



Universidade do Minho
Escola de Engenharia

Development and CAD/CAM production of
ultra-thin, high translucent 3Y-TZP zirconia veneers

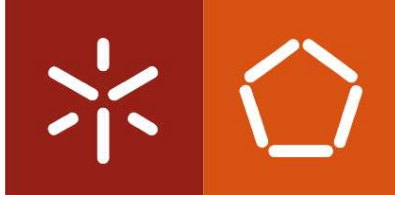
Paulo Filipe Salgado Pinto

Development and CAD/CAM production of
ultra-thin, high translucent 3Y-TZP
zirconia veneers

Paulo Filipe Salgado Pinto

Uminho | 2026

janeiro de 2026



Universidade do Minho

Escola de Engenharia

Paulo Filipe Salgado Pinto

**Development and CAD/CAM production of
Ultra-Thin, High Translucent 3Y-TZP Zirconia
Veneers**

Tese de Doutoramento

Programa Doutoral em Engenharia Mecânica

Trabalho efetuado sob a orientação de

Professor Doutor Filipe Samuel Pereira Correia da Silva

janeiro de 2026

DIREITOS DE AUTOR E CONDIÇÕES DE UTILIZAÇÃO DO TRABALHO POR TERCEIROS

Este é um trabalho académico que pode ser utilizado por terceiros desde que respeitadas as regras e boas práticas internacionalmente aceites, no que concerne aos direitos de autor e direitos conexos. Assim, o presente trabalho pode ser utilizado nos termos previstos na licença abaixo indicada. Caso o utilizador necessite de permissão para poder fazer um uso do trabalho em condições não previstas no licenciamento indicado, deverá contactar o autor, através do RepositóriUM da Universidade do Minho.

Licença concedida aos utilizadores deste trabalho



Atribuição-NãoComercial-SemDerivações CC BY-NC-ND

<https://creativecommons.org/licenses/by-nc-nd/4.0/>

Universidade do Minho, ____/____/____

Assinatura: _____

Acknowledgments

This PhD thesis marks the culmination of a demanding journey on every level – intellectually, emotionally, and personally. None of this would have been possible without the support, inspiration, and, above all, the constant presence of truly special people.

To my supervisor, Professor Filipe Silva, I express my deepest gratitude: for the initial encouragement, for the trust placed in me, and for his continuous availability throughout this journey. A true example of leadership and human excellence.

To my colleagues and friends at the Microfabrication and Integrated Systems Laboratory, thank you for every moment of help, camaraderie, and support. A special thank you to Óscar, Sara, and Zé Pires for your direct help with this work, and to Rita, João Pinto, and Flávio for your support in other ways – your generosity made this path lighter.

To all those from other labs or departments who, directly or indirectly, contributed to the development of this work, my sincere thanks – Miguel, Leonor and Joel.

To my family – always the most important part of everything – my eternal love. To my wife, Eduarda, and my daughters, Margarida and Mafalda, the women of my life: your love, support, and presence were my true source of strength and inspiration.

To my parents, Custódia and João, who have always encouraged me and stood by my side through every stage of life – all that I am, I owe to you. To my brother João, a true example of resilience – having you as a brother is a privilege.

To my parents-in-law, Conceição and Manuel, thank you for always treating me as one of your own.

To my sisters-in-law, Inês and Marinha, and my brother-in-law, Nuno, thank you for your constant good spirits and support. To my dear niece Beatriz, an example of courage and fearlessness; to my nephew Gonçalo, a source of intelligence and positivity; and to my godson Alexandre, for his kind heart and inner strength. Each of your qualities has been a powerful source of inspiration.

To the memory of my grandmother Josefa – you will always be my guardian angel.

To my STAFF friends – António, Bruno, Gonçalo and Silva – thank you for making everything lighter with your joy and friendship. A toast to you, and also to Ana, Danielle, Daniela and Diana, who always welcomed and supported this spirit of friendship! A special word of thanks to Bruno, a long-time friend, who was at the origin of this journey and whose pursuit of excellence continues to be a true inspiration.

Finally, I gratefully acknowledge the Foundation for Science and Technology (FCT) for the financial support provided through the PhD grant SFRH/BD/145421/2019. I also thank the Centre for Micro-Electro Mechanical Systems (CMEMS) for the opportunity to work within the scope of the projects UIDB/04436/2020 and UIDP/04436/2020.

To all those who were part of this journey – thank you!

STATEMENT OF INTEGRITY

I hereby declare having conducted this academic work with integrity. I confirm that I have not used plagiarism or any form of undue use of information or falsification of results along the process leading to its elaboration.

I further declare that I have fully acknowledged the Code of Ethical Conduct of the University of Minho.

University of Minho, ____/____/____

Assinatura: _____

Paulo Filipe Salgado Pinto

Resumo

Desenvolvimento e produção CAD/CAM de facetas ultra-finas e com elevada translucência em Zircônia 3Y-TZP

Na procura por soluções restauradoras estéticas e minimamente invasivas, as facetas ultrafinas em zircónia surgem como uma abordagem promissora. Esta tese de doutoramento aborda o desenvolvimento, a produção CAD/CAM e a avaliação funcional de facetas ultrafinas produzidas a partir de zircónia tetragonal estabilizada com 3 mol% de ítria (3Y-TZP). Estas restaurações visam aliar elevada translucência, excelente resistência mecânica com o intuito de se promover as restaurações minimamente invasivas, respondendo assim às exigências clínicas atuais. Foi desenvolvido um programa experimental abrangente e multidisciplinar, estruturado em quatro áreas principais: comportamento ao desgaste, resistência adesiva e influência da pressão de cimentação e desempenho ótico em espessuras ultrafinas. Os resultados demonstraram que a zircónia 3Y-TZP apresenta elevada resistência ao desgaste, mantendo a rugosidade superficial abaixo do limiar clínico de retenção bacteriana, mesmo após 16 anos de simulação de escovagem. Os ensaios de adesão revelaram uma forte correlação inversa entre a pressão aplicada e a espessura da camada de cimento, com os melhores valores de resistência obtidos sob pressão controlada e camadas finas. A análise ótica confirmou que, em espessuras $\leq 0,15$ mm, a zircónia 3Y-TZP atinge níveis de translucência comparáveis ou mesmo superiores aos da zircónia altamente translúcida 5Y-PSZ.

Adicionalmente, foi validado um fluxo digital completo para a produção de uma faceta com 0,15 mm de espessura, confirmando a sua precisão dimensional e viabilidade para aplicações clínicas sem preparação. Conclui-se que as facetas ultrafinas em 3Y-TZP, com espessuras entre 0,15–0,30 mm, são viáveis do ponto de vista clínico, permitindo a preservação da estrutura dentária saudável, ao mesmo tempo que garantem estabilidade funcional e estética a longo prazo. A abordagem proposta está totalmente alinhada com os princípios da odontologia minimamente invasiva e abre novas perspectivas para restaurações anteriores à base de zircónia.

Palavras-chave (5): *3Y-TZP; CAD/CAM; faceta ultrafina; odontologia adesiva; zircónia.*

Abstract

Development and CAD/CAM production of Ultra-Thin, High Translucent 3Y-TZP Zirconia Veneers

In the pursuit of aesthetic and minimally invasive restorative solutions, ultra-thin veneers made of zirconia emerge as a promising approach. This PhD thesis addresses the development, CAD/CAM-based production, and functional evaluation of ultra-thin veneers made from 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP). These restorations aim to combine high translucency, excellent mechanical strength, and minimal removal of dental structure, meeting current clinical demands. A comprehensive and multidisciplinary experimental program was developed, structured around three main areas: wear behavior, adhesive bond strength and the influence of cementation pressure, and optical performance at ultra-thin thicknesses. The results demonstrated that 3Y-TZP zirconia exhibits high wear resistance, maintaining surface roughness below the clinical threshold for bacterial retention even after 16 years of simulated toothbrushing. Adhesion tests revealed a strong inverse correlation between applied pressure and cement thickness, with the best bond strength values obtained under controlled pressure and thin cement layers. Optical analysis confirmed that at thicknesses ≤ 0.15 mm, 3Y-TZP zirconia reaches translucency levels comparable to or even exceeding those of highly translucent 5Y-PSZ zirconia.

Moreover, a fully digital workflow for producing a 0.15 mm veneer was validated, confirming its dimensional accuracy and feasibility for non-prep clinical applications. It is concluded that ultra-thin 3Y-TZP veneers, with thicknesses between 0.15–0.30 mm, are clinically viable, allowing for the preservation of healthy tooth structure while ensuring long-term functional and aesthetic stability. The proposed approach is fully aligned with the principles of minimally invasive dentistry and opens new perspectives for anterior zirconia-based restorations.

Keywords (5): *3Y-TZP; adhesive dentistry; CAD/CAM; ultra-thin veneer; zirconia.*

Contents

Acknowledgments	iii
Resumo	vi
Abstract	vii
List of symbols, abbreviations and notations	xi
List of figures	xii
List of tables.....	xv
CHAPTER 1. Introduction	1
1.1 Motivation.....	2
1.2 Objectives.....	3
1.3 Structure of the thesis.....	4
References	6
CHAPTER 2. State of the art	8
2.1 Tooth Structure and Function	9
2.2 Dental Restorations.....	12
2.2.1 Inlays/Onlays.....	13
2.2.2 Crowns and Bridges	13
2.2.3 Veneers	15
2.3 The evolution of dental veneers	15
2.4 Dental materials used in restorations	17
2.4.1 Metals.....	19
2.4.1.1. Stainless Steel.....	19
2.4.1.2. Cobalt-Chromium alloys.....	21
2.4.1.3. Titanium and its alloys.....	23
2.4.2 Polymers.....	25
2.4.2.1 Poly(methyl) methacrylate (PMMA)	26
2.4.2.2 Polyetheretherketone (PEEK)	27
2.4.3 Composites.....	30
2.4.3.1 Resin-based composites for direct veneers	30
2.4.3.2 Thermally Polymerized Composites (Laboratory-Fabricated).....	33
2.4.3.3 Reinforced CAD/CAM composites.....	34
2.4.4 Ceramics	36
2.4.4.1 Traditional dental ceramics.....	37
2.4.4.2 Glass ceramics.....	38
2.4.4.2.1 Leucite reinforced feldspathic ceramics.....	38
2.4.4.2.2 Lithium disilicate	39
2.4.4.3 High strength core ceramics	39
2.4.5 Zirconia based ceramics.....	41
2.5 Adhesion	44
2.6 CAD/CAM Indirect restoration processing.....	45
2.7 Zirconia properties evaluation.....	50
2.7.1 Tribological evaluation	50
2.7.2 Bond strength evaluation.....	52
2.7.3 Optical properties evaluation	54

References	55
CHAPTER 3. Comparative toothbrush abrasion resistance and surface analysis of different dental restorative materials	66
Abstract	67
3.1 Introduction	67
3.2 Materials and methods	69
3.2.1 Specimen preparation	69
3.2.2 Simulated toothbrushing	70
3.2.3 Surface characterization	71
3.2.4 Surface roughness analysis	71
3.2.5 Color difference evaluation	71
3.2.6 Toothbrushing abrasion resistance	73
3.2.7 Surface characterization	73
3.3 Results and Discussion	73
3.3.1. Surface morphological characterization	73
3.3.2. Surface roughness.....	75
3.3.3. Color and lightness difference	76
3.3.4. Toothbrushing abrasion resistance.....	80
3.4 Conclusions.....	81
Acknowledgements.....	82
References	82
CHAPTER 4. Influence of applied pressure and thickness variation on the bond strength between 3Y-TZP zirconia and self-adhesive resin cement..	85
Abstract	86
4.1 Introduction	86
4.2 Materials and methods	88
4.2.1 Specimen preparation	88
4.2.2 Surface treatment on zirconia surfaces	89
4.2.3 Physical bonding process to resin-matrix cement	89
4.2.4 Characterization methods.....	92
4.2.4.1 Surface morphology characterization	92
4.2.4.2 Shear bond strength test	92
4.2.4.3 Statistical analysis	93
4.3 Results	94
4.3.1 Surface and roughness characterization	94
4.3.2 Zirconia to resin-matrix cement interface thickness analysis	94
4.3.3 Shear Bond Strength.....	95
4.3.4 Correlation between cement layer thickness and shear bond strength	96
4.4 Discussion.....	97
4.5 Conclusions.....	101
Acknowledgements.....	101
References	102
CHAPTER 5. Can 3Y-TZP Zirconia reach 5Y-PSZ Zirconia translucency?	106
Abstract	107
5.1. Introduction.....	107

5.2 Materials and Methods	109
5.2.1 Specimens preparation	109
5.2.2 Optical measurements	111
5.2.3 Microstructure analysis/Grain Size	113
5.3 Results and Discussion	113
5.3.1 Optical properties and translucency behaviour	113
5.3.2 Microstructural characterization and grain size impact	120
5.3.3 Flexible behavior in Ultra-Thin 3Y-TZP Zirconia and potential applications	123
5.4 Conclusions.....	125
Acknowledgements.....	126
References	126
CHAPTER 6. Digital workflow for ultra-thin 3Y-TZP dental veneers:	
CAD/CAM manufacturing approach	131
6.1 Introduction	132
6.2 Acquisition of the digital model.....	133
6.3 CAD design of the veneer.....	134
6.4 CAM and milling parameters.....	137
6.5 Sintering process and inspection	139
6.6 Surface finishing and surface treatment	142
6.7 Discussion and Critical Considerations	145
References	146
CHAPTER 7. General discussion and conclusions.....	148
7.1 General discussion and conclusions.....	149
7.2 Suggestions for Future Works	150

List of symbols, abbreviations and notations

3Y-TZP	3 mol% yttria-stabilized tetragonal zirconia polycrystals
4Y-PSZ	4 mol% yttria partially stabilized zirconia
5Y-PSZ	5 mol% yttria partially stabilized zirconia
CAD/CAM	Computer-Aided Design / Computer-Aided Manufacturing
RDA	Relative Dentin Abrasivity
MPa	Megapascal
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive X-ray Spectroscopy
Ra	Average surface roughness
Rz	Average maximum height of the profile
SBS	Shear Bond Strength
N	Newton
Y ₂ O ₃	Yttrium oxide
ZrO ₂	Zirconium dioxide (zirconia)
SiO ₂	Silicon dioxide
Al ₂ O ₃	Aluminium oxide (alumina)
CeO ₂	Cerium oxide
HAp	Hydroxyapatite
N/A	Not Applicable
SD	Standard Deviation

