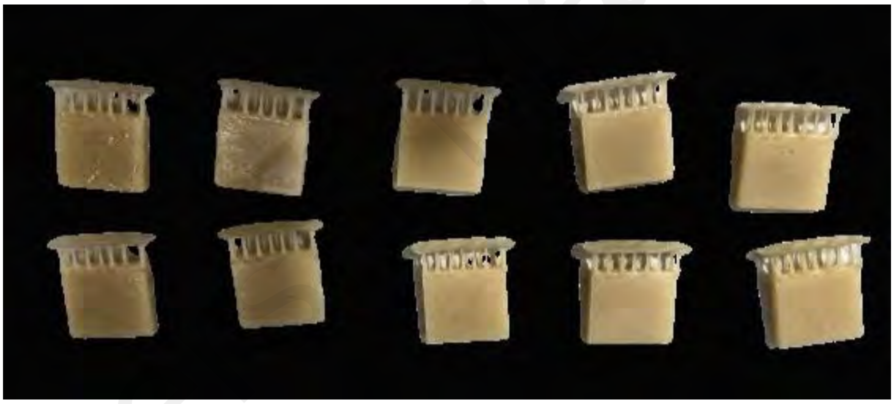

Figure 3.5: 3D printed VSplus samples with the supporting structure

3.4 CAD/CAM Milling Lithium Disilicate Samples Fabrication

The STL file was used to design the milling crowns using CAD software (exocad DentalCAD). The sample group of lithium disilicate (IPS e.max CAD for Cerec and Inlab; Ivoclar–Vivadent, Schaan, Liechtenstein) were milled by using a 4-axis dental milling machine (Aidite AMW 400 Wet Milling Machine, China) (Figure 3.6) to form the rectangular-shaped samples with similar size of VSplus samples with desired dimensions (14×12×3 mm). After milling, the lithium disilicate samples were cleaned to remove any residues or debris. The samples were being inspected for any defects or inaccuracies that may have occurred during the milling process. Then, the lithium disilicate samples crystalised in a dental furnace (Ivoclar Vivadent Programat P310, Liechtenstein) (Figure 3.7) to achieve its final mechanical and optical properties following the manufacturer's recommendation crystallisation parameters.

Figure 3.6: Aidite AMW 400 Wet Milling Machine

Figure 3.7: Ivoclar Vivadent Programat P310

Figure 3.8: Emax samples (Post-milling)

3.5 Finishing and Polishing of the Samples

Samples of VSplus and Emax groups were finished with silicon carbide abrasive papers with grit P600 to P1200 at 250rpm for 30 seconds under constant water irrigation using Beta 2 grinder-polisher machine (Buehler, Germany) (Figure 3.9), the finishing procedure were standardized for both groups' samples. The samples were polished on one side of each sample with a polishing technique that is recommended by the manufacturer for clinical use. For the VSplus group, pumice stone and polishing compound are used, with a critical emphasis on preventing resin overheating during polishing. (BEGO, 2019). For the Emax group, the polishing was performed using a rubber polisher and polishing paste until polished to high gloss, ensuring not to overheat the ceramic (Vivadent, 2016, 2017).

The final thickness for all samples of the tested groups were checked using a digital calliper (Mitutoyo Corporation, Kawasaki, Japan) to ensure a thickness of 3 mm ±0.15 mm (Figure 3.10). Samples were then cleaned in an ultrasonic bath in distilled water for 5 minutes. All samples were stored in a dry, closed container until further testing.

Figure 3.9: Beta 2 grinder-polisher machine (Buehler, Germany)

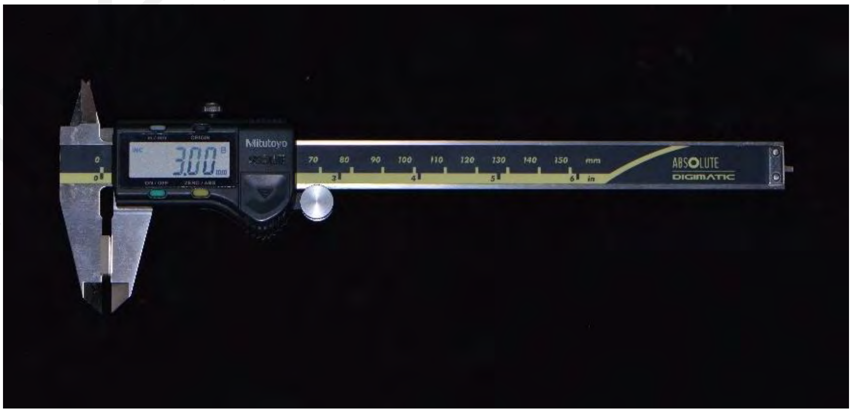

Figure 3.10: Digital caliper Mitutoyo Corporation, Kawasaki, Japan

3.6 Measurements of the Surface Roughness (*Ra* value)

Ra values of before and after ageing were measured using the Infinite Focus Optical 3D Measurement G4e machine (Alicona Imaging GmbH, Raaba, Austria). The magnification of the three-dimensional (3D) surface analyser was set at 20x with a vertical of 325.66 nm and lateral resolutions of 2.93µm. The reading was taken at three uniformly distributed areas of each sample. The *Ra* values were determined using IFM version 3.5 software (Alicona Imaging GmbH, Raaba, Austria) (Figure 3.11). The measurements were tabulated, and mean values for each material/ medium combination were computed.

Figure 3.11: Alicona Imaging GmbH, Raaba, Austria

3.7 Scanning Electron Microscope (SEM)

A representative sample from each group was randomly selected and examined with Field Emission Scanning Electron Microscopy (FE-SEM) (Quanta FEG 450, Thermo Fisher Scientific, Brno, Czech Republic) (Figure 3.12) before and after the ageing process. The magnifications were set at 5,000x at the centre of the representative samples to provide an in-depth view of the material surface's microstructure, revealing fine details in nanometre-scale roughness and any presence of micro-cracks or voids. This level of magnification provides a comprehensive assessment of the surface morphology and microstructural changes that are essential for understanding material properties before and after artificial ageing.

Figure 3.12: FE-SEM Quanta FEG 450, Thermo Fisher Scientific

3.8 Thermocycling (Artificial Ageing)

Samples were subjected to artificial ageing using the thermocycling machine designed by Zecttron Sdn Bhd, Faculty of Engineering, Universiti Malaya (Figure 3.13). The extreme temperature bath environments are between 5 and 55°C. The dwell time in the thermal bath used in this study is 20 seconds for each temperature environment, and the transfer time between baths is 5-10 seconds (ISO, 1994). The total number of cycles chosen in the study is 5000 cycles, which corresponds to approximately 6 months in the oral cavity (Gale & Darvell, 1999). The thermocycling test starts with 24 hours of storage in water at 37 C (Figure 3.14). Then, 500 cycles ran continuously for about 9 H per day, and the samples were stored in the water bath at 37 C while waiting for the next cycle to start. Approximately 10 days were needed for the 5,000 cycles.