List of figures

Chapter 2

Figure 2.1 - Classification and types of permanent human teeth [1].	9
Figure 2.2 - Cross-sectional anatomy of a canine tooth, illustrating the crown and root regions, and identifying key structural components such as enamel, dentin, pulp chamber, periodontal membrane, cementum, and alveolar bone. Adapted from Ferraro's Fundamentals of Maxillofacial Surgery [3].	10
Figure 2.3 - Microstructural features of enamel, dentin, and the enamel–dentin interface. Adapted from Ferracane et al. [4].	11
Figure 2.4 - Illustrative differences between inlay, onlay, and crown restorations [19].	13
Figure 2.5 - Illustration of dental crown placement [21].	14
Figure 2.6 - Steps involved in the placement of a dental bridge (adapted from [22]).	14
Figure 2.7 - Demonstration of veneer placement [23].	15
Figure 2.8 - Comparison between two types of dental veneer preparation. a) Conventional approach involving significant dental tissue removal to accommodate traditional ceramic veneers; b) Minimally invasive technique associated with ultra-thin “contact lens” veneers, requiring little to no enamel reduction and preserving the original tooth structure.	17
Figure 2.9 - Clinical example of stainless steel crowns used in pediatric dentistry for the restoration of primary molars [Case Reports in Dentistry].	20
Figure 2.10 - Cobalt-Chromium alloy frameworks [50].	22
Figure 2.11 - Schematic illustration showing the main components that form the basic structure of dental resin composites [74].	31
Figure 2.12 - Classification diagram of ceramic materials used in dental restorations.	37
Figure 2.13 - Intraoral Scanner from 3 Shape® [128].	47
Figure 2.14 - DWX 51D five-axis milling unit by Roland DG. (A) Overview of the equipment used for milling ceramic restorations. (B) Detail of the zirconia disc fixation process within the milling unit. (C) Close-up of the simultaneous milling of multiple structures in the zirconia block. [Adapted from Roland DG, Japan].	48
Figure 2.15 - Different types of blocks used in the production of dental restorations through milling [133].	49

Chapter 3

Figure 3.1 - Schematic representation of toothbrushing tests.	71
Figure 3.2 - SEM images of the surface topography of specimens before (left column) and after 8h of toothbrushing (right column). Polymer (a, a1), composite (b, b1), feldspathic porcelain (c, c1) and zirconia (d, d1).	74
Figure 3.3 - Variation of surface roughness, Ra after 8h of toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).	76
Figure 3.4 - Color change (ΔE_{00}) for each material after 8h of simulated toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).	78
Figure 3.5 - Evolution of color change for each dental material caused by toothbrushing with time.	78

Figure 3.6 - Lightness difference for each material after 8h of simulated toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).....	79
Figure 3.7 - Evolution of lightness difference for each material caused by toothbrushing with time.	80
Figure 3.8 - Specific wear rate of the tested restorative dental materials. The asterisks (*) indicate statistically significant differences ($p < 0.05$).....	81

Chapter 4

Figure 4.1 - Sequential depiction of the 3Y-TZP zirconia cylinders' production process, encompassing machining from a pre-sintered block, sintering under specified conditions, and subsequent polishing to achieve a smooth finish.	89
Figure 4.2 - Sequential representation of the bonding procedure, detailing the application of the self-adhesive resin cement and controlled assembly of the paired zirconia cylinders for shear bond strength testing.....	90
Figure 4.3 - Custom-designed bonding device for zirconia cylinders. (A) Components; (B) Unload position (C) Load position.	91
Figure 4.4 - Schematic of the shear test device	93
Figure 4.5 - Scanning electron microscopy (SEM) images of zirconia surfaces after grit-blasting with Al_2O_3 particles (50 μm) at 2 bar, at magnifications of x1000, x2000 and x10000.	94
Figure 4.6 - Cross-Sectional scanning electron microscopy (SEM) images of zirconia-resin cement interface under different applied pressures. The quantitative thickness values (mean $\pm$ SD) are provided below each image.....	95
Figure 4.7 - Mean shear bond strength (SBS) and standard error of the mean (SEM) across different applied pressure conditions. The asterisks (*) indicate statistically significant differences ($p < 0.05$).	96
Figure 4.8 - Scatter plot showing the correlation between cement layer thickness (μm) and shear bond strength (MPa). The fitted quadratic regression curve ($R^2=0.82$) indicates a non-linear relationship.	97

Chapter 5

Figure 5.1 - Schematic representation of the experimental procedure used in this study: samples production with different thicknesses, and optical and microstructure analysis.	111
Figure 5.2 - Nomenclature system for sample identification, categorized by Zirconia type and respective thicknesses.	111
Figure 5.3 - Visual Representation for Qualitative Analysis of Translucency in 3Y-TZP, 4Y-PSZ, and 5Y-PSZ Zirconia Specimens. At thickness levels of 1.00, 0.45, 0.30, 0.15, and 0.05 mm, highlighting the observable translucency variations.	114
Figure 5.4 - Translucency parameter (TP) as a function of ceramic thickness (mm) for three types of zirconia in this study: 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. Error bars represent the standard deviation (SD) from three independent specimens, with three measurements per specimen.	115
Figure 5.5 - Contrast Ratio (CR) as a function of ceramic thickness (mm) for three types of zirconia in this study: 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. Error bars represent the standard deviation (SD) from three independent specimens, with three measurements per specimen.....	117
Figure 5.6 - Light transmittance through zirconia ceramics as a function of thickness (0.05 to 1.00 mm) for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconias. Figures 6a, 6b, and 6c illustrate the transmittance	

behaviour of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ at various thicknesses, respectively. Figure 6d compares the transmittance values of 3Y-TZP (at 0.15 mm and 0.05 mm) with 4Y-PSZ and 5Y-PSZ at higher thicknesses (0.30 mm, 0.45 mm, and 1.00 mm).....	118
Figure 5.7 - Grain size (μm) as a function of thickness (mm) for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconias. Error bars represent the standard deviation (SD)	121
Figure 5.8 - Scanning Electron Microscopy (SEM) images at 10,000x magnification showing the grain structure of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconia ceramics at two thicknesses: 0.05 mm (top row) and 1.00 mm (bottom row).....	122
Figure 5.9 - Zirconia sample (3Y-TZP, 0.05 mm thickness) held by a fixation device. In image A, the sample is not subjected to any applied force, while in image B, a force is applied to demonstrate the flexibility of the zirconia, as it returns to its original position after the force is removed.....	124
Chapter 6	
Figure 6.1 - Realistic anatomical dental model used (NISSIN Dental Products, Japan).	132
Figure 6.2 - Medit i500 intraoral scanner (A) and scanning process (B).....	133
Figure 6.3 - Three-dimensional model of the maxillary and mandibular arches of the anatomical training model used in this study.....	134
Figure 6.4 - ExoCAD software interface for restoration type selection.....	135
Figure 6.5 – Margin line creation step (green).	136
Figure 6.6 – Superimposition of the veneer (white) over the CAD model.	136
Figure 6.7 - Thickness verification of the veneer.	137
Figure 6.8 - Virtual representation of the zirconia blank holder and milling position of the veneer (centered).....	138
Figure 6.9 - Different stages of the virtual milling simulation. A – Start of roughing on side L1; B – End of roughing on side L2; C – Finishing on side L1; D – Finishing on side L2.	139
Figure 6.10 - Veneer centered on the refractory base inside the Zirkonofen 700 furnace.	140
Figure 6.11 - Thermal cycle used for sintering the 3Y-TZP zirconia veneer.....	140
Figure 6.12 - Final appearance of the veneer after sintering.....	141
Figure 6.13 - Dimensional inspection using ImageJ software (6 $\times$ magnification).....	142
Figure 6.14 - Zirconia Adjustment & Polishing Kit (DIATECH, Switzerland) for zirconia finishing and polishing.....	143
Figure 6.15 - Polishing sequence of the vestibular surface of the veneer. (A) Initial positioning and inspection of the veneer; (B) Pre-polishing using a green silicone disc (97SG14RA); (C) Continued pre-polishing; (D) High-gloss polishing	143
Figure 6.16 - Internal surface grit-blasting with 50 μm Al_2O_3 , 2 bar pressure, 15 mm distance, 20 seconds.....	144
Figure 6.17 - Final ultra-thin 3Y-TZP zirconia veneer ready for try-in.....	144
Figure 6.18 - Aesthetic integration assessment of the veneer in the anatomical model. (A) Model without veneer; (B) Model with veneer placed on tooth 11; (C) Color difference map indicating a small interproximal discontinuity (arrow indicates interproximal area).....	145

List of tables

Chapter 2

Table 2.1 - Comparative mechanical properties of metals discussed in this thesis: Stainless steel 316L, Co-Cr alloy, Titanium and Ti-6Al-4V [48,56].	25
Table 2.2 - Comparative table of the properties of the polymeric materials discussed, PMMA and PEEK. [52,60,63,66,67]	29
Table 2.3 – Comparison of some properties of composites [84-86].	36
Table 2.4 - Mechanical and thermal properties of 3Y-TZP zirconia [103,104]	43
Table 2.5 - General milling parameters for pre-sintered zirconia [Roland DG].	48

Chapter 3

Table 3.1 - Dental restorative materials tested in this study	70
Table 3.2 - Initial Ra surface roughness (mean $\pm$ SD) after polishing.	75
Table 3.3 - Initial color (E_{00}) values of each type of tested material.	77

Chapter 4

Table 4.1 - Chemical composition of the commercial 3Y-TZP zirconia used in this study (wt%).	88
Table 4.2 - Standard composition (wt%) of the self-adhesive and autopolymerizing resin cement SpeedCEM Plus, as provided by the manufacturer (Ivoclar Vivadent, Liechtenstein).	90
Table 4.3 - Experimental groups categorized according to applied force (N) and corresponding applied pressure (kPa), calculated based on a constant contact area of 50.27 mm ² . Each group consisted of 10 pairs of zirconia cylinders ($n_1 = 10$).	92

Chapter 5

Table 5.1 - Summary of the chemical composition (wt%) and mechanical properties of Zirconia materials used (3Y-TZP, 4Y-PSZ, 5Y-PSZ) (Manufacturer-provided data).	110
Table 5.2 - Manufacturer's specified sintering instructions for different types of zirconia blocks.	110

Chapter 6

Table 6.1 - Accuracy of IOS Medit i500 usado neste estudo. * Accuracy test in vitro was conducted by Yonsei University College of Dentistry according to the methods in "Accuracy comparison analysis on scan data of single tooth, quadrant, and full arch between two types of intraoral scanner, i500 and Trios 3" Ji-Man Park, 19 December 2018. ** Accuracy test in vivo was conducted by Medit	133
Table 6.2 - Design parameters in ExoCAD for the ultra-thin veneer	135
Table 6.3 - Initial parameters defined in HyperDent Compact software	138

CHAPTER 1.

Introduction

1.1 Motivation

The increasing aesthetic demands of patients, combined with the search for minimally invasive and long-lasting solutions, have significantly transformed restorative clinical practice. The smile has become a key factor in self-esteem and well-being, especially among adolescents and young adults [1–3]. Simultaneously, the growing appreciation of dental aesthetics as a component of health and quality of life has driven substantial growth in the field of cosmetic dentistry worldwide.

The global cosmetic dentistry market was valued at approximately **€27.8 billion in 2022**, with projections estimating a value exceeding **€98.6 billion by 2032**, representing a compound annual growth rate (CAGR) of **13.5%** [4]. In Europe, the dental CAD/CAM market is expected to grow from around **€400 million in 2021 to €750 million by 2028**, with a CAGR of **9.3%** [5].

In Portugal, the *Barómetro de Saúde Oral 2023* [6] revealed that only **41.1% of individuals aged 15 or older have a complete dentition** (excluding third molars), and **59% are missing at least one natural tooth**. This scenario, combined with a growing rate of regular dental visits (**64.4% of the population**) and an increasing concern for dental aesthetics, highlights a significant potential for innovative, minimally invasive, high-quality restorative solutions.

In this context, **ceramic veneers** have become one of the most effective solutions for anterior aesthetic rehabilitation, with high long-term success rates when properly indicated and cemented. However, materials such as feldspathic porcelain and lithium disilicate require minimum thicknesses **above 0.5 mm**, often necessitating irreversible enamel removal. Literature reports [7] that **preserving ≥ 80% of enamel** at the adhesive interface is a critical factor for the clinical longevity of ceramic veneers, as bonding to enamel provides superior strength and durability, while exposure to dentin is associated with significantly higher failure rates.

A 2024 review by Alghazzawi [8] confirmed **that dental veneers** show a clinical survival rate **greater than 90% over 10 years**, particularly when applied **with minimal or no preparation**. The amount of preserved enamel was the most decisive factor in long-term success. Fracture was identified as the main failure mechanism, followed by debonding and color changes, with higher incidence in patients with parafunctional habits.

Accordingly, there has been increasing interest in the development of **ultra-thin veneers** (< 0.3 mm), which can be applied with minimal or no tooth preparation, without compromising

aesthetics or function. Among the available materials, **3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP)** stands out for its flexural strength > 900 MPa, high fracture toughness, proven biocompatibility, and low rejection rate. However, its initial opacity limited its use in aesthetic zones.

Advancements in CAD/CAM technology have enabled the fabrication of restorations with high precision and aesthetic quality, significantly reducing clinical time and treatment cost. Despite this, the production of 3Y-TZP veneers with < 0.3 mm thickness using high-precision CAD/CAM processes remains poorly documented in the literature. Existing studies focus mostly on simplified specimens that do not reflect the geometric complexity, real thickness, or surface treatment of actual veneers. Furthermore, critical variables such as abrasion resistance, bonding pressure, resin cement layer thickness, and optical behavior have not been sufficiently investigated in this context.

This thesis aims to address this gap by developing and characterizing ultra-thin veneers made from 3Y-TZP zirconia, fabricated using a patented CAD/CAM-based process. The investigation is structured around three main axes:

- (i) abrasion resistance of ceramic surfaces subjected to simulated toothbrushing (Chapter 3);
- (ii) bonding performance of ultra-thin zirconia veneers to self-adhesive resin cements, as a function of cementation pressure and resin cement thickness (Chapter 4);
- (iii) comparative analysis of the optical properties of 3Y-TZP and 5Y-PSZ zirconias (Chapter 5).

Chapter 6 presents, the complete fabrication workflow of a clinically shaped, ultra-thin 3Y-TZP zirconia veneer, based on the developed method.

In an increasingly demanding clinical environment, these solutions aim to contribute to the development of restorative materials that combine advanced aesthetics, functional longevity, and maximum preservation of dental structure.

1.2 Objectives

The quality and longevity of an indirect restoration strongly depend on the optical, microstructural, and adhesive properties of the material used, as well as on the precision of its manufacturing process. In the case of ultra-thin veneers made of 3Y-TZP zirconia, the technical

challenge lies in maintaining structural integrity and natural aesthetics at reduced thicknesses, while combining high-precision CAD/CAM processing with appropriate bonding protocols.

This thesis aims to develop, characterize, and validate the application of ultra-thin veneers in translucent 3Y-TZP zirconia, fabricated through a patented process based on CAD/CAM technology. The investigation is structured around three main axes: tribological, adhesive, and optical.

Accordingly, the following general objectives were defined:

- To explore the technical feasibility of producing ultra-thin 3Y-TZP zirconia veneers using CAD/CAM technology;
- To characterize the tribological, adhesive, microstructural, and optical performance of 3Y-TZP zirconia.

To accomplish these general goals, the following specific objectives were established:

- To evaluate the wear resistance and surface stability of 3Y-TZP zirconia subjected to simulated toothbrushing, and to compare its performance with other restorative materials (polymers, composites, and glass-ceramics);
- To investigate the influence of cementation pressure and resin cement layer thickness on the adhesive strength between 3Y-TZP zirconia and self-adhesive resin cements, considering surface modification;
- To analyze the translucency and optical properties of 3Y-TZP zirconia in comparison with more translucent zirconias, from conventional thicknesses (≥ 0.45 mm) down to ultra-thin thicknesses (0.05 mm), in order to determine how microstructure and yttria content affect translucency;
- To describe in detail the CAD/CAM manufacturing and finishing process of a clinically shaped, ultra-thin 3Y-TZP zirconia veneer, applying the method developed in this thesis.

1.3 Structure of the thesis

This PhD thesis is organized as a compilation of scientific articles, published or submitted to internationally indexed ISI journals. In addition to the three research papers, the thesis also

includes four additional chapters: Chapter 1 (Introduction), Chapter 2 (State of the Art), and Chapters 6 and 7, dedicated to the practical development and general discussion, respectively.

Chapter 1 – Introduction presents the motivation for the work, the general and specific objectives, and the overall structure of the thesis.

Chapter 2 – State of the Art provides an in-depth review of the relevant literature on dental anatomy, direct and indirect restoration types, dental materials used in aesthetic veneers, adhesive protocols, and CAD/CAM processing techniques, with particular emphasis on 3Y-TZP zirconia. This chapter establishes the scientific foundation for exploring new solutions for ultra-thin veneers, highlighting the limitations of current materials and techniques, and the knowledge gaps that the present research aims to address.

Chapter 3 – "Tribological behavior and surface analysis of restorative dental materials subjected to simulated toothbrushing" corresponds to a research article published in *Tribology International* (Elsevier, Volume 175, 2022, 107799; impact factor 6.1, Quartile 1). This study evaluates the abrasion behavior and surface integrity of various restorative materials, including 3Y-TZP zirconia, after simulated toothbrushing, providing a relevant comparison for ultra-thin veneers.

Chapter 4 – "Influence of applied pressure and thickness variation on the bond strength between 3Y-TZP zirconia and self-adhesive resin cement" presents a research article published in the *Journal of Biomedical Materials Research Part B: Applied Biomaterials* (Volume 113, March 2025; impact factor 3.2, Quartile 2). This study investigates the influence of cementation pressure and veneer thickness (with a focus on ultra-thin configurations) on the adhesive strength of 3Y-TZP zirconia bonded to self-adhesive resin cements.

Chapter 5 – "Can 3Y-TZP Zirconia reach 5Y-PSZ Zirconia translucency?" corresponds to a research article submitted to the *Journal of Biomedical Materials Research Part B: Applied Biomaterials* in April 2025. This work provides a comparative analysis of the optical

properties—particularly translucency—of highly translucent 3Y-TZP zirconia versus 5Y-PSZ zirconia, critically assessing their suitability for ultra-thin veneer applications.

Chapter 6 – Digital Workflow for Ultra-Thin 3Y-TZP Dental Veneers: CAD/CAM

Manufacturing Approach offers a practical description of the fabrication process of a real clinical veneer in 3Y-TZP zirconia with a thickness of 0.15 mm. The chapter details each step of the workflow, including design, milling, sintering and finishing, based on the patented method developed within the scope of this thesis. This chapter bridges the gap between laboratory investigation and clinical application.

Chapter 7 – General discussion and conclusions presents a general discussion and conclusions of the developed work (Development and CAD/CAM production of Ultra-Thin, High Translucent 3Y-TZP Zirconia Veneers) and some suggestions for future work.

References

- [1] L. Gavic, M. Budimir, A. Tadin, The association between self-esteem and aesthetic component of smile among adolescents, *Prog. Orthod.* 25 (2024) 4–11. <https://doi.org/10.1186/s40510-023-00508-w>.
- [2] A. Militi, F. Sicari, M. Portelli, E.M. Merlo, A. Terranova, F. Frisone, R. Nucera, A. Alibrandi, S. Settineri, Psychological and social effects of oral health and dental aesthetic in adolescence and early adulthood: An observational study, *Int. J. Environ. Res. Public Health.* 18 (2021) 2–9. <https://doi.org/10.3390/ijerph18179022>.
- [3] L.G. Davis, P.D. Ashworth, L.S. Spriggs, Psychological effects of aesthetic dental treatment, *J. Dent.* 26 (1998) 547–554. [https://doi.org/10.1016/S0300-5712\(97\)00031-6](https://doi.org/10.1016/S0300-5712(97)00031-6).
- [4] P.M. Research, Cosmetic Dentistry Market to Gain USD 106.43 Billion by 2032, (2024). <https://www.globenewswire.com/news-release/2024/01/26/2817631/0/en/Cosmetic-Dentistry-Market-to-Gain-USD-106-43-Billion-by-2032-According-to-Persistence-Market-Research.html> (accessed October 8, 2024).
- [5] Healthcare IT, Europe Dental CAD/CAM Market Forecast to 2028, (2022) 206. <https://www.businessmarketinsights.com/reports/europe-dental-cad-cam-market> (accessed October 8, 2024).

- [6] Ordem dos Médicos Dentistas, Barómetro da Saúde oral 8a edição | Portugal 2023, (2023) 55.
- [7] R.L.. Lago, S. R.; Marçal, Durability rate of ceramic laminates : literature review, Scire Salut. 12 (2022) 248–258. <https://doi.org/10.6008/CBPC2236-9600.2022.003.0028>.
- [8] T.F. Alghazzawi, Clinical Survival Rate and Laboratory Failure of Dental Veneers: A Narrative Literature Review, J. Funct. Biomater. 15 (2024). <https://doi.org/10.3390/jfb15050131>.

CHAPTER 2.

State of the art

2.1 Tooth Structure and Function

Teeth are anatomical structures of the human body whose primary functions are food mastication and phonetics, particularly in sound articulation and speech. In addition, and especially in contemporary society, teeth play a crucial role in human aesthetics.

The complete permanent dentition of an adult human consists of 32 teeth, with 16 located in the maxilla (upper arch) and 16 in the mandible (lower arch). For easier identification, these 32 teeth are conventionally divided into four quadrants, each comprising 2 incisors, 1 canine, 2 premolars, and 3 molars.

Teeth can also be grouped according to their position in the mouth: anterior and posterior teeth. Anterior teeth are the most visible and are located at the front (incisors and canines), while posterior teeth include the remaining premolars and molars. Figure 2.1 illustrates the different classes and types of teeth in the permanent human dentition.

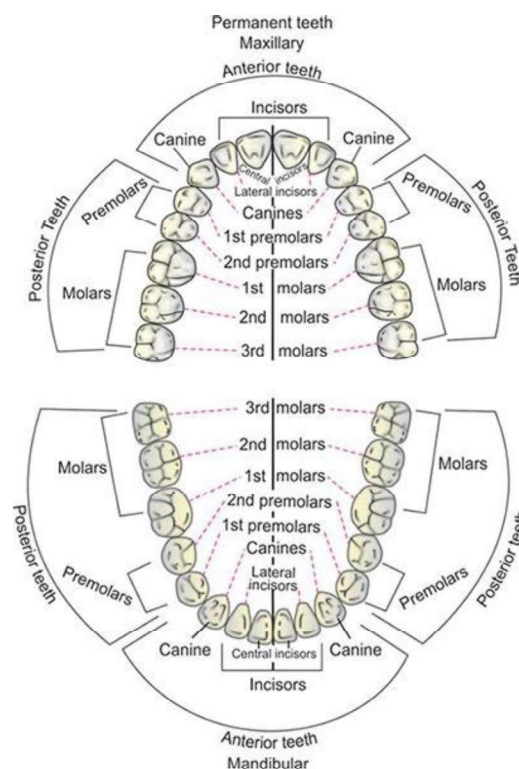


Figure 2.1 - Classification and types of permanent human teeth [1].

Teeth, being responsible for a variety of functions, are anatomically described in three regions: the crown, the neck, and the root.

The **crown** is typically recognized as the free or visible part of the tooth, and its shape is crucial for the specific function of each type of tooth. Accordingly, the crown appears flat and blade-

like in incisors, making them suitable for cutting food; pointed in canines, ideal for tearing; and broad and flat in premolars and molars, suited for grinding—together enabling proper mastication.

The **neck** is the transitional area between the crown and the root, located at the level of the gingival margin. It is a narrow region that provides both structural and functional connection between these two parts. The **root**, in turn, is embedded in the alveolar bone and its primary role is to anchor the tooth within the bony socket, ensuring its stability [2].

Figure 2.2 illustrates the anatomy of a tooth, specifically a cross-sectional view of a canine tooth, showing its internal structures. For the purposes of this study, the most relevant tissues are the enamel and dentin, as these are typically the areas involved in tooth preparation for restorative procedures.

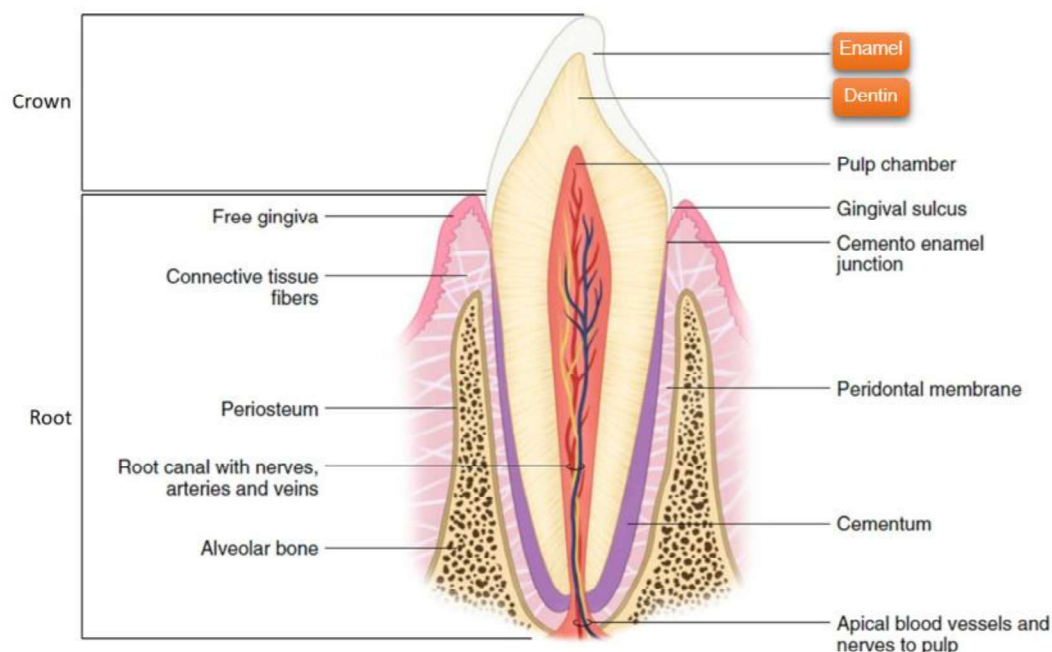


Figure 2.2 - Cross-sectional anatomy of a canine tooth, illustrating the crown and root regions, and identifying key structural components such as enamel, dentin, pulp chamber, periodontal membrane, cementum, and alveolar bone. Adapted from Ferraro's Fundamentals of Maxillofacial Surgery [3].

The structure of the tooth is predominantly composed of hydroxyapatite (HAp), a crystalline calcium phosphate mineral.

Enamel is the outermost layer of the tooth crown. Characterized by its white, smooth, and glossy appearance, it is the most mineralized tissue in the human body, which gives it a high degree of hardness. Its main function is to cover the crown and protect the underlying dentin. Enamel is almost entirely mineralized, containing only a small amount of organic matrix mainly protein (3 vol%) and water (12 vol%) [4].

At the microstructural level, enamel crystals are arranged in long, rod-like structures with a well-defined radial anisotropy, as shown in Figure 2.3 [5].

Dentin, located immediately beneath the enamel, is the most extensive dental tissue and is less mineralized than enamel. It is composed of approximately 47 vol% mineral content, 33 vol% protein, primarily collagen, and about 20 vol% water. Its tubular structure (Figure 2.3) is less dense near the enamel and becomes denser toward the pulp. Therefore, its properties vary depending on its location within the tooth [5].