Figure 3.13: Thermocycling machine (Zecttron Sdn Bhd, Faculty of Engineering, Universiti Malaya)

Figure 3.14: Incubator set at 37^{0}C

3.9 Statistical Analysis

The normality of the data distribution was evaluated using the Shapiro-Wilk test. The data was presented as mean and standard deviation for *Ra* values. While assessing the *Ra* values of different material groups before and after ageing, the Paired t-test was used. Meanwhile, Independent sample t-test was used to compare the means of the *Ra* value between the two groups before and after ageing. All analyses were performed with software (IBM SPSS 26 ® Statistical Package for Scientific Studies, IBM Corp), and significance was set at $p \leq 0.05$.

CHAPTER 4: RESULTS

4.1 Surface Roughness

Table 4.1 presents the mean values (Ra) of surface roughness and descriptive statistics of the VSplus and Emax groups before and after ageing. The VSplus sample showed lesser surface roughness (0.145µm) compared to Emax before the ageing process. The highest surface roughness was in Emax samples following the ageing process (0.232 µm). The surface roughness values of both groups increased following ageing. The output data can be found in Appendix A.

Table 4.1: Descriptive Statistics of Surface Roughness (*Ra* value)

	Before Ageing				After ageing			
	Mean (µm)	Std. Dev	Min (µm)	Max (µm)	Mean (µm)	Std. Dev	Min (µm)	Max (µm)
VSplus	0.145	0.040	0.094	0.228	0.168	0.036	0.121	0.231
Emax	0.223	0.051	0.148	0.301	0.232	0.049	0.155	0.306

Figure 4.1 shows that the surface roughness means of the VSplus are lower than those of the Emax group before and after artificial ageing, and the surface roughness of each group showed an increase in roughness following the artificial ageing process. The differences between mean values of surface roughness are shown in Figure 4.2.

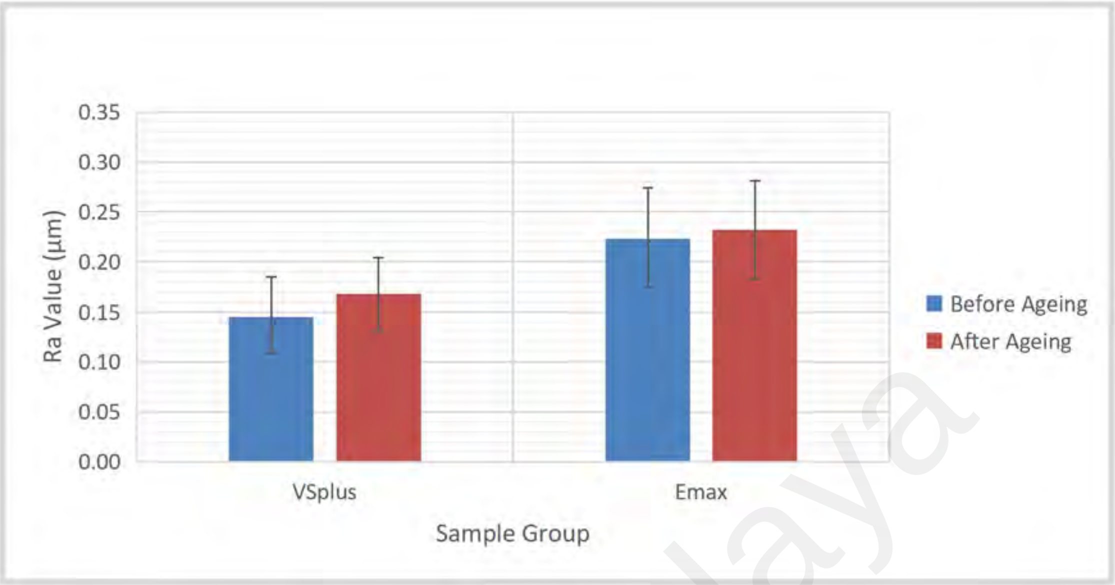

Figure 4.1: Mean surface roughness (*Ra*) for VSplus and Emax group before and after artificial ageing

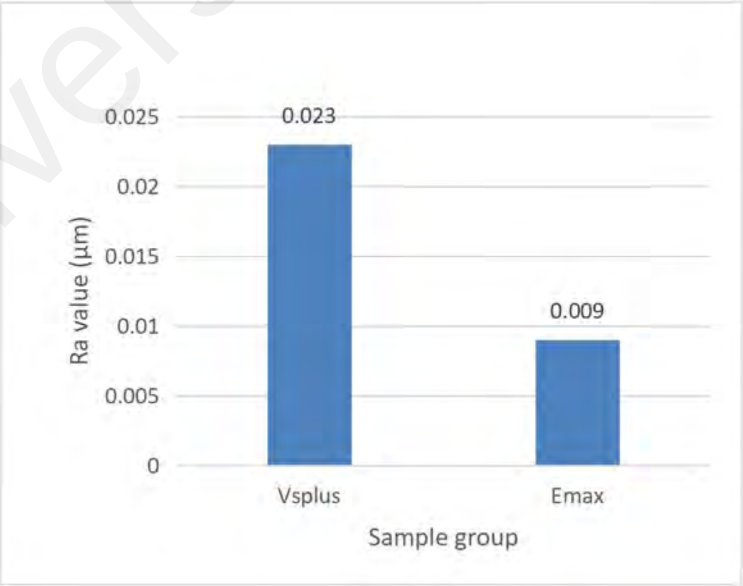

Figure 4.2: *Ra* means difference before and after artificial ageing for VSplus and Emax group

The Shapiro-Wilk test was utilized to assess the normality of the data. The data showed a normal distribution. A paired sample t-test was conducted to compare the mean surface roughness (*Ra* value) for both groups before and after artificial ageing. Meanwhile, an independent sample t-test was applied to compare the means in surface roughness between the two groups before and after artificial ageing (Table 4.2).

The Independent *t-test* identified significant differences in *Ra* values between VSplus and Emax groups under both conditions, before and after artificial ageing with *p* value of less than 0.005. After artificial ageing, all sample groups demonstrated an increase in surface roughness. For both groups, the paired *t-test* indicates a significant increase in *Ra* value from before to after artificial ageing with *p* value of 0.006 (*p* value< 0.05) for VSplus group and *p* value of 0.018 (*p* value<0.05) for Emax group.