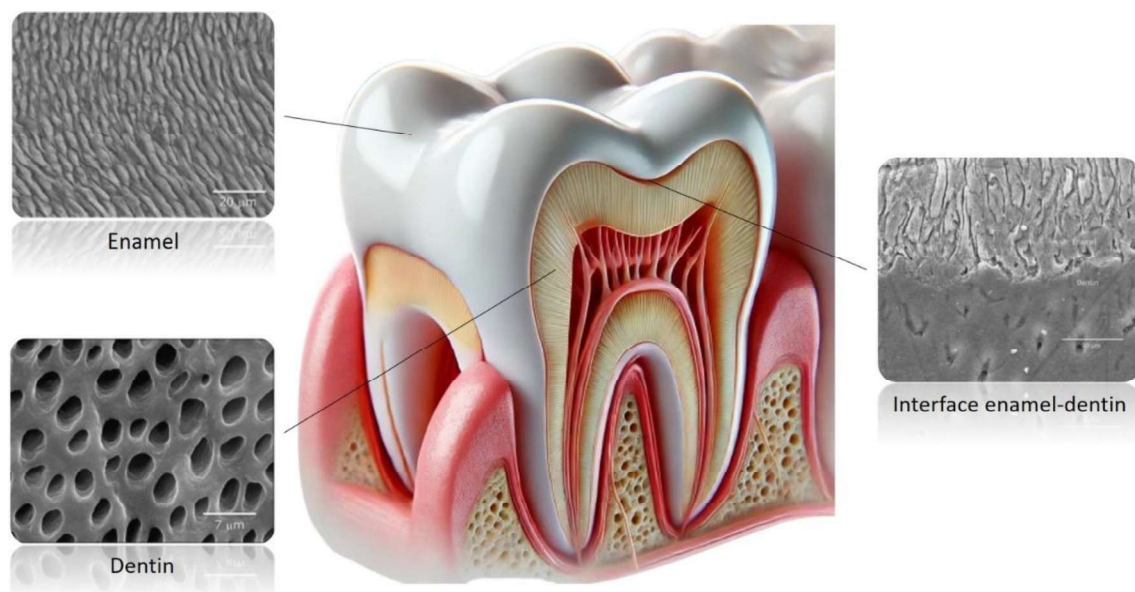


Figure 2.3 - Microstructural features of enamel, dentin, and the enamel–dentin interface. Adapted from Ferracane et al. [4].

Both enamel and dentin exhibit unique mechanical and structural properties. Enamel has a high modulus of elasticity, ranging from 70 to 110 GPa, which grants it considerable hardness—approximately 5 to 6 times higher than that of dentin. However, this structure is also more brittle, with relatively low tensile strength (10–70 MPa) and fracture toughness ranging from 0.7 to 2.1 MPa√m, depending on the direction of the enamel prisms [5]. Despite this brittleness, enamel demonstrates significant resistance to masticatory forces, exhibiting stress-strain behaviour similar to certain metals [6].

Dentin, by contrast, has a much lower modulus of elasticity (~ 20 GPa) compared to enamel, yet it is significantly more flexible, presenting a tensile strength of between 60–100 MPa and fracture toughness ranging from 1.5–2.1 MPa√m. This flexibility is primarily attributed to its higher organic content and a collagen-rich tubular matrix structure [5]. Such structure provides dentin the

capability to absorb stress, thereby protecting the integrity of the tooth even under intense loading conditions [7,8].

Between enamel and dentin lies the enamel-dentin junction (Figure 2.3), a gradual transition measuring approximately 10 to 100 μm [4]. This junction plays a fundamental role in stress dissipation, acting as a barrier that prevents crack propagation into the enamel and harmoniously distributes masticatory forces [7]. The thickness of these layers varies according to factors such as age, gender, and tooth type. Enamel thickness typically ranges from 0.7 to 1.8 mm, while dentin thickness ranges approximately from 0.8 to 1.5 mm [9,10], depending on the tooth's location and function.

Having provided context regarding the dental tissues most relevant to this study enamel and dentin it is important to describe the factors that may lead to the necessity for dental restoration. In general, tooth deterioration may occur when certain bacteria produce lactic acid. This lactic acid lowers the pH on the tooth surface to values below 5, thereby promoting dissolution of hydroxyapatite and consequent deterioration [11]. Tooth discoloration may also occur due to extrinsic factors, such as smoking or drinking wine and coffee, or intrinsic causes, such as discoloration resulting from dentin degradation that becomes visible through enamel due to its natural translucency [12].

2.2 Dental Restorations

Dental restoration involves treatments aimed at rehabilitating the function, integrity, and structure of one or several teeth affected by caries, trauma, or discoloration [13]. Literature indicates that approximately 60% of dental clinic workload is related to the placement and replacement of restorations [14, 15]. These restorations can be broadly divided into two basic types: direct restorations—completed in a single dental visit—and indirect restorations, which require multiple visits due to the complexity of the procedure or the necessity of involving a dental prosthetics laboratory to complete the treatment [16].

The choice of a direct restoration typically applies when the patient's tooth structure is sufficiently intact and healthy. However, minor tooth preparation may be necessary to provide adequate space for the restorative material.

Indirect restorations generally involve multiple and more complex procedures, including components fabricated in a dental laboratory, which may be located either within the dental clinic

itself or externally. In these cases, while the patient awaits the final restorative component, the dentist places a temporary restoration to prevent further damage. Examples of indirect restorations include inlays/onlays, veneers, bridges, and crowns.

2.2.1 Inlays/Onlays

The distinction between inlays and onlays is not consensual; however, many authors argue that an inlay is placed on the chewing surface between the tooth cusps, whereas an onlay covers one or more cusps.

Generally, inlays and onlays are forms of indirect restorations used when a molar or premolar is too damaged to support a basic filling, not yet damaged enough to necessitate a crown. These restorations are prepared outside the patient's mouth and subsequently fitted and cemented into the cavity. Figure 2 4 illustrates the differences between inlays, onlays, and crowns.

Inlays/onlays are intended to rebuild a large portion of a tooth's chewing surface, whereas fillings are direct restorations designed to fill a smaller hole in the enamel. Overall, inlays and onlays are not as extensive as crowns, which typically cover most of the tooth structure. Onlays, sometimes referred to as partial crowns, may be employed when more than half of the tooth's chewing surface is decayed or requires repair [17] [18].



Figure 2.4 - Illustrative differences between inlay, onlay, and crown restorations [19]

2.2.2 Crowns and Bridges

A dental crown is an artificial cap fabricated in a dental laboratory used to cover a damaged tooth. This cap precisely replicates the geometry of the tooth it is designed to cover, fitting accurately over it. The crown is bonded to the pre-treated tooth using specialized resins and

adhesives. When properly designed, dental crowns closely resemble natural teeth and are indistinguishable from them. Dentists may recommend a dental crown for several reasons, particularly when it is necessary to protect, restore, or stabilize a damaged tooth. Like all dental restorations, crowns allow the restoration of both the appearance and functionality of the affected tooth [20] [21]. Figure 2 5 illustrates the placement of a dental crown.



Figure 2.5 - Illustration of dental crown placement [21]

A dental bridge is a fixed dental prosthesis used to replace one or more missing teeth within the dental arch. At least one tooth on either side of the missing tooth is required to anchor and support the bridge. A Figure 2 6 shows four illustrative steps involved in the placement of a dental bridge.

A dental bridge can serve as an alternative to a dental implant in situations where, for example, the patient's bone structure is inadequate for implant accommodation. Patients often favor this alternative as it is considered a less invasive procedure.



Figure 2.6 - Steps involved in the placement of a dental bridge (adapted from [22]).

2.2.3 Veneers

A veneer is a thin layer of material placed over a tooth to correct or mask various conditions such as discoloration; to close diastemas (gaps) between teeth; to modify tooth shape and alignment; or even to whiten the entire labial segment [13]. Figure 2 7 shows the placement of a dental veneer.



Figure 2.7 - Demonstration of veneer placement [23]

For many years, the most predictable and long-lasting aesthetic correction of anterior teeth was achieved through the preparation of crowns rather than veneers. However, this approach was undoubtedly more invasive, involving the removal of significant amounts of healthy dental tissue, which could potentially result in adverse effects on the adjacent pulp and periodontal tissues [24, 25].

Significant progress in adhesive techniques—both to enamel and dentin—enabled the introduction of hybrid resin composites, leading to less invasive treatments. Nevertheless, such restorations had limited longevity, as these composites are prone to discoloration, wear, and fracture, thus compromising long-term aesthetic outcomes [26, 27].

2.3 The evolution of dental veneers

In today's world, increasing social pressure to achieve ever-higher standards of beauty has led to an unprecedented demand for aesthetic solutions. Focusing specifically on the smile, dental veneers are applied to anterior teeth with the aim of correcting their shape or color to meet the patient's expectations. However, the indication for this type of aesthetic restoration may be limited in the presence of conditions such as bruxism¹ [28].

¹ The international community of bruxism experts has defined Sleep Bruxism as “a masticatory muscle activity during sleep that is characterized as rhythmic (phasic) or nonrhythmic (tonic) and is not a movement disorder or a sleep disorder in otherwise healthy individuals.” Awake Bruxism has been

The popularity of dental veneers stems not only from the final aesthetic result they provide but also from their minimally invasive nature, often involving little or no removal of healthy dental tissue.

In the 1920s, American dentist Dr. Charles Pincus applied the first temporary veneers to Hollywood actors [29], enhancing their appearance during filming. With advances in adhesive dentistry and the development of new materials such as porcelain and resin composites, the 1960s saw the emergence of more effective adhesive systems, allowing veneers to be fixed more permanently [30]. In the 1980s, the use of enamel acid etching and the introduction of porcelain as the main material for veneers significantly improved adhesion, achieving a good balance between aesthetics and strength [31,32], although still limited [feldspathic porcelain – 70–90 MPa] [33].

Technological progress, particularly from the 1990s onwards, led to the development of CAD/CAM systems—digital tools that enable the scanning, 3D modeling, and additive or subtractive manufacturing of dental restorations. This advancement greatly enhanced the customization and precision of veneers, thereby improving patient comfort and restoration durability [34]. What was once used exclusively for aesthetic purposes could now also address functional problems, such as tooth spacing and wear [35].

In the years following 2010, the concept of dental contact lenses emerged. These ultra-thin veneers have thicknesses ranging from 0.2 to 0.5 mm and require minimal or no enamel reduction, thereby preserving the original tooth structure [33]. This contrast with conventional preparation methods is illustrated in Figure 2.8.

defined as “a masticatory muscle activity during wakefulness that is characterized by repetitive or sustained tooth contact and/or by bracing or thrusting of the mandible and is not a movement disorder in otherwise healthy individuals.



Figure 2.8 - Comparison between two types of dental veneer preparation. a) Conventional approach involving significant dental tissue removal to accommodate traditional ceramic veneers; b) Minimally invasive technique associated with ultra-thin “contact lens” veneers, requiring little to no enamel reduction and preserving the original tooth structure.

It is important to note that enamel removal may lead to adverse effects, such as increased tooth sensitivity, changes in surface roughness that favor bacterial plaque accumulation, and potentially reduced tooth strength [36,37].

This thesis focuses on this type of ultra-thin veneer, produced using a patented manufacturing method, which allows for thicknesses below 0.2 mm and is intended for clinical cases in which no removal of dental tissue is required.

2.4 Dental materials used in restorations

Dental materials play a critical role in replacing or modifying tooth structure, whether for aesthetic or functional purposes. Throughout the history of dentistry, the challenge has been to develop and select materials that are biocompatible, durable, and suitable for either direct restorations or indirect prostheses, while also withstanding the harsh conditions of the oral cavity.

In this context, engineering plays a crucial role in the development and selection of dental materials, taking into account not only mechanical and biological properties but also optical and functional aspects that ensure the longevity and aesthetics of restorations. This is essential for materials to endure the challenges of the oral environment, such as masticatory forces, temperature variations, and exposure to chemical agents.

Recent advancements in dental materials are the result of significant progress achieved through multidisciplinary scientific knowledge, aiming to strike a balance between structural strength, biocompatibility, and aesthetics.

As described by Anusavice (2013) [39], dental materials can be classified into three main categories: **preventive**, **restorative**, and **auxiliary materials**.

Preventive materials include sealants applied to pits and fissures, agents that block infiltration, materials with antibacterial properties, and substances such as protective coatings, liners, and cements that release fluoride, chlorhexidine, or other therapeutic compounds intended to prevent or slow down the development of carious lesions.

Restorative materials, on the other hand, encompass a wide range of synthetic components used to restore or replace dental structures. These include primers, bonding agents, protective coatings, base cements, amalgams, resin composites, compomers, hybrid ionomers, cast metals, metal-ceramics, all-ceramics, and denture polymers. These materials may be used for temporary and short-term treatments (such as provisional cements or resins for temporary crowns and bridges), as well as for definitive solutions such as inlays, onlays, crowns, veneers, removable dentures, fixed dentures, and other orthodontic applications.

Restorative materials can be further subdivided into direct and indirect restorative materials. Direct materials are applied directly within the oral cavity, enabling the construction of restorations or prosthetic devices directly onto the teeth or soft tissues. Indirect materials, on the other hand, are manipulated outside the oral cavity, being molded or fabricated using replicas of teeth or tissues—such as gypsum models or CAD/CAM technology.

In addition, there are **auxiliary materials**, which play a crucial role in the production of dental prostheses and other devices, supporting their fabrication. Examples include impression materials, casting substances, gypsum for models, and other materials used in the manufacturing process and by the clinician during procedures.

Over the years, the evolution of dental restorative materials has been guided by the pursuit of mimicking the natural characteristics of teeth, following the principles of biomimetics² [40]. Advances to date demonstrate a continuous effort to reproduce the natural appearance of teeth, with particular emphasis on mechanical, aesthetic, and optical properties.

This thesis focuses on an **indirect restorative material: 3Y-TZP zirconia**. The following sections will briefly cover general classes of **metals and polymers**, which are not typically used for the fabrication of dental veneers. In contrast, more emphasis will be given to materials

² Biomimetics refers to the study of multi-disciplinary mechanisms and biologically produced materials to design novel products to mimic nature [40,154].

commonly applied in veneers, such as **composites**, and finally **ceramics**, which include 3Y-TZP zirconia – the material selected and tested in this work.

2.4.1 Metals

Metals have played a central role in the evolution of dental restorations throughout history. The Etruscans³, for instance, used bones and gold wires to replace missing teeth. In the early 20th century, modern dentistry began utilizing precious metals such as gold, along with experimental use of materials like zinc, steel, copper, and brass [41]. However, fluctuations in the price of gold—particularly the extreme increase in the 1980s stimulated the development of more affordable alternative metal alloys suitable for clinical use.