Meanwhile, the difference in *Ra* values before and after artificial ageing is 0.023µm for the VSplus group and 0.09µm for the Emax group.

Table 4.2: Inter and Intra group comparison for the mean *Ra* values

Sample Group	Mean ± Standard Deviation (µm)		*p* value
	Before Ageing	After Ageing	
VSplus	0.145 ± 0.040	0.168 ± 0.036	0.006
Emax	0.223 ± 0.051	0.232 ± 0.049	0.018
p value	0.001	0.004	

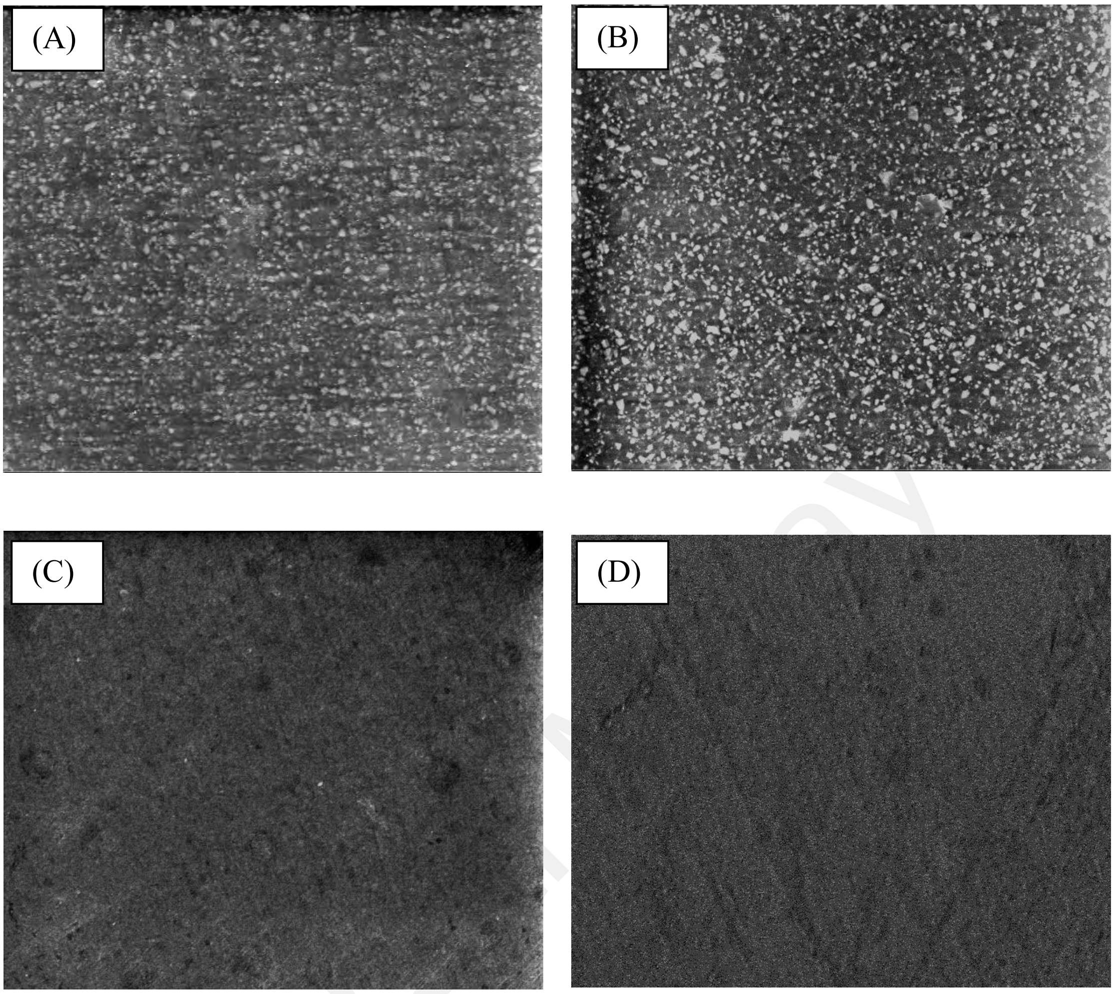

Figure 4.3: SEM surface images at 5000x magnification: (A)VSplus before artificial ageing; (B)VSplus after artificial ageing; (C)Emax before artificial ageing; (D)Emax after artificial ageing

At 5000x magnification, the SEM images provide detailed insights into the microstructures of VSplus and Emax, with each material presenting a different surface topography (Figure 4.3). For VSplus, the SEM images before artificial ageing reveal a relatively uniform filler particle within the resin matrix (Figure 4.3 (A)). After artificial ageing, the filler particles in VSplus become more irregular (Figure 4.3 (B)). In contrast, the SEM images of Emax show some lines and striation, and several pores (Figure 4.3 (C)). After artificial ageing, the surface of Emax shows more lines (Figure 4.3 (D))

CHAPTER 5: DISCUSSION

5.1 Surface Roughness Assessment

In this in-vitro study, assessment of the surface roughness *Ra* of 3D-printed ceramic-filled hybrid material (VSplus group) and milled lithium disilicate (Emax group) was performed before and after inducing artificial ageing. There was a significant difference in the surface roughness between VSplus and Emax group before and after artificial ageing ($p < 0.005$); hence the null hypothesis of the study is rejected

The mean *R*a value of the VSplus group is 0.145µm before ageing and 0.168µm after ageing; the values are within the documented clinically acceptable threshold of 0.2µm for dental restoration materials (Bollen et al., 1997a; Çakmak, Donmez, et al., 2022b; Quirynen & Bollen, 1995). The mean *R*a of the Emax group before ageing (0.223µm) and after ageing (0.232µm) can be considered acceptable, given that a difference of less than 0.04 μm can be clinically negligible (Çakmak et al., 2023).

Earlier research comparing hybrid materials produced using additive and subtractive manufacturing methods has also reported similar findings regarding the surface roughness value of VSplus which is *Ra* below 0.2µm (Çakmak et al., 2023). Specifically, VSplus polished with the conventional polishing technique exhibited lower surface roughness after polishing compared to glazing, *Ra* values consistently measuring below 0.2 µm, as reported by Çakmak et al. (2023). This finding is consistent with the manufacturer's guidelines, which advocate for conventional polishing techniques. Specifically, the recommended procedure involves the use of pumice stone and polishing compound, with a critical emphasis on preventing resin overheating during the polishing process. This methodology ensures that the optimal surface quality is achieved following the post-curing phase (BEGO, 2019).

Meanwhile, previous study by Yuan et. al indicated that the surface roughness of lithium disilicate CAD (such as Emax CAD) specimens after all interventions with simulated thermocycling was below the clinical threshold of 0.2 µm (Yuan et al., 2018). However, the preparation methods utilized for the samples were different, specifically, the Emax CAD block samples were cut with a slow-speed diamond saw under water cooling (Isomet; Buehler) and subsequently glazed after crystallization. In contrast, this study utilized a milling machine to prepare the Emax CAD block samples, aiming to more closely replicate clinical conditions. The conventional polishing technique were chosen for both sample groups to standardise the sample preparation.

The type of material has a significant effect on the baseline surface roughness values. The type of restorative dental material affected surface roughness values, in this study there was a significant difference in surface roughness between VSplus with Emax, the composition, hardness, and polishability may affected their surface roughness these differences become evident due to their distinct compositions and fabrication processes, hence the significant difference in surface roughness both before and after ageing.

VSplus is a ceramic-filled hybrid resin material designed for 3D printing, the composition, including esterification products, silanized dental glass, and various photoinitiators with total content of 30% – 50% mass of inorganic fillers (particle size 0.7 µm) which provides a balanced ratio of hardness and flexibility (BEGO, 2020). This balance composition may facilitate easier polishing and results in a smoother surface, further, the precise control of 3D printing technology may contributes to the lower surface roughness values observed in the VSplus group.

Meanwhile, lithium disilicate CAD/CAM materials like IPS e.max CAD are known for their high strength and excellent aesthetic properties. These materials consist of approximately 40% lithium metasilicate (Li2SiO3) crystals and lithium disilicate (Li2Si2O5) crystal nuclei embedded in a glassy matrix (Furtado de Mendonca et al., 2019; Zarone et al., 2016). The intermediate crystallization process before milling and the subsequent post-milling crystallization result in a very hard material. While this hardness contributes to the strength and durability of the restorations, it also makes the material become more challenging to polish to the same smoothness level as hybrid resin materials. VSplus with its resin-based composition is easier to polish, resulting in smoother surfaces. In contrast, the harder lithium disilicate material requires more effort to achieve a comparable surface finish, leading to relatively higher surface roughness (Baldi et al., 2022).

5.2 Effect of Artificial Ageing (Thermocycling) on Surface Roughness

The simulated ageing with thermocycling significantly increases the surface roughness in both groups (p value< 0.05); hence, the null hypothesis is rejected. These results are in accordance with previous studies (Çakmak et al., 2023; Yuan et al., 2018)

The increase in surface roughness of VSplus group following artificial ageing might be due to the dissolution effect from the thermocycling process, which can lead to changes in surface texture (Çakmak et al., 2023). Water absorption causes expansion and plasticization of the resin, which leads to hydrolysis of the silane coupling agent and eventual loss of the surface fillers. In addition, the absorbed water can diffuse into the interface between the ceramic and polymeric phases, creating microcracks on the surface and further increasing the surface roughness (Toledano et al., 2003).

A previous study indicated that the surface roughness of lithium disilicate CAD/CAM ceramic restorations increased after the simulated brushing alone, thermocycling alone, and both interventions. This suggests that brushing and the thermocycling process can lead to surface wear and texture changes over time (Yuan et al., 2018).

Another study reported significant differences in surface roughness (*Ra* values) among the various CAD/CAM restorative material groups before and after thermal and mechanical cycling (TCML) ageing. They found that hybrid resin-ceramics exhibited higher surface roughness after ageing compared to lithium disilicate glass-ceramics**.** Despite that, the study did not include VarseoSmile Crown Plus, instead they included CS; Cerasmart to represent the hybrid resin-ceramic, but they did use the same material to represent lithium disilicate glass-ceramic which is IPS e.max CAD (S.-H. Kim et al., 2022).

Although ageing by thermal cycling does not involve direct physical contact with the sample surface, thermocycling can affect surface roughness in several ways such as expansion and contraction of the material, the repeated heating and cooling cycles can cause materials to expand and contract. This thermal stress can lead to micro-cracking or deformation of the surface, more prominent fissures and lines, hence increasing roughness (Çakmak et al., 2023; Morresi et al., 2014; Yuan et al., 2018). Thermal cycling can also facilitate the accumulation of contaminants or residues on the surface, which can further contribute to roughness (Çakmak et al., 2023).

Even though VSplus demonstrates significantly lower surface roughness compared to Emax both before and after inducing artificial ageing, the difference in *Ra* values before and after artificial ageing in VSplus group is greater than Emax group. This suggests that with prolonged thermocycling, the surface roughness of the VSplus group may increase more substantially than that of the Emax group. Consequently, its long-term stability may warrant further investigation, particularly in the context of extended ageing and clinical application. (Çakmak et al., 2023, S.-H. Kim et al., 2022, Yuan et al., 2018)

5.3 SEM Images Observation

A representative sample from each group was randomly selected before ageing and after ageing and examined at high magnifications (5,000x) to provide an in-depth view of the material surface's microstructure. Before ageing, the SEM images of VSplus reveal predominantly uniform filler particles within the resin matrix (Figure 4.3 (A)), whereby this uniformity is integral in maintaining the material’s mechanical properties and aesthetic quality. This may also reflect the efficiency of the polishing protocols employed. A well-executed polishing technique reduces the initial surface roughness (Ra) to a level of clinically acceptable thresholds and makes it less prone to accumulating stains and debris, which can contribute to roughness over time (Çakmak et al., 2023). After ageing, the VSplus surface appears rougher with the filler particles in VSplus become more irregular and layering lines from the 3D printing process become more pronounced (Figure 4.3 (B)). These changes are likely due to the degradation of the resin matrix (Çakmak, Donmez, et al., 2022a; Çakmak et al., 2023)

Meanwhile, before ageing, Emax’s SEM images reveal features of some lines and striation, and several pores (Figure 4.3 (C)). These features may reflect the distribution of the elongated, small, needle-shaped crystals that are closely integrated within the glassy matrix of the lithium disilicate (Furtado de Mendonca et al., 2019; Zarone et al., 2016). The presence of pores on the surface of Emax was due to the after-milling processes. These pores are attributed to the removal of highly soluble spherical lithium phosphate crystals during the milling of the material's intermediate state (Mota et al., 2017; Schestatsky et al., 2020). After ageing, the surface of Emax CAD shows more prominent distinct lines (Figure 4.3 (D)), which may contribute to the higher surface roughness value after ageing.

In summary, significant differences can be observed with both materials exhibiting increased surface roughness and microstructural changes. This observational finding of the SEM image changes aligns with the changes in the *Ra* value of both materials before and after artificial ageing. However, despite the changes at the microstructural level, the *Ra* value can still be maintained near the threshold value even after artificial ageing.