Nowadays, base metal alloys, which do not contain precious metals, are widely used due to their specific properties and lower cost. Among the most relevant alloys in dental practice are stainless steel, cobalt-chromium (Co-Cr) alloys, and titanium and its alloys. These materials, progressively introduced throughout the 20th century, continue to be essential in dental restorations due to their strength, durability, and adaptability to a wide range of clinical applications.

2.4.1.1. Stainless Steel

Stainless steel, particularly medical-grade 316L, is widely used in dentistry for dental crowns, orthodontic brackets, and other restorative devices due to its corrosion resistance, durability, and mechanical properties [42]. This material is especially effective in the restoration of primary teeth and permanent molars, particularly in pediatric dentistry (Figure 2.9), where stainless steel crowns are adapted to fully cover the surfaces of affected teeth. Its composition, containing iron (Fe), chromium (Cr), nickel (Ni), and molybdenum (Mo), enables the formation of a passive chromium oxide layer that protects the material against chemical degradation.

³ The Etruscans were an ancient civilization that inhabited the region of Etruria, in what is now central Italy. This society flourished between the 9th and 1st centuries BCE, prior to the rise of the Roman Empire.



Figure 2.9 - Clinical example of stainless steel crowns used in pediatric dentistry for the restoration of primary molars [Case Reports in Dentistry]

Some of its properties are presented in Table 2.1, including a tensile strength of 515 MPa and a hardness of 155 HV, which ensure robustness and wear resistance. Its yield strength of 170 MPa and elongation of 49% provide ductility and the ability to withstand cyclic loads—essential for dental applications such as crowns and orthodontic brackets. The modulus of elasticity, at 193 GPa, guarantees the rigidity needed for dimensional stability [43,44].

Although generally well tolerated, localized corrosion may occur under adverse conditions such as acidic pH or the presence of bacterial biofilms. Moreover, nickel release can trigger allergic reactions in sensitive patients [42]. Aesthetically, its metallic shine limits its use in visible areas.

Advantages:

- High corrosion resistance: The chromium content in stainless steel forms a passive layer that protects against chemical reactions, especially in humid environments like the oral cavity;
- Durability: Offers long service life due to its resistance to mechanical and chemical wear;
- Relatively low cost: Compared to noble materials such as precious metal alloys, stainless steel is economically accessible;
- Ease of adjustment and placement: Stainless steel crowns are pre-fabricated, reducing clinical chair time.

Disadvantages:

- Metal ions release: Under certain conditions, such as in the presence of bacterial biofilms (e.g., *Porphyromonas gingivalis*), the release of nickel, iron, and chromium ions can occur, potentially triggering adverse reactions like allergies or toxicity;
- Limited aesthetics: Its metallic appearance is not ideal for visible areas, making it less appealing for adult patients;
- Risk of stress corrosion: In acidic environments or in the presence of sulfur compounds, issues like intergranular corrosion may arise, compromising the material's structural integrity.

2.4.1.2. Cobalt-Chromium alloys

Cobalt-chromium (Co-Cr) alloys are widely used in dentistry due to their excellent mechanical properties, high corrosion resistance, and good biocompatibility. This alloy is predominantly composed of cobalt (55–65 wt.%) and chromium (25–30 wt.%), with the addition of molybdenum (4–5 wt.%), tungsten, nickel, iron, and other elements. Chromium (25–30%) is essential for forming a passive oxide layer that protects the material from chemical degradation, while molybdenum reduces the coefficient of thermal expansion and increases corrosion resistance in acidic environments. The addition of tungsten and nickel improves wear resistance and ductility, respectively [45,46]

The mechanical properties of this alloy vary depending on the production method such as casting, milling, or laser sintering but typically include a tensile strength of around 850 MPa, yield strength between 342–584 MPa, Vickers hardness ranging from 360–430 HV, and a modulus of elasticity between 190–230 GPa [47,48]. Although these features provide high strength and rigidity, the elongation at fracture tends to be limited, typically between 2–8%, reflecting lower ductility compared to other alloys such as nickel-chromium.

However, studies have highlighted some concerns associated with Co-Cr alloys. Metal ion release, particularly cobalt and chromium ions, can occur under adverse conditions such as exposure to acidic saliva, bacterial biofilms, or repeated mechanical loads. This release may lead to adverse tissue responses, including hypersensitivity, inflammation, and, in rare cases, cytotoxicity [49]. Moreover, the presence of eutectic particles in cast alloys may promote internal crack formation, reducing fracture resistance and increasing brittleness. Some studies also report thermal incompatibility between Co-Cr alloys and dental porcelains, which can lead to cracks at the metal-ceramic interface [47].

In dental practice, Co-Cr alloys are widely used for the fabrication of crown and bridge frameworks (Figure 2.10), removable partial denture structures, and implant-supported superstructures. Their high mechanical strength and durability allow them to withstand intense masticatory forces without significant deformation. In addition, modern additive manufacturing techniques, such as laser sintering, enable the production of components with higher density and lower porosity, optimizing material performance.

Some properties of Cobalt-Chromium alloys are presented in Table 2.1, allowing for a comparative view against other metals discussed in this thesis.



Figure 2.10 - Cobalt-Chromium alloy frameworks [50]

Advantages:

- Excellent corrosion resistance, even in humid and acidic oral environments;
- High mechanical strength and rigidity, ensuring durability and structural stability;
- Good biocompatibility, with minimal adverse interaction with oral tissues;
- More cost-effective compared to gold or titanium alloys.

Disadvantages:

- Limited ductility, resulting in lower deformation capacity before fracture;
- Manufacturing challenges, such as porosity and coarse grain sizes in casting techniques;
- Lower aesthetic compatibility due to metallic appearance;
- Potential release of metal ions, leading to adverse reactions in sensitive patients;
- Thermal incompatibility with dental porcelains, increasing the risk of failure in metal-ceramic restorations.

2.4.1.3. Titanium and its alloys

Titanium (Ti) is also one of the most popular metals in dentistry, particularly in dental implants, due to its combination of mechanical strength, low density (4.5 g/cm³), biocompatibility, and corrosion resistance [51,52]. This material was introduced in implantology by Brånemark in the 1960s [53] following the discovery of osseointegration, which revolutionized the use of metals in dentistry. Since then, both commercially pure titanium (cpTi) and its alloys, such as Ti-6% Aluminum 4% Vanadium (Ti-6Al-4V) have demonstrated high survival and long-term success rates.

According to the American Society for Testing and Materials (ASTM), cpTi is available in different grades (I–IV), which vary in their oxygen and iron content. These variations result in different physical and mechanical properties. Grade I titanium has the lowest mechanical strength, whereas Grade IV, due to its higher oxygen content, provides greater strength but reduced ductility [51].

The mechanical properties of the Ti-6Al-4V alloy stand out for their yield strength up to 795 MPa, tensile strength of 1000 MPa, and elongation at fracture of 14%. Its elastic modulus ranges from 85 to 115 GPa [49], which, although lower than that of other metal alloys, is still significantly higher than that of natural bone (10–30 GPa) [54]. This discrepancy increases the likelihood of stress shielding⁴. Additionally, titanium spontaneously forms a passive titanium dioxide (TiO₂) layer upon exposure to oxygen, which provides exceptional corrosion resistance—even in humid and acidic environments such as the oral cavity [45,51,55].

Despite its many advantages, some issues have been reported in the use of titanium and its alloys. Tribocorrosion, a phenomenon resulting from the interaction between mechanical wear and corrosion, can degrade the titanium oxide layer at the implant–crown interface. This can lead to the release of metal particles and ions, including aluminum and vanadium, which may trigger inflammatory reactions, hypersensitivity, or localized toxicity in some patients. Gingival discoloration may also occur in patients with thin gingival biotypes, compromising aesthetics. Under extreme conditions such as prolonged exposure to acidic saliva or excessive wear forces localized corrosion and associated mechanical failures may also occur [55].

⁴ Stress shielding is a phenomenon that occurs when an implant, due to its high elastic modulus compared to the surrounding bone, absorbs the majority of the applied mechanical loads, thereby reducing the load transmitted to the bone. This load deviation can lead to bone resorption around the implant, compromising its long-term stability.

Advantages:

- High corrosion resistance: The passive TiO_2 layer protects the material from chemical degradation in humid and acidic environments;
- Excellent biocompatibility: Titanium is widely tolerated by tissues and promotes osseointegration;
- Low density and high strength: This combination makes implants lightweight yet durable;
- Long-term durability: Capable of withstanding cyclic loading over time, with high clinical success rates.

Disadvantages:

- Tribocorrosion and ion release: The interaction between wear and corrosion may degrade the passive layer and lead to ion release, potentially causing adverse reactions;
- Aesthetic limitations: Metallic color may be visible in patients with thin gingival tissues;
- Challenging manufacturing: Techniques such as vacuum casting or precision machining require advanced technology, increasing production costs;
- Localized corrosion: Under extreme conditions, titanium may exhibit localized corrosion, compromising structural integrity.

Table 2.1 compares the mechanical properties of the previously discussed metallic materials commonly used in dental restorations. The table includes properties such as density, tensile strength, elastic modulus, Vickers hardness, and ductility.

Table 2.1 - Comparative mechanical properties of metals discussed in this thesis: Stainless steel 316L, Co-Cr alloy, Titanium and Ti-6Al-4V [48,56].

Property	Stainless steel 316L	Co-Cr alloy	Titanium	Ti-6Al-4V
Density (g/cm³)	7,95	8.9	4.5	4.5
Tensile Strength (MPa)	515	850	520	1000
Elastic modulus (GPa)	193	190-230	110	85-115
Hardness (Vickers)	155	360-430	200	320-400
Ductility (%)	38-49	2-8	20	14

2.4.2 Polymers

Polymer-based materials play a significant role in dentistry, being widely used in a range of applications due to their surface characteristics, mechanical and biological properties, ease of processing, and cost-effectiveness [57,58].

Among the most commonly used polymers are polymethyl methacrylate (PMMA), polyurethane (PU), polyethylene (PE), polycarbonate (PC), polyether ether ketone (PEEK), polyethylene glycol (PEG), polydimethylsiloxane (PDMS), polylactic acid (PLA), poly(ϵ -caprolactone) (PCL), acrylonitrile butadiene styrene (ABS), and polypropylene (PP) [59]. Their applications range from direct and indirect restorations to implantology, with synthetic PEEK recently being proposed as a material for dental implants [60].

In line with the focus of this thesis, only polymeric materials used for indirect restorations via CAD/CAM milling technology will be addressed. Among these, PMMA stands out due to its excellent machinability and low cost, while PEEK is noted for its high mechanical and chemical resistance.

2.4.2.1 Poly(methyl) methacrylate (PMMA)

Poly(methyl methacrylate) (PMMA) is a thermoplastic polymer primarily used in temporary indirect restorations, such as provisional crowns and bridges, due to its combination of mechanical, esthetic, and biological properties, as well as its chemical stability in the oral environment [61]. In recent years, the use of PMMA has been boosted by its integration with CAD/CAM technology, which allows to produce provisional restorations from pre-polymerized blocks with greater precision and, consequently, better marginal adaptation compared to traditional techniques [62,63].

Despite its advantages, PMMA presents some limitations that restrict its use in more demanding clinical situations. One of its main drawbacks is brittleness, as the material has lower impact resistance when compared to ceramics or reinforced composites. In terms of flexural strength, typical values range between 65 and 110 MPa [63], with an elastic modulus in the range of 2 to 3 GPa—characteristics that limit its use in areas subjected to high masticatory loads. Another significant limitation is wear with prolonged use, PMMA can undergo substantial abrasion, reducing marginal precision and compromising long-term performance [64]. Water absorption is also a concern, as it may lead to dimensional changes and degradation of mechanical properties over time.

To overcome these issues, recent advances in PMMA formulations have focused on the incorporation of nanoparticles such as zirconium oxide (ZrO_2) and silica, which significantly enhance the material's flexural strength, hardness, and dimensional stability [65].

Advantages:

- Optical properties: High translucency and a wide range of available shades, enabling a natural appearance similar to dental enamel;
- Cost-effective: PMMA is more affordable compared to ceramics or high-performance polymers such as PEEK;
- Ease of production: Easy to handle in both traditional techniques (direct and indirect) and CAD/CAM milling, making it versatile for various clinical applications;
- Biocompatibility: Low-density material, well tolerated by oral tissues.

Disadvantages:

- Limited mechanical strength: Lower flexural strength compared to reinforced composites or ceramics, making it more prone to fracture under high masticatory forces;
- Higher wear susceptibility: Reduced impact resistance and tendency to wear over time, limiting its long-term performance in provisional restorations;
- Water absorption: Can lead to dimensional instability and deterioration of mechanical and aesthetic properties over time;
- Monomer release risk: If inadequately polymerized, may release residual monomers—such as methyl methacrylate (MMA)—which can irritate oral tissues.

2.4.2.2 Polyetheretherketone (PEEK)

Polyetheretherketone (PEEK) is a high-performance thermoplastic polymer that is widely used as a biomaterial in various healthcare fields, including dentistry and implantology [66].

Its use in indirect restorations has grown significantly due to its unique combination of physical, chemical, and esthetic properties, positioning it as an alternative to some metals and ceramics [67]. CAD/CAM technology enables PEEK to be precisely milled, allowing for the fabrication of customized, well-adapted restorations, which is essential for indirect procedures such as crowns, bridges, and inlays.

For implant use, one of the main features of PEEK is its elastic modulus, which—when reinforced with TiO₂, carbon fibers, or glass fibers [52,67] reaches values between 12–18 GPa. This is much closer to that of natural bone, significantly reducing the likelihood of stress shielding commonly observed with more rigid materials like titanium, and allowing for a more uniform distribution of stress to the surrounding bone.

PEEK also exhibits flexural strength between 100 and 120 MPa [66] and high resistance to chemical degradation, properties that are crucial for indirect restorations where materials must withstand masticatory forces without compromising structural integrity.

It is also biocompatible and bioinert, significantly reducing the risk of adverse reactions such as allergies and inflammation. Its radiolucency is another valuable characteristic, allowing for proper diagnostic imaging via X-rays and CBCT scans. However, PEEK has a low surface energy, which can compromise adhesion to luting agents, requiring surface treatments such as sandblasting, acid etching, or plasma modification [60]. Moreover, long-term clinical evidence

supporting the use of PEEK in fixed and removable restorations remains limited [68]. Despite its favorable mechanical and biological properties, PEEK's low translucency and grayish color prevent its use as a monolithic esthetic restorative material. To improve its appearance, esthetic coatings are often necessary [68].