5.4 Limitations of Study and Future Improvements

While this study provides significant insights, it has some limitations. The study was conducted in vitro, and the clinical environment may introduce additional variables that could affect the results. Future studies should include long-term clinical evaluations to validate the findings in clinical settings. Thermocycling alone could not completely simulate intraoral situations as some factors, for instance, saliva, environmental pH or solution thermocycling, such as coffee thermocycling, were not included (Çakmak et al., 2023; Munusamy, 2020; Zarone et al., 2016). Moreover, no mechanical ageing, such as cyclic loading or brushing, which could deteriorate sample surfaces and affect tested parameters, was performed.

Additionally, the study only considered surface roughness as an indicator of material degradation. Other clinically relevant properties like surface wettability, colour stability, water absorption, dissolution rate, microhardness, fracture toughness, wear resistance, biocompatibility and biofilm adherence should be considered for further investigation to provide a more comprehensive assessment of material performance (Bollen et al., 1997b; Çakmak et al., 2023; Munusamy, 2020; Ozer et al., 2023; Schestatsky et al., 2019; Yuan et al., 2018)

CHAPTER 6: CONCLUSION

Within the limitation of this in vitro study, the following conclusions were drawn:

1. There is a significant increase in the surface roughness of the 3D printed crown material (Varseosmile Crown plus) after inducing artificial ageing
2. There is a significant increase in the surface roughness of the milled lithium disilicate crown material (IPS e.max CAD) after inducing artificial ageing
3. Between the 2 groups, the 3D printed crown material (Varseosmile Crown plus) has significantly lower surface roughness values both before and after ageing compared to the milled lithium disilicate crown material(IPS e.max CAD).
4. Microstructural changes can be observed in both the 3D printed (Varseosmile Crown plus) and milled lithium disilicate (IPS e.max CAD) crown material after ageing.

In summary, while both materials experienced an increase in surface roughness over time, both crown materials remained relatively stable with minimal changes after ageing, maintaining surface roughness close to the threshold of 0.2 µm indicating that they both maintain good surface properties despite the effects of ageing

REFERENCES

Abduo, J., Lyons, K., & Bennamoun, M. (2014). Trends in computer-aided manufacturing in prosthodontics: a review of the available streams. *International Journal of Dentistry*, *2014*, 783948.

Albrecht, N. (2020). *Abrasion and Surface Roughness 5-year Toothbrushing Simulation of VarseoSmile Crown plus*. S. B. Report.

Alharbi, N., Osman, R., & Wismeijer, D. (2016). Effects of build direction on the mechanical properties of 3D-printed complete coverage interim dental restorations. *Journal of Prosthetic Dentistry*, *115*(6), 760-767.

Amaral, F. L., Colucci, V., Palma-Dibb, R. G., & Corona, S. A. (2007). Assessment of in vitro methods used to promote adhesive interface degradation: a critical review. *Journal of Esthetic Restorative Dentistry*, *19*(6), 340-353; discussion 354.

Amaya-Pajares, S. P., Ritter, A. V., Vera Resendiz, C., Henson, B. R., Culp, L., & Donovan, T. E. (2016). Effect of Finishing and Polishing on the Surface Roughness of Four Ceramic Materials after Occlusal Adjustment. *Journal of Esthetic Restorative Dentistry*, *28*(6), 382-396.

Arafa, A. M., & Ghanem, L. (2023). Wear and Surface Roughness of 3D Printed and Milled CAD-CAM Restorative Materials. *Al-Azhar Journal of Dental Science*, *26*(2), 147-159.

Awada, A., & Nathanson, D. (2015). Mechanical properties of resin-ceramic CAD/CAM restorative materials. *The Journal of Prosthetic Dentistry*, *114*.

Baldi, A., Carossa, M., Comba, A., Alovisi, M., Femiano, F., Pasqualini, D., Berutti, E., & Scotti, N. (2022). Wear Behaviour of Polymer-Infiltrated Network Ceramics, Lithium Disilicate and Cubic Zirconia against Enamel in a Bruxism-Simulated Scenario. *Biomedicines*, *10*(7), 1682.

Bedran-de-Castro, A. K., Pereira, P. N., Pimenta, L. A., & Thompson, J. Y. (2004). Effect of thermal and mechanical load cycling on nanoleakage of Class II restorations. *The Journal of Adhesive Dentistry*, *6*(3), 221-226.

BEGO. (2019). *VarseoSmile Crown plus – Instructions for Use*. B. Report. https://www.bego.com

BEGO. (2020). *Summary of the scientific studies on VarseoSmile Crown plus*. B. Report. https://www.bego.com

Bollen, C. M., Lambrechts, P., & Quirynen, M. (1997a). Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. *Dental Materials*, *13*(4), 258-269.

Brecx, M., Theilade, J., & Attström, R. (1983). An ultrastructural quantitative study of the significance of microbial multiplication during early dental plaque growth. *Journal of Periodontal Research*, *18*(2), 177-186.

Bremer, F., Grade, S., Kohorst, P., & Stiesch, M. (2011). In vivo biofilm formation on different dental ceramics. *Quintessence International*, *42*(7), 565-574.

Brodine, B. A., Korioth, T. V., Morrow, B., Shafter, M. A., Hollis, W. C., & Cagna, D. R. (2021). Surface Roughness of Milled Lithium Disilicate With and Without Reinforcement After Finishing and Polishing: An In Vitro Study. *Journal of Prosthodontics*, *30*(3), 245-251.

Çakmak, G., Oosterveen-Rüegsegger, A. L., Akay, C., Schimmel, M., Yilmaz, B., & Donmez, M. B. (2023). Influence of polishing technique and coffee thermal cycling on the surface roughness and color stability of additively and subtractively manufactured resins used for definitive restorations. *Journal of Prosthodontics*.

Çakmak, G., Donmez, M. B., Atalay, S., de Paula, M. S., Fonseca, M., Schimmel, M., & Yilmaz, B. (2022). Surface roughness and stainability of CAD-CAM denture base materials after simulated brushing and coffee thermocycling. *Journal of Prosthetic Dentistry*.

Çakmak, G., Rusa, A. M., Donmez, M. B., Akay, C., Kahveci, Ç., Schimmel, M., & Yilmaz, B. (2022). Trueness of crowns fabricated by using additively and subtractively manufactured resin-based CAD-CAM materials. *Journal of Prosthetic Dentistry*.

Catalbas, B., Uysal, T., Nur, M., Demir, A., & Gunduz, B. (2010). Effects of thermocycling on the degree of cure of two lingual retainer composites. *Dental Materials Journal*, *29*(1), 41-46.

Cenci, M. S., Pereira-Cenci, T., Donassollo, T. A., Sommer, L., Strapasson, A., & Demarco, F. F. (2008). Influence of thermal stress on marginal integrity of restorative materials. *Journal of Applied Oral Science*, *16*(2), 106-110.

Chaudhary, R., Fabbri, P., Leoni, E., Mazzanti, F., Akbari, R., & Antonini, C. (2023). Additive manufacturing by digital light processing: a review. *Progress in Additive Manufacturing*, *8*(2), 331-351.

D'Amario, M., Campidoglio, M., Morresi, A. L., Luciani, L., Marchetti, E., & Baldi, M. (2010). Effect of thermocycling on the bond strength between dual-cured resin cements and zirconium-oxide ceramics. *Journal of Oral Science*, *52*(3), 425-430.

De Munck, J., Van Landuyt, K., Coutinho, E., Poitevin, A., Peumans, M., Lambrechts, P., & Van Meerbeek, B. (2005). Micro-tensile bond strength of adhesives bonded to Class-I cavity-bottom dentin after thermo-cycling. *Dental Materials*, *21*(11), 999-1007.

Doerr, C. L., Hilton, T. J., & Hermesch, C. B. (1996). Effect of thermocycling on the microleakage of conventional and resin-modified glass ionomers. *American Journal of Dentistry*, *9*(1), 19-21.

Dolev, E., Bitterman, Y., & Meirowitz, A. (2019). Comparison of marginal fit between CAD-CAM and hot-press lithium disilicate crowns. *The Journal of Prosthetic Dentistry*, *121*(1), 124-128.

Duret, F., Blouin, J.-L., & Duret, B. (1988). CAD-CAM in dentistry. *The Journal of the American Dental Association*, *117*(6), 715-720.

El-Araby, A. M., & Talic, Y. F. (2007). The effect of thermocycling on the adhesion of self-etching adhesives on dental enamel and dentin. *The Journal of Contemporary Dental Practice*, *8*(2), 17-24.

Ellakany, P., Fouda, S. M., Mahrous, A. A., AlGhamdi, M. A., & Aly, N. M. (2022). Influence of CAD/CAM Milling and 3D-Printing Fabrication Methods on the Mechanical Properties of 3-Unit Interim Fixed Dental Prosthesis after Thermo-Mechanical Aging Process. *Polymers*, *14*(19), 4103.

Elshiyab, S. H., Nawafleh, N., Öchsner, A., & George, R. (2018). Fracture resistance of implant-supported monolithic crowns cemented to zirconia hybrid-abutments: zirconia-based crowns vs. lithium disilicate crowns. *The Journal of Advanced Prosthodontics*, *10*(1), 65-72.

Eva Jerman, M. S. M. E. L. S., B.Sc.; Dr. Marcel Reymus; Prof. Dr. Dipl.-Ing. (FH) Bogna Stawarczyk, M.Sc. (2020). *Fracture load and two-body wear of 3D printed and conventionally fabricated crowns: artificial aging of 10 in-vivo years*. S. B. Report.

Fischer, H., Weber, M., & Marx, R. (2003). Lifetime Prediction of All-ceramic Bridges by Computational Methods. *Journal of Dental Research*, *82*(3), 238-242.

Flury, S., Lussi, A., & Zimmerli, B. (2010). Performance of Different Polishing Techniques for Direct CAD/CAM Ceramic Restorations. *Operative dentistry*, *35*, 470-481.

Fraga, S., Amaral, M., Bottino, M. A., Valandro, L. F., Kleverlaan, C. J., & May, L. G. (2017). Impact of machining on the flexural fatigue strength of glass and polycrystalline CAD/CAM ceramics. *Dental Materials*, *33*(11), 1286-1297.

Francisconi, L. F., Honório, H. M., Rios, D., Magalhães, A. C., Machado, M. d. A., & Buzalaf, M. A. R. (2008). Effect of erosive pH cycling on different restorative materials and on enamel restored with these materials. *Operative dentistry*, *33*(2), 203-208.

Fu, L., Engqvist, H., & Xia, W. (2020). Glass-Ceramics in Dentistry: A Review. *Materials (Basel)*, *13*(5).

Furtado de Mendonca, A., Shahmoradi, M., Gouvêa, C. V. D. d., De Souza, G. M., & Ellakwa, A. (2019). Microstructural and mechanical characterization of CAD/CAM materials for monolithic dental restorations. *Journal of Prosthodontics*, *28*(2), e587-e594.

Gale, M. S., & Darvell, B. W. (1999). Thermal cycling procedures for laboratory testing of dental restorations. *Journal of Dentistry*, *27*(2), 89-99.

Gibbs, C. H., Anusavice, K. J., Young, H. M., Jones, J. S., & Esquivel-Upshaw, J. F. (2002). Maximum clenching force of patients with moderate loss of posterior tooth support: a pilot study. *Journal of Prosthetic Dentistry*, *88*(5), 498-502.

Grzebieluch, W., Kowalewski, P., Grygier, D., Rutkowska-Gorczyca, M., Kozakiewicz, M., & Jurczyszyn, K. (2021). Printable and Machinable Dental Restorative Composites for CAD/CAM Application—Comparison of Mechanical Properties, Fractographic, Texture and Fractal Dimension Analysis. *Materials*, *14*(17), 4919.

Hatanaka, K., Irie, M., Tjandrawinata, R., & Suzuki, K. (2006). Effect of Thermocycling on Interfacial Gap-formation in Class V Cavities and Mechanical Properties of Spherical Silica Filler Addition to Resin-modified Glass Ionomer Restorations. *Dental Materials Journal*, *25*(4), 655-663.

Hisbergues M, V. S., Vendeville P. (2009). Zirconia: Established facts and perspectives for a biomaterial in dental implantology. *Journal of Biomedical Materials Research Part B*, *88*(2), 519-529.

Honório, H. M., Rios, D., Francisconi, L. F., Magalhães, A. C., Machado, M. A. d. A. M., & Buzalaf, M. A. R. (2008). Effect of prolonged erosive pH cycling on different restorative materials. *Journal of Oral Rehabilitation*, *35*(12), 947-953.

ISO. (1994). *Guidance on Testing of Adhesion to Tooth Structure. ISO/TR 11405* (Dental Materials, Issue. ISO.

ISO. (1997). *Geometrical Product Specifications (GPS) 4287:1997- Surface Texture: Profile Method - Terms, Definitions And Surface Texture Parameters*. ISO.

ISO. (2021). *ISO/ASTM 52900. Additive manufacturing- General principles-Fundamentals and vocabulary*. ISO.