In the context of indirect restorations, PEEK is used primarily as a substructure material for crowns and bridges due to its low density, neutral color, and excellent wear resistance.

Advantages:

- High biocompatibility: Non-allergenic and suitable for patients with metal allergies;
- Low density: Provides greater comfort compared to metallic materials;
- Shock absorption: Reduces stress concentration during mastication;
- CAD/CAM compatibility: Allows for precise and personalized restorations through milled blocks.

Disadvantages:

- Bioinert surface: Limited biological activity requires surface modification to improve bonding and integration;
- High cost: More expensive than materials such as PMMA;
- Low adhesion potential: Requires surface pretreatment for effective bonding with dental cements;
- Limited esthetics: Despite its neutral tone, PEEK has low translucency compared to ceramics and often requires additional veneering.

Table 2.2 compares the mechanical properties of the polymeric materials previously discussed, commonly used in CAD/CAM-milled dental restorations, including density, tensile strength, elastic modulus, Vickers hardness, and ductility.

Table 2.2 - Comparative table of the properties of the polymeric materials discussed, PMMA and PEEK.
[52,60,63,66,67]

Property	PMMA	PEEK
Density (g/cm ³)	1.2	1.3
Tensile Strength (MPa)	50-70	90-100
Elastic modulus (GPa)	2-3	3-4
Hardness (Vickers)	20-25	20-24
Ductility (%)	5-10	10-20

Despite their numerous advantages, polymers have significant limitations that render them unsuitable for dental veneer applications. The opacity of polymers—even advanced ones such as PEEK—limits their ability to replicate the translucency of natural enamel, resulting in an artificial appearance that is unacceptable in high-demand esthetic restorations. In addition, wear resistance in materials like PMMA is insufficient to endure the oral environment, especially in anterior veneers where cyclic loading and abrasion are frequent [69]. Dimensional stability of polymers in intraoral settings also remains a challenge. Water absorption, particularly in materials like PMMA, can lead to deformation and compromised bonding between the dental substrate and the restorative material. This issue is further exacerbated by the fact that polymers have higher coefficients of thermal expansion compared to enamel, increasing the risk of marginal microleakage and adhesive failure.

In summary, both metals and polymers fail to meet the specific demands of dental veneers: metals due to their lack of esthetics, and polymers due to their limited mechanical performance and dimensional stability. These requirements are more effectively addressed by advanced composites and ceramics, which combine excellent esthetics with superior mechanical behavior and will be discussed in detail in the following sections.

2.4.3 Composites

As previously mentioned, dental veneers can be classified as direct (fabricated chairside by the dentist) or indirect (requiring an external laboratory service). Direct veneers are typically made using resin-based composites, while indirect veneers are produced using heat-cured composites or reinforced composite blocks

2.4.3.1 Resin-based composites for direct veneers

Direct composite veneers are applied directly onto the tooth and polymerized in situ. In this technique, the dentist manually sculpts the tooth by layering composite resin, and as such, the quality of the final outcome heavily depends on the clinician's skill and experience [70,71]. Compared to traditional ceramic veneers, this type of restoration is generally faster and more cost-effective [72].

Resin composites are composed of a polymeric matrix, typically a dimethacrylate (e.g., Bis-GMA, UDMA, TEGMA), and reinforcing fillers, which are usually radiopaque glass or other inorganic particles. Additionally, they contain a silane coupling agent that promotes adhesion between the fillers and the resin matrix, and chemical components (photoinitiators) that initiate or regulate the polymerization reaction [73]. Alluhaidan et al. [74] proposed a schematic representation of the components of resin composites (Figure 2.11).

Over time, the evolution of resin composites has focused primarily on optimizing the quantity and particle size of the fillers to improve polishability, wear resistance, and reduce polymerization shrinkage [73,75,76].

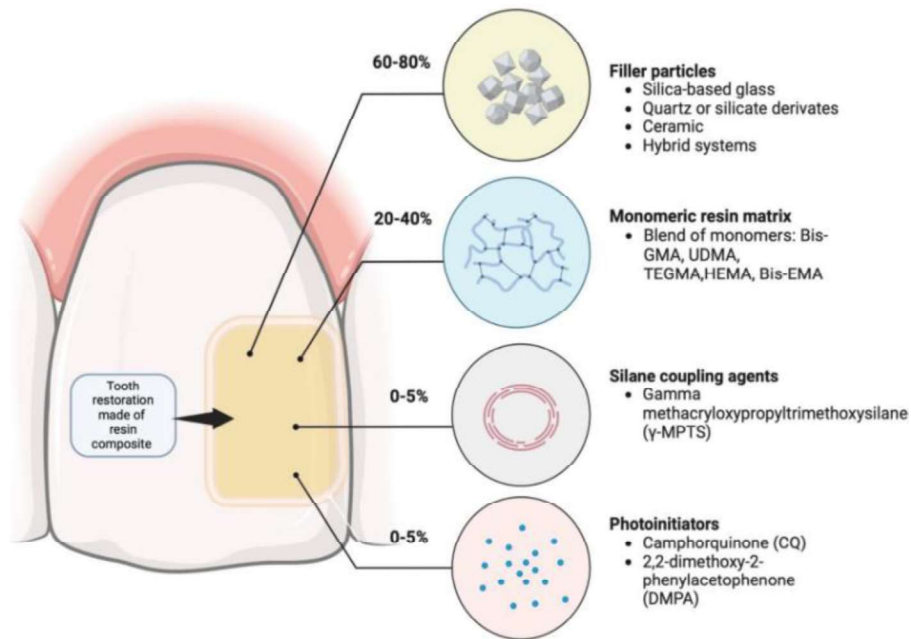


Figure 2.11 - Schematic illustration showing the main components that form the basic structure of dental resin composites [74].

Initially, macrofilled composites used particles larger than 1 μm , reaching up to 50 μm . These composites were highly resistant but difficult to polish and did not maintain a smooth surface over time [72,73].

To enhance long-term esthetics, manufacturers developed microfilled composites, a term meant to highlight the smaller filler sizes. However, from a technical standpoint, these were nanocomposites, as the spherical amorphous silica particles used had an average diameter of around 40 nm [73]. Today, materials within this range are categorized under nanotechnology, and these early microfills would more accurately be classified as nanofills. Their main limitation was a low filler content, which affected mechanical performance an issue addressed with the development of small-particle hybrid composites. Initially, midfilled composites combined particles slightly larger than 1 μm with smaller 40 nm pyrogenic⁵ silica particles. As grinding and milling techniques improved, minifill composites emerged, containing fillers between 0.4 and 1.0 μm —eventually termed microhybrid composites. These became widely adopted due to their balance of mechanical strength and polishability. Further refinement led to the development of nanofill

⁵ Pyrogenic silica, is a type of silica (silicon dioxide – SiO_2) produced through the combustion of silicon compounds in a high-temperature flame. During this process, tiny silica particles are formed in suspension in the air and subsequently aggregated, resulting in an ultrafine powder (5–50 nm) with a high specific surface area.

composites (containing only nanoscale particles) and nanohybrid composites, which combine nanoparticles with prepolymerized filler clusters.

The choice of composite for direct veneers depends on the balance between mechanical and esthetic properties to ensure suitable clinical performance.

The mechanical properties of these materials are directly influenced by the filler content within the resin matrix: higher filler loads result in greater strength, rigidity, and toughness. The elastic modulus of resin composites ranges from 6 to 16 GPa, offering more flexibility than ceramic materials. Flexural strength values typically range between 70 and 160 MPa, and fracture toughness between 0.85 and 1.75 MPa.m^{1/2}, giving them mechanical properties comparable to dental metal alloys and feldspathic porcelains, though still inferior to high-strength ceramics [73,77].

The longevity of composite restorations remains a clinical challenge due to chemical and mechanical degradation in the oral environment. Such degradation may result from processes like hydrolysis of the polymer matrix, surface abrasion, and stresses induced by polymerization shrinkage. Several studies [74,78] have reported that volumetric shrinkage during curing can induce internal stress, leading to marginal gaps that allow bacterial infiltration and the development of secondary caries [79]. Another critical issue is the biodegradation of the resin matrix, caused by continuous exposure to pH and temperature variations, and the presence of salivary enzymes, all of which accelerate the deterioration of the tooth–restoration interface. Furthermore, composites are prone to softening and expansion due to water absorption, which can weaken their mechanical stability [74].

To address these issues, bioactive composites have emerged as a promising solution. These materials interact with the oral environment to promote remineralization and reduce bacterial colonization [74]. Another recent innovation includes resin composites with microencapsulated self-healing monomers, which can be activated by mechanical stimuli to seal microcracks before they propagate [74]. Additionally, new photoactivation protocols involving incremental curing and high-intensity LED units have been explored to improve monomer conversion rates, thereby reducing internal stress and enhancing wear resistance [76]. Preheating composite resins prior to application has also been suggested to increase flowability and minimize polymerization shrinkage [80].

With advances in nanotechnology and materials engineering, future composites are expected to become stronger, more esthetic, and less prone to degradation. New strategies, such as hybrid

composites containing ceramic particles, are being investigated to enhance wear resistance and long-term dimensional stability [76]. Furthermore, the development of smart materials, capable of responding to mechanical or chemical stimuli, may revolutionize restorative dentistry by enabling more predictable and durable restorations.

2.4.3.2 Thermally Polymerized Composites (Laboratory-Fabricated)

Thermally polymerized composites, also known as laboratory composites, are compositionally similar to conventional light-cured resin composites [81] and are used in the fabrication of indirect restorations such as veneers, inlays/onlays, and partial crowns. These composites differ from direct light-cured resins in that they undergo an additional thermal curing process, which significantly enhances their mechanical properties and long-term stability [74]. Thermal polymerization allows for a higher degree of monomer conversion, reducing the number of residual monomers and minimizing the adverse effects of chemical degradation in the oral environment [82]. The fabrication process typically involves two main phases: An initial shaping and light-curing phase, which provides preliminary hardening; Followed by thermal curing, in which the restoration is subjected to temperatures typically ranging from 120°C to 170°C, under controlled pressure and time conditions. This secondary thermal step significantly improves monomer conversion, reduces polymerization shrinkage, and increases the mechanical strength of the material.

As a result, the mechanical properties of thermally polymerized composites surpass those of direct resin composites, positioning them as a viable alternative to dental ceramics in specific clinical applications. In a study conducted by Takeshige et al. [83] the application of heat following initial polymerization was shown to markedly improve fracture toughness, flexural strength, and elastic modulus, making the composites more rigid and wear-resistant. Additionally, thermal curing has been reported to reduce both the thermal expansion coefficient and hygroscopic expansion, resulting in better dimensional stability and a lower risk of marginal failure or microleakage. However, water absorption was not significantly altered by thermal curing, suggesting that other factors—such as chemical formulation and the ratio of inorganic filler—also play important roles in this property.

When compared to ceramics, these composites exhibit notable differences in mechanical behavior and processing. Ceramics, with their high elastic modulus, are more rigid but also more brittle, whereas composites offer greater flexibility and impact resistance due to their lower

stiffness. Furthermore, ceramics do not undergo volumetric shrinkage as they are sintered, while composites—even when thermally polymerized—still experience some shrinkage. However, composites allow for easier intraoral adjustments and repairs, whereas ceramic restorations often require full replacement in the event of failure.

Recent advancements in restorative dentistry have led to the introduction of reinforced composites for CAD/CAM, offering a new level of precision and strength due to their industrial fabrication and computer-aided machining, which will be discussed in the next section.

2.4.3.3 Reinforced CAD/CAM composites

Reinforced CAD/CAM composites have gained prominence in restorative dentistry as an innovative alternative to traditional materials. These composites differ from conventional resin composites primarily in their manufacturing process and enhanced mechanical properties, which result from thermal and pressure polymerization performed at an industrial scale. Their use in CAD/CAM systems enables the fabrication of highly precise and customized indirect restorations, including single crowns, inlays/onlays, and veneers.

These materials combine a highly cross-linked polymeric matrix with a significant fraction of inorganic fillers, such as silica and zirconia. The inorganic content varies significantly among different products, reaching ceramic loadings between 80–86%, which approximates the composition of traditional ceramics, or featuring a higher resin fraction (25–40%), offering greater flexibility and milling ease [84]. This compositional variability allows clinicians to select the material based on specific clinical requirements, balancing strength, flexibility, and machinability.

Compared to conventional resin composites, these reinforced composites exhibit a more efficient polymerization reaction due to the combined thermal and pressure processing used in the production of blocks. This results in lower polymerization shrinkage and improved wear resistance and fatigue behavior [85]. The high inorganic content also contributes to enhanced polishability and abrasion resistance, both of which are essential for the longevity of restorations [86].

In terms of mechanical performance, studies report that reinforced CAD/CAM composites exhibit flexural strength ranging from 120 to 274 MPa, with an elastic modulus between 8 and 23 GPa, depending on composition and processing. These values are significantly higher than those of direct composites but still lower than those of ceramics [84–86].

The first CAD/CAM reinforced composite block developed was Paradigm MZ100 (3M ESPE), composed of 85% ultrafine silica-zirconia ceramic particles. This was followed by Lava Ultimate

(3M ESPE) and Cerasmart (GC), which incorporate zirconia and silica nanoparticles to enhance flexural strength and provide an elastic modulus similar to dentin [86].

The thermal and high-pressure polymerization process, sometimes using Hot Isostatic Pressing (HIP), improves the internal structure of the material by reducing porosity and increasing the polymer matrix density. This contributes to predictable and durable mechanical behavior [84]. Furthermore, these materials exhibit low water absorption and higher Vickers hardness, resulting in superior clinical longevity compared to direct and thermally cured composites [86]. In table 2.3 are presented some properties of composites materials discussed above for comparison.

From an aesthetic perspective, these composites are formulated to offer a wide range of shades and translucencies, promoting better visual integration with natural dentition. The presence of ceramic fillers improves gloss and color stability, reducing the risk of staining and discoloration over time.

Some CAD/CAM composites, such as Brilliant Crios (COLTENE) and Cerasmart (GC), have a refractive index close to that of dental tissues, enabling natural appearance and balanced translucency, which makes them suitable for veneers and anterior restorations where visual impact is critical.