J. Goldstein, D. N., D. Joy,, & C, L., P. Echlin, E. Lifshin, L. Sawyer, and J. Michae. (2003). *Scanning Electron Microscopy and X-Ray Microanalysis.* (3 ed.). Springer New York, NY.

Joda, T., Zarone, F., & Ferrari, M. (2017). The complete digital workflow in fixed prosthodontics: a systematic review. *BMC Oral Health*, *17*(1), 124.

Kara, D., Tekçe, N., Fidan, S., Demirci, M., Tuncer, S., & Balcı, S. (2021). The Effects of Various Polishing Procedures on Surface Topography of CAD/CAM Resin Restoratives. *Journal of Prosthodontics*, *30*(6), 481-489.

Kim, G. T., Go, H. B., Yu, J. H., Yang, S. Y., Kim, K. M., Choi, S. H., & Kwon, J. S. (2022). Cytotoxicity, Colour Stability and Dimensional Accuracy of 3D Printing Resin with Three Different Photoinitiators. *Polymers (Basel)*, *14*(5).

Kim, S.-H., Choi, Y.-S., Kang, K.-H., & Att, W. (2022). Effects of thermal and mechanical cycling on the mechanical strength and surface properties of dental CAD-CAM restorative materials. *The Journal of Prosthetic Dentistry*, *128*(1), 79-88.

Matalon, S., Safadi, D., Meirowitz, A., & Ormianer, Z. (2021). The Effect of Aging on the Roughness and Bacterial Adhesion of Lithium Disilicate and Zirconia Ceramics. *Journal of Prosthodontics*, *30*(5), 440-446.

Matzinger, M., Hahnel, S., Preis, V., & Rosentritt, M. (2019). Polishing effects and wear performance of chairside CAD/CAM materials. *Clinical Oral Investigations*, *23*(2), 725-737.

Mjor, I., Jokstad, A., & Qvist, V. (1990). Longevity of posterior restorations. *International Dental Journal*, *40*(1), 11-17.

Mohammad Ali Saghiri, K. A., Mehrdad Lotfi, Kasra Karamifar, Ali Mohammad Saghiri, Prasanna Neelakantan, James L. Gutmann, Ahmad Sheibaninia. (2012). Back-scattered and secondary electron images of scanning electron microscopy in dentistry: A new method for surface analysis. *Acta Odontologica Scandinavica*, 1-7.

Mohammadibassir, M., Rezvani, M. B., Golzari, H., Moravej Salehi, E., Fahimi, M. A., & Kharazi Fard, M. J. (2019). Effect of two polishing systems on surface roughness, topography, and flexural strength of a monolithic lithium disilicate ceramic. *Journal of Prosthodontics*, *28*(1), e172-e180.

Morresi, A. L., D'Amario, M., Capogreco, M., Gatto, R., Marzo, G., D'Arcangelo, C., & Monaco, A. (2014). Thermal cycling for restorative materials: does a standardized protocol exist in laboratory testing? A literature review. *Journal of Mechanical Behaviour of Biomedical Materials*, *29*, 295-308.

Mota, E. G., Smidt, L. N., Fracasso, L. M., Burnett Jr, L. H., & Spohr, A. M. (2017). The effect of milling and postmilling procedures on the surface roughness of CAD/CAM materials. *Journal of Esthetic and Restorative Dentistry*, *29*(6), 450-458.

Munusamy, S. M., Yap, A. U., Ching, H. L., & Yahya, N. A. (2020). Degradation of Computer-aided Design/Computer-aided Manufacturing Composites by Dietary Solvents: An Optical Three-dimensional Surface Analysis. *Operative dentistry*, *45*(4), E176–E184.

Naseer, M. U., Kallaste, A., Asad, B., Vaimann, T., & Rassõlkin, A. (2021). A Review on Additive Manufacturing Possibilities for Electrical Machines. *Energies*.

Nassani, M. Z., Ibraheem, S., Shamsy, E., Darwish, M., Faden, A., & Kujan, O. (2021). A Survey of Dentists' Perception of Chair-Side CAD/CAM Technology. *Healthcare*, *9*(1), 68.

Özarslan, M., Bilgili Can, D., Avcioglu, N. H., & Çalışkan, S. (2022). Effect of different polishing techniques on surface properties and bacterial adhesion on resin-ceramic CAD/CAM materials. *Clinical Oral Investigations*, *26*(8), 5289-5299.

Özel Bektas, Ö., Eren, D., Herguner Siso, S., & Akin, G. E. (2012). Effect of thermocycling on the bond strength of composite resin to bur and laser treated composite resin. *Lasers Medical Science*, *27*(4), 723-728.

Ozer, N. E., Sahin, Z., Yikici, C., Duyan, S., & Kilicarslan, M. A. (2023). Bacterial adhesion to composite resins produced by additive and subtractive manufacturing. *Odontology*, 1-12.

Patel, T., Nathwani, N., Fine, P., & Leung, A. (2022). A Scoping Review of Marginal and Internal Fit Accuracy of Lithium Disilicate Restorations. *Dentistry Journal*, *10*(12), 236.

Pilecco, R. O., Dalla-Nora, F., Guilardi, L. F., Pereira, G. K. R., de Andrade, G. S., de Melo, R. M., Valandro, L. F., & Rippe, M. P. (2021). In-lab simulation of CAD/CAM milling of lithium disilicate glass-ceramic specimens: Effect on the fatigue behavior of the bonded ceramic. *Journal of the Mechanical Behavior of Biomedical Materials*, *121*, 104604.

Quirynen, M., & Bollen, C. M. (1995). The influence of surface roughness and surface-free energy on supra- and subgingival plaque formation in man. A review of the literature. *Journal of Clinical Periodontology*, *22*(1), 1-14.

Revilla-León, M., & Özcan, M. (2017). Additive Manufacturing Technologies Used for 3D Metal Printing in Dentistry. *Current Oral Health Reports*, *4*, 1-8.

Rezaie, H. R., Rizi, H. B., Khamseh, M. M. R., & Öchsner, A. (2020). *A review on dental materials*. Springer.

Saboia, V. P., Silva, F. C., Nato, F., Mazzoni, A., Cadenaro, M., Mazzotti, G., Giannini, M., & Breschi, L. (2009). Analysis of differential artificial ageing of the adhesive interface produced by a two-step etch-and-rinse adhesive. *European Journal of Oral Sciences*, *117*(5), 618-624.

Sailer, I., Benic, G. I., Fehmer, V., Hämmerle, C. H., & Mühlemann, S. (2017). Randomized controlled within-subject evaluation of digital and conventional workflows for the fabrication of lithium disilicate single crowns. Part II: CAD-CAM versus conventional laboratory procedures. *The Journal of Prosthetic Dentistry*, *118*(1), 43-48.