A major advantage of CAD/CAM composites over ceramic materials lies in their ease of milling and intraoral repairability. While ceramics require stricter adhesive protocols and are more prone to chipping during milling, composites can be easily adjusted, repaired, and polished chairside, eliminating the need for additional procedures such as sintering [85]. These blocks can be milled using high-precision milling machines, allowing for the rapid and accurate fabrication of veneers and crowns.

Despite their numerous advantages in the context of indirect restorations, the mechanical strength of reinforced CAD/CAM composites still falls short of advanced ceramics, such as lithium disilicate and zirconia.

The continued evolution of dental biomaterials shows that ceramics remain the gold standard when prioritizing fracture resistance and long-term chemical stability. Therefore, reinforced CAD/CAM composites can be considered an intermediate solution between traditional resin composites and ceramics.

Table 2.3 – Comparison of some properties of composites [84-86].

Property		Direct Composites	Thermally Cured Composites	CAD/CAM Reinforced Composites
Density (g/cm³)		1.2	1.3	-
Flexural Strength (MPa)		70-160	120-180	120-274
Elastic modulus (GPa)		6-16	8-18	8-23
Hardness (Vickers)		25-50	40-60	60-120
Fracture Toughness (MPa·m^{1/2})		0.8-1.75	1.5-2.5	2.0-3.5
Estimated Longevity (years)	Clinical	3-7	5-10	7-12

2.4.4 Ceramics

Ceramic materials play a fundamental role in oral rehabilitation due to their biocompatibility, chemical stability, and superior aesthetic properties [26,33,87–89]. Over the past decades, the evolution of dental ceramics has enabled the development of all-ceramic restorations, eliminating the need for supporting frameworks made of metal or other materials. This transition is grounded in the enhancement of the mechanical properties of modern ceramics, allowing their application in regions subject to high masticatory loads [6].

All-ceramic restorations can be categorized into traditional dental ceramics, which are primarily silica-based; glass ceramics, which represent an evolution of traditional ceramics; and high-strength core ceramics, developed to overcome the mechanical limitations of traditional and

glass ceramics. Figure 2.12 illustrates the classification diagram of the ceramic types used in dental restorations.

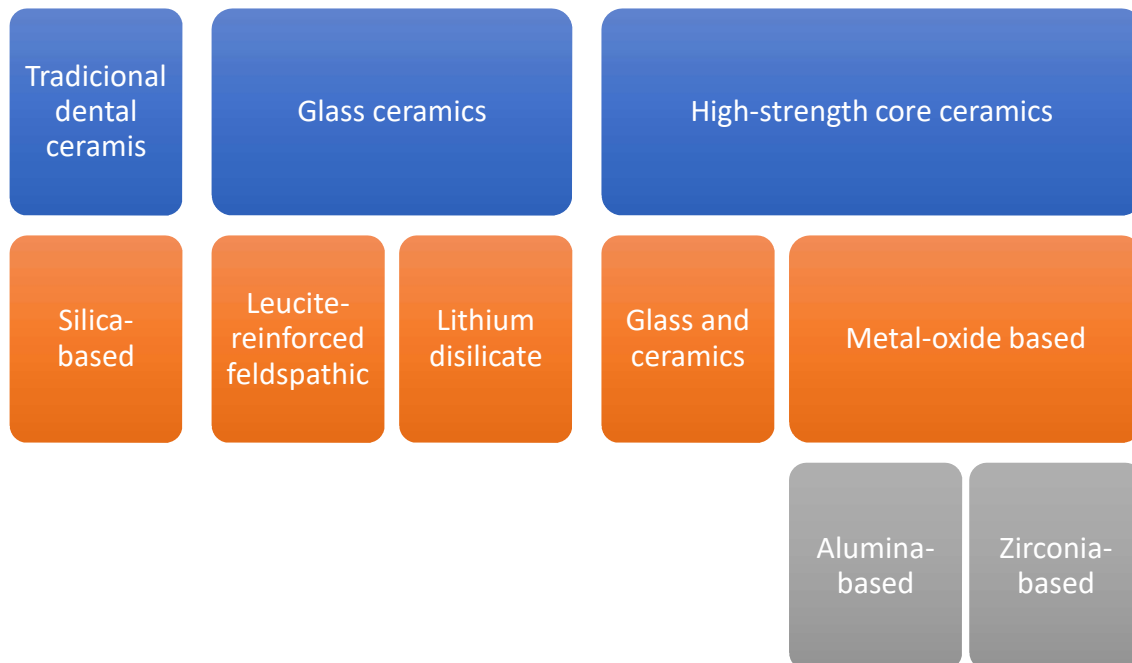


Figure 2.12 - Classification diagram of ceramic materials used in dental restorations.

2.4.4.1 Traditional dental ceramics

Silica-based ceramics were the first materials used in dentistry for the fabrication of porcelain prostheses. Also known as feldspathic ceramics, they belong to the group of traditional ceramics widely employed in all-ceramic restorations. These ceramics undergo a vitrification process typically at temperatures ranging from 900 °C to 1100 °C, in which the silica glass matrix surrounds numerous dispersed crystalline phases, resulting in a structure characterized by high translucency and excellent aesthetics [6]. The composition of these ceramics consists predominantly of 70–75% potassium feldspar ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$), 15–20% quartz (SiO_2) as the crystalline phase, and the remainder kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) as a binder. The addition of small amounts of Al_2O_3 improves their mechanical strength, while pigments are included to control opacity and create various tooth shades [87]. This ability to mimic the natural shades of teeth makes them particularly well-suited for veneers [33]. Despite their superior aesthetic properties, feldspathic ceramics exhibit low mechanical strength, with flexural strength ranging from 60 to 100 MPa. Due to their inherent fragility, this type of ceramic is often used as a veneering layer over metallic substructures (metal-ceramics or PFM) or over stronger ceramic cores such as lithium disilicate or zirconia [6,33]. The fabrication process involves mixing fine glass particles (approximately 25 μm in diameter) with

polymeric binders in an aqueous medium to form a paste that can be applied directly onto a metallic or ceramic substrate. During the sintering process, the porcelain layer is gradually heated to evaporate the binder, enabling particle coalescence and structural densification. Controlled cooling is essential to avoid cracking and residual stress, thereby ensuring dimensional stability [11]. Despite their aesthetic advantages, bilayered structures such as these present disadvantages due to the mismatch in mechanical properties between layers. Chipping of the porcelain layer and delamination are common failure modes in this type of restoration, primarily due to weak bonding between the core and the veneering porcelain. Furthermore, differences in the coefficients of thermal expansion between layers can induce residual stresses post-sintering, compromising the longevity of the restoration [6]. To minimize such issues, an alternative approach is the use of monolithic ceramics, such as glass ceramics and oxide ceramics, which are fabricated directly through CAD/CAM technology or via press technology. These techniques significantly reduce the risk of fractures associated with layer adhesion failures [87].

2.4.4.2 Glass ceramics

Glass ceramics represent an evolution of traditional ceramics. These materials feature a glassy matrix reinforced by dispersed crystals, which enhance their mechanical properties. The presence of these crystalline phases allows for an optimal balance between structural strength and optical characteristics, making glass ceramics a popular choice for minimally invasive aesthetic restorations.

Within this category, two major groups stand out: leucite-reinforced feldspathic ceramics and lithium disilicate ceramics, each offering distinct properties suited to different clinical applications.

2.4.4.2.1 Leucite reinforced feldspathic ceramics

Leucite-reinforced feldspathic ceramics are enhanced compositions of traditional silica-based dental ceramics. The addition of leucite crystals (KAlSi_2O_6) dispersed within the glassy matrix increases flexural strength, typically ranging from 120 to 180 MPa—significantly higher than that of pure feldspathic ceramics. This improvement is primarily due to leucite's ability to dissipate internal stresses and hinder crack propagation [11].

The microstructure of these ceramics typically consists of feldspar (60–65%), leucite (20–30%), and quartz (10–15%). During processing, these ceramics undergo a controlled heat cycle that promotes the nucleation and growth of leucite crystals, resulting in a more homogeneous

material. Sintering is carried out at temperatures around 900–1000 °C, which facilitates chemical bonding between the matrix and the dispersed crystals [87].

Despite the improved mechanical performance, leucite-reinforced ceramics still present some brittleness and are therefore primarily indicated for anterior applications such as veneers, inlays, and onlays. Their high translucency and ability to replicate the natural nuances of enamel make them particularly suitable for aesthetic restorations in the smile zone, where visual outcomes are of critical importance to the patient [33].

2.4.4.2.2 Lithium disilicate

Lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) is currently one of the most widely used ceramic materials in restorative dentistry due to its excellent balance between mechanical strength and optical properties. With a flexural strength ranging from 350 to 450 MPa, this material offers a reliable solution for cases requiring enhanced structural performance without compromising esthetics [6].

Its composition consists of 60–70% lithium disilicate crystals embedded in a glassy matrix, providing an excellent degree of translucency. The fabrication process can be performed via hot-pressing technology or through milling using CAD/CAM systems, making this material highly versatile for both dental laboratories and clinical settings [11].

One of lithium disilicate's key advantages is its ability to form a chemical bond with dental enamel, enabling minimally invasive techniques for monolithic restorations. Furthermore, its compatibility with resin cements enhances long-term stability by reducing the risks of delamination or debonding. Its thermal expansion coefficient (CTE) is well-matched with dental tissues, helping to prevent residual stresses after cementation [87].

The clinical versatility of lithium disilicate allows its use in single-unit crowns, veneers, inlays, onlays, and short-span fixed dental prostheses, making it suitable for both anterior and posterior applications. However, for extensive restorations or areas under heavy occlusal load, this material is generally not recommended [6].

2.4.4.3 High strength core ceramics

High-strength core ceramics were first introduced in the mid-1960s by McLean and Hughes, who developed a core material based on feldspathic porcelain reinforced with alumina, giving rise to alumina-reinforced porcelain. Later, around the 1980s, glass-infiltrated high-strength ceramics were developed (e.g., In-Ceram, Vita). By the 1990s, fully dense polycrystalline alumina cores (e.g.,

Techceram, ProCera AllCeram, Nobel Biocare) were introduced. Eventually, yttria-stabilized zirconia (YSZ) became widely adopted for the production of crown cores and bridge frameworks [90].

Alumina (aluminum oxide, Al_2O_3) is a chemically inert ceramic material with clinical applications due to its biocompatibility, high density, mechanical strength, and excellent wear resistance. However, its use in veneers is limited, primarily due to its relative brittleness and inherent opacity. Initially, high-purity alumina ceramics (>99.5%) were used in the fabrication of crown and fixed prosthetic frameworks, benefiting from their corrosion resistance and chemical stability. Nevertheless, pure polycrystalline alumina lacks sufficient translucency for aesthetic applications. Moreover, while it offers higher fracture resistance compared to conventional feldspathic ceramics, this strength is insufficient for ultra-thin structures, increasing the risk of fracture or debonding under masticatory loads.

To overcome alumina's inherent brittleness and broaden its clinical applicability, zirconia-toughened alumina (ZTA) composites were developed. In this material, tetragonal zirconia particles are dispersed within an alumina matrix, enabling a transformation toughening mechanism. This occurs when zirconia particles undergo a tetragonal-to-monoclinic phase transformation ($t \rightarrow m$) under stress, resulting in a 3–5% volume expansion that generates compressive stresses within the surrounding alumina matrix. This hinders crack propagation and significantly improves fracture toughness.

However, the applicability of ZTA in veneers remains limited. Despite enhanced fracture resistance, translucency remains a critical limitation. Studies have shown that, even with modifications in grain size and composition, ZTA fails to achieve the optical properties required for high-aesthetic outcomes. Additionally, the thin design required for veneers (typically 0.3 to 0.7 mm) compromises the effectiveness of the toughening mechanism, as thinner sections do not generate the same compressive stress fields necessary to prevent crack propagation unlike in thicker core structures.

Another important factor in the selection of materials for veneers is bonding to resin cement. While glass ceramics like lithium disilicate allow effective hydrofluoric acid (HF) etching for strong adhesion, ZTA and alumina, being polycrystalline ceramics, are not etchable with HF acid. They require the use of specific adhesion promoters, such as silane or phosphate monomer-based

primers (e.g., MDP), which can compromise long-term retention, especially in minimally invasive preparations.

Although ZTA has demonstrated greater mechanical stability and resistance to low-temperature degradation (LTD) than conventional zirconia (3Y-TZP), its long-term clinical performance remains uncertain. Recent studies, such as that by Jalkh et al. [91], have shown that ZTA maintains its mechanical strength after accelerated aging, in contrast to 3Y-TZP, which exhibits a significant increase in monoclinic phase content leading to a loss in strength. However, this degradation mechanism may be less clinically relevant for veneers, which are not subjected to the same mechanical and environmental stresses as posterior crowns or bridges.

2.4.5 Zirconia based ceramics

Zirconia (ZrO_2) has emerged as one of the most promising ceramic materials in dentistry due to its excellent mechanical strength, high biocompatibility, and evolving optical properties [92]. Since its introduction in restorative dentistry, zirconia has been extensively used in the fabrication of crowns, bridges, and dental veneers. Its use is largely justified by its ability to replace metals in core frameworks, offering both superior strength and a more natural appearance. Recent advancements in zirconia formulations have significantly improved translucency, thus broadening its indications for ultra-thin dental veneers. However, these improvements have introduced new challenges, particularly in optimizing the bonding of zirconia to the tooth substrate [93], as well as ensuring the mechanical stability of such restorations at reduced thicknesses.

Pure zirconia (ZrO_2) is a polymorphic material, meaning it can assume different crystalline phases at atmospheric pressure depending on the temperature: monoclinic (below 1170°C), tetragonal (between 1170°C and 2370°C), and cubic (above 2370°C). During cooling, the transition from the tetragonal (t) to the monoclinic (m) phase leads to a significant volumetric increase ($\sim 3\text{--}5\%$), which can compromise the integrity of the material or even lead to fracture [94]. According to the literature [95], this transformation is irreversible and typically initiates around 950°C during the cooling cycle. The monoclinic phase is undesirable in dental applications due to its associated volumetric expansion, which may cause cracks in the restoration, resulting in premature failure. Therefore, maintaining zirconia in the tetragonal or cubic phase at room temperature is essential to ensure structural stability.