Sakaguchi, R. L., & Powers, J. M. (2011). *Craig's Restorative Dental Materials-E-Book: Craig's Restorative Dental Materials-E-Book*. Elsevier Health Sciences.

Schestatsky, R., Zucuni, C. P., Dapieve, K. S., Burgo, T. A. L., Spazzin, A. O., Bacchi, A., Valandro, L. F., & Pereira, G. K. R. (2020). Microstructure, topography, surface roughness, fractal dimension, internal and marginal adaptation of pressed and milled lithium-disilicate monolithic restorations. *Journal of Prosthodontic Research*, *64*(1), 12-19.

Schestatsky, R., Zucuni, C. P., Venturini, A. B., de Lima Burgo, T. A., Bacchi, A., Valandro, L. F., & Rocha Pereira, G. K. (2019). CAD-CAM milled versus pressed lithium-disilicate monolithic crowns adhesively cemented after distinct surface treatments: Fatigue performance and ceramic surface characteristics. *Journal of Mechanical Behavior of Biomedical Materials*, *94*, 144-154.

Schuckar, M., & Geurtsen, W. (1997). Proximo-cervical adaptation of Class II-composite restorations after thermocycling: a quantitative and qualitative study. *Journal of Oral Rehabilitation*, *24*(10), 766-775.

Shi, H. Y., Pang, R., Yang, J., Fan, D., Cai, H., Jiang, H. B., Han, J., Lee, E. S., & Sun, Y. (2022). Overview of Several Typical Ceramic Materials for Restorative Dentistry. *BioMed Research International*, *2022*, 8451445.

Stewardson, D. A., Shortall, A. C., & Marquis, P. M. (2010). The effect of clinically relevant thermocycling on the flexural properties of endodontic post materials. *Journal of Dentistry*, *38*(5), 437-442.

Stokes, D. (2008). Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy (VP-ESEM). *Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy (VP-ESEM)*, 1-221.

Szczesio-Wlodarczyk, A., Fronczek, M., Ranoszek-Soliwoda, K., Grobelny, J., Sokolowski, J., & Bociong, K. (2022). The First Step in Standardizing an Artificial Aging Protocol for Dental Composites-Evaluation of Basic Protocols. *Molecules*, *27*(11).

Tahayeri, A., Morgan, M., Fugolin, A. P., Bompolaki, D., Athirasala, A., Pfeifer, C. S., Ferracane, J. L., & Bertassoni, L. E. (2018). 3D printed versus conventionally cured provisional crown and bridge dental materials. *Dental Materials*, *34*(2), 192-200.

Tekçe, N., Fidan, S., Tuncer, S., Kara, D., & Demirci, M. (2018). The effect of glazing and aging on the surface properties of CAD/CAM resin blocks. *Journal of Advanced Prosthodontics*, *10*(1), 50-57.

Temizci, T., & Bozoğulları, H. N. (2024). Effect of thermocycling on the mechanical properties of permanent composite-based CAD-CAM restorative materials produced by additive and subtractive manufacturing techniques. *BMC Oral Health*, *24*(1), 334.

Toledano, M., Osorio, R., Osorio, E., Fuentes, V., Prati, C., & García-Godoy, F. (2003). Sorption and solubility of resin-based restorative dental materials. *Journal of Dentistry*, *31*(1), 43-50.

Ulker, M., Ozcan, M., Sengün, A., Ozer, F., & Belli, S. (2010). Effect of artificial aging regimens on the performance of self-etching adhesives. *Journal of Biomedical Materials Research Part B*, *93*(1), 175-184.

Vasiliu, R. D., Porojan, S. D., Bîrdeanu, M. I., & Porojan, L. (2020). Effect of Thermocycling, Surface Treatments and Microstructure on the Optical Properties and Roughness of CAD-CAM and Heat-Pressed Glass Ceramics. *Materials (Basel)*, *13*(2).

Vivadent, I. (2016). *IPS e.max CAD Monolithic Solutions Chairside: Instructions for Use*. S. Ivoclar Vivadent AG, Liechtenstein.

Vivadent, I. (2017). *IPS e.max CAD - LAB: Monolithic Solutions LABSIDE Instructions for Use*. S. Ivoclar Vivadent AG, Liechtenstein.

Vo, D. T., Arola, D., Romberg, E., Driscoll, C. F., Jabra-Rizk, M. A., & Masri, R. (2015). Adherence of Streptococcus mutans on lithium disilicate porcelain specimens. *The Journal of Prosthetic Dentistry*, *114*(5), 696-701.

Wahab, F. K., Shaini, F. J., & Morgano, S. M. (2003). The effect of thermocycling on microleakage of several commercially available composite Class V restorations in vitro. *Journal of Prosthetic Dentistry*, *90*(2), 168-174.

Wegner, S. M., Gerdes, W., & Kern, M. (2002). Effect of different artificial aging conditions on ceramic-composite bond strength. *International Journal of Prosthodontics*, *15*(3), 267-272.

Xie, C., Han, Y., Zhao, X. Y., Wang, Z. Y., & He, H. M. (2010). Microtensile bond strength of one- and two-step self-etching adhesives on sclerotic dentin: the effects of thermocycling. *Operative dentistry*, *35*(5), 547-555.

Yuan, J. C., Barão, V. A. R., Wee, A. G., Alfaro, M. F., Afshari, F. S., & Sukotjo, C. (2018). Effect of brushing and thermocycling on the shade and surface roughness of CAD-CAM ceramic restorations. *Journal of Prosthetic Dentistry*, *119*(6), 1000-1006.

Zafar, M. S., Amin, F., Fareed, M. A., Ghabbani, H., Riaz, S., Khurshid, Z., & Kumar, N. (2020). Biomimetic Aspects of Restorative Dentistry Biomaterials. *Biomimetics (Basel)*, *5*(3).

Zarone, F., Ferrari, M., Mangano, F. G., Leone, R., & Sorrentino, R. (2016). “Digitally oriented materials”: focus on lithium disilicate ceramics. *International Journal of Dentistry*, *2016*(1), 9840594.

Zhou, L., Miller, J., Vezza, J., Mayster, M., Raffay, M., Justice, Q., Al Tamimi, Z., Hansotte, G., Sunkara, L. D., & Bernat, J. (2024). Additive Manufacturing: A Comprehensive Review. *Sensors (Basel)*, *24*(9).

Zhou, W., & Wang, Z. L. (2007). *Scanning Microscopy for Nanotechnology. Techniques and Applications*.