To stabilize the tetragonal phase at room temperature and achieve optimal mechanical performance, stabilizing oxides such as Y_2O_3 (yttria), CeO_2 , MgO , or CaO are incorporated. This

stabilization enables a mechanism known as transformation toughening [96,97], which enhances fracture resistance. The type and amount of stabilizer directly influence the mechanical and optical behavior of zirconia.

Even though 3 mol% yttria-stabilized tetragonal zirconia (3Y-TZP) is resistant to spontaneous transformation to the monoclinic phase at ambient conditions, phase transformation can still occur under localized stress or in humid environments—especially at temperatures between 150°C and 400°C [95,98]. This phenomenon, known as low-temperature degradation (LTD) or aging, is considered a key limitation of the material.

Degradation typically starts at the grain surface and progresses inward as water molecules infiltrate the zirconia grains and occupy oxygen vacancies [98]. Studies have shown that LTD may either enhance or impair the mechanical performance of Y-TZP. In some instances, LTD can trigger the transformation toughening effect and improve mechanical behavior. However, excessive aging can lead to surface roughening, grain disintegration, and microcrack formation, ultimately compromising structural integrity [99].

Based on the type and amount of stabilizer, zirconia ceramics can be classified into four major microstructural groups [100]:

- FSZ (Fully Stabilized Zirconia) – Predominantly cubic structure.
- ZTC (Zirconia Toughened Composite) – Tetragonal zirconia grains dispersed in a ceramic matrix (e.g., alumina).
- PSZ (Partially Stabilized Zirconia) – Comprises mainly cubic zirconia with some monoclinic and tetragonal precipitates.
- TZP (Tetragonal Zirconia Polycrystal) – Primarily tetragonal metastable microstructure.

The development of zirconia has evolved through four generations aimed at optimizing both mechanical and aesthetic properties [6]. The first generation consisted of zirconia stabilized with 3 mol% yttria (Y_2O_3) and contained approximately 0.25% alumina, which compromised its esthetic performance and necessitated veneering with feldspathic porcelains to achieve clinically acceptable outcomes. The second generation aimed to enhance translucency by reducing the alumina content and optimizing the sintering parameters; however, aesthetic limitations, particularly in terms of light transmission, persisted. In the third generation, the yttria content was increased to 4–5 mol%, resulting in a higher proportion of the cubic phase, which improved translucency and light diffusion. However, this improvement in optical properties was accompanied by a reduction in mechanical

strength, due to the lower fracture toughness of the cubic phase compared to the tetragonal phase.. The most recent development, referred to as the fourth generation, introduced a multilayer design that gradually transitions between layers with varying cubic phase content to mimic the natural gradation of color and translucency in human teeth. Although this innovation has significantly advanced aesthetic outcomes, a key challenge remains: understanding how the interlayer interfaces behave under intraoral conditions, particularly given the reduced fracture toughness associated with the cubic phase compared to the tetragonal counterpart [6].

Among these, 3Y-TZP remains the most widely used zirconia type in dentistry, due to its high flexural strength (approximately twice that of alumina), excellent biocompatibility, chemical inertness, and resistance to wear and corrosion. Moreover, its fracture toughness (6–10 MPa.m^{1/2}) is superior to that of other ceramics [92,101,102]. Table 2.4 summarizes key mechanical and thermal properties of 3Y-TZP.

Table 2.4 - Mechanical and thermal properties of 3Y-TZP zirconia [103,104].

MECHANICAL PROPERTIES	
Density (g/cm³)	6.08
Elastic modulus (GPa)	153-210
Hardness (HV30)*	1350
Fracture toughness (MPa.m^{1/2})*	6-10
Flexural strength (MPa)*	900-1200
Compressive strength (MPa)*	2000
THERMAL PROPERTIES	
Thermal conductivity [W/(m*K)]	2.09
Coefficient of thermal expansion [K⁻¹]	10 ⁻⁶
Melting temperature (°C)	2715

One of the goals of this research is to enhance the optical properties of second-generation zirconia to match the aesthetic quality of third- and fourth-generation materials.

The color and appearance of monolithic zirconia restorations are influenced by both intrinsic (material-related) and extrinsic (environment-related) factors [105]. Parameters such as cement layer thickness, restoration thickness, and LTD-induced surface changes affect light transmission. Microstructural features, including grain boundaries and porosity, serve as light-scattering centers, determining whether the material appears translucent or opaque [106]. Thus, translucency and opacity depend on how the incident light is reflected, scattered, refracted, transmitted, or absorbed [107,108].

2.5 Adhesion

The advancement of adhesive dentistry has significantly improved the biomechanical bond between the tooth and the restoration, allowing for more efficient restorative procedures. This evolution has promoted a more conservative and minimally invasive approach, aiming to preserve as much of the natural tooth structure as possible.

The success of zirconia-based restorations also depends on the proper adhesion between the dental substrate (enamel or dentin) and the adhesive cement, as well as the bond between the cement and the zirconia framework.

According to the literature [109–114] zirconia is considered not only a promising material but also one of the most challenging to bond. A previous study reported that many clinical failures—ranging between 2.3% and 8%—are attributed to the cementation process [115]. The integrity of the bond between the adhesive cement and the ceramic surface plays a crucial role in the longevity of the restoration, highlighting the need to develop reliable surface treatment methods to enhance this critical area [116]. Available surface treatments can be categorized into mechanical and chemical methods [115].

Research has shown that mechanical pre-treatments—such as alumina particle air abrasion, tribochemical silica coating, laser irradiation, chemical etching, and ceramic coatings—can improve the bond strength between zirconia and adhesive cements. This is mainly due to the increased surface roughness and mechanical interlocking promoted by these methods [117]. Clinically acceptable surface roughness values (Ra) for monolithic zirconia crowns are reported to be around 200 nm, which is considered optimal to minimize bacterial plaque accumulation [118]. However, internal restoration surfaces with this level of roughness tend to show suboptimal adhesive performance [119].

Some studies [120,121] have found that roughness values around 0.41 μm can be achieved through tribochemical silica-coated alumina air abrasion, yielding satisfactory bond strength results. In contrast, surfaces after CAD/CAM machining and sintering may exhibit roughness values around 1.8 μm [122].

Chemical pre-treatment using functional monomers—typically applied as primers to the zirconia surface—also contributes to long-term chemical bonding [123,124].

Studies indicate that combining mechanical and chemical surface treatments yields superior bond strength compared to using either method alone [117], [118].

Bond strength values of up to 50 MPa have been reported when using the combination of tribochemical treatment and adhesive primers [125].

Regarding the adhesive cement layer, Giovana et al. [125] investigated the effect of cement thickness on dentin bond strength using thicknesses of 75, 340, and 630 μm . Their results showed that the best bond strength—approximately 23 MPa—was obtained at a 75 μm cement layer.

These findings are supported by additional studies emphasizing that a thin adhesive cement layer and a proper bond to the ceramic structure are essential to improve retention and enhance fracture resistance in all-ceramic crowns. It is therefore recommended to maintain a minimum cement thickness of approximately 50 μm to reduce the negative effects of water absorption on the mechanical properties of the adhesive [125].

2.6 CAD/CAM Indirect restoration processing

The evolution of minimally invasive restorative concepts—emphasizing maximal preservation of dental structure and minimizing the "biological costs" associated with conventional preparations—has significantly transformed contemporary clinical practice [126]. CAD/CAM systems include additive technologies (e.g., 3D printing) and subtractive technologies (e.g., milling). In this work, only subtractive techniques for indirect restorations will be addressed.

Within this paradigm, ceramics have become the material of choice for indirect aesthetic restorations, currently manufactured through various methods: traditional manual layering on refractory models, conventional pressing, and increasingly, digital CAD/CAM processes.

Manual layering of feldspathic porcelains continues to yield outstanding aesthetic outcomes; however, it is a time-consuming process, requires high technical expertise, and flexural strength remains limited (≤ 120 MPa) until the restoration is cemented [155]. The pressed lithium disilicate technique, on the other hand, simplifies fabrication and improves mechanical performance (≈ 350 – 450 MPa), while maintaining excellent marginal adaptation. Nevertheless, it involves several analog steps, increasing both laboratory time and overall cost [155].

CAD/CAM systems offer a digital response to these limitations, comprising either additive technologies (e.g., 3D printing) or subtractive technologies (e.g., milling). In this study, the focus will be on subtractive technology for indirect restorations.

A complete subtractive CAD/CAM system can be divided into three main components [127]:

- (1) Data Acquisition Unit, which captures information from the target area, adjacent and opposing structures, and then converts them into virtual models. This is typically done using intraoral scanners although many clinicians still rely on conventional impression techniques due to cost, training requirements, or equipment availability;
- (2) Processing Unit, which refers to the software responsible for virtual model processing and CAM programming, including calculation of tool paths;
- (3) Production Unit, which involves a computerized milling device that manufactures the restoration from a solid block of restorative material.

Intraoral scanners capture dentition areas by overlaying a series of individual images that are processed in real time by image acquisition software to reconstruct a three-dimensional virtual model. The scanning camera is positioned at different angles to ensure accurate results. In areas where information cannot be directly captured, the software extrapolates from adjacent data to complete the virtual model [126]. Figure 2.13 shows an example of an intraoral scanner from 3Shape®.

Digital scanning offers several advantages over conventional impression techniques (e.g., mock-ups), especially in terms of patient comfort [126,127], ease of use for the dentist, and the creation of a virtual database that can be accessed when needed.



Figure 2.13 - Intraoral Scanner from 3 Shape® [128].

According to comparative studies of scanners using different technologies, the reliability of intraoral scanners is not affected by the type of technology used (optical, laser, or contact), but rather by specific system parameters. Moreover, digital scanning is more accurate on smooth and regular surfaces [129].

In 2017, Imburgia et al. [130] assessed the accuracy and precision of four reference intraoral scanners. They concluded that the scanners offered accuracy between 46 and 62 μm and precision between 19 and 25 μm for partial maxillary scans. These values increase with full-arch scanning due to cumulative errors.

Some studies [126], suggest that optical scanners may eventually be replaced by ultrasonic scanners, which use ultrasound waves capable of penetrating gingival tissue non-invasively and are unaffected by oral fluids.

Once the data is acquired, the restoration is designed using CAD software, allowing for precise and customized planning—the processing phase. During this step, the operator can adjust the cement gap in specific regions of the preparation, which is a critical variable for optimal restoration fit [131]. However, there is no clear consensus regarding the ideal cement gap thickness or its clinical impact [127].

Once the CAD model is finalized, parameters such as block size, milling strategies, and cutting tools are defined using CAM software. These settings are compiled into a machine-readable file that instructs the milling unit to remove material accordingly the production phase. Zirconia milling is performed using high-precision CNC milling machines, such as the Roland DG DWX-51D, which features 5 axes and allows for simultaneous milling of multiple restorations with high reproducibility (Figure 2.14). The process begins with fixing the zirconia disc in the machine (Figure 2.14 B), a critical step to avoid vibrations that may compromise structural integrity. Milling proceeds via multi-axis technology, allowing to control the material reduction and to produce restorations with excellent detail (Figure 2.14 C) and fit.



Figure 2.14 - DWX 51D five-axis milling unit by Roland DG. (A) Overview of the equipment used for milling ceramic restorations. (B) Detail of the zirconia disc fixation process within the milling unit. (C) Close-up of the simultaneous milling of multiple structures in the zirconia block. [Adapted from Roland DG, Japan].

Generic parameters for zirconia milling are typically recommended by the CAD/CAM system manufacturer. Table 2.5 provides standard values for pre-sintered zirconia milling. However, these parameters can be optimized by the user depending on the specific material and tool used.

Table 2.5 - General milling parameters for pre-sintered zirconia [Roland DG].

Material	Tool	Process	Spindle Speed (RPM)	Cutting Speed X/Y (mm/min)	Feed (mm)	Cutting Speed Z (mm/min)	Cutting depth Z (mm)	Finishing Margin (mm)
Zirconia	Ø 2 mm ball-end	Roughing	25000	2100	1	1200	0.8	0.15
	Ø 1 mm ball-end	Finishing	25000	900	0.1	900	0.1	-

CAD/CAM restorations are fabricated from blocks of varying shapes, sizes, and shades (Figure 2.15), depending on the clinical case, design, and desired final geometry. These blocks also vary in mechanical strength and translucency depending on their intended application [132].



Figure 2.15 - Different types of blocks used in the production of dental restorations through milling [133].

For zirconia restorations, pre-sintered blocks (more porous) are commonly used. After the final shape is achieved, the restoration undergoes sintering in a furnace to fully densify the material. During this step, a volume shrinkage of approximately 20% occurs, which is compensated for during the CAD design stage [134,135]. Milling pre-sintered zirconia is the preferred method because the final sintering process eliminates the residual stresses induced during machining [122]. In addition, tool lifespan is extended due to reduced wear compared to milling fully sintered zirconia.

Since zirconia blocks vary in density across manufacturers, machining parameters must be adjusted to achieve the highest quality outcome. Demarbaix et al. reviewed the literature and identified a lack of data on the impact of machining parameters on milling quality. Most studies referenced cutting speed, neglecting other critical factors such as spindle speed, penetration rate, feed rate, and tool geometry. The authors successfully applied the NF E66-520-5 standard to determine the optimal working range of a cutting tool for Y-TZP zirconia milling [136].

Despite the efficiency of CAD/CAM production, discrepancies can exist between the virtual model and the final restoration. Scientific evidence shows that there is no consensus on clinically acceptable marginal discrepancies, with reported values ranging from 50 to 200 μm [137]. Since the clinical goal for marginal gap is typically 25 to 40 μm , CAD/CAM manufacturers strive to keep deviations within that range.

Zirconia blocks are typically produced by pressing zirconia powder with a binder inside a mold using axial pressing. To ensure uniform (partial) densification, cold isostatic pressing is used,

applying equal pressure in all directions. After pressing, the block is placed in a furnace at approximately 1000 °C for pre-sintering, giving it sufficient strength to withstand machining forces.

Color and translucency are also influenced by the block production process, particularly during pre-sintering. Sufficient porosity must be maintained to allow for liquid dye absorption, ensuring the desired shade and level of translucency [132].

2.7 Zirconia properties evaluation

In restorative dentistry, the evaluation of material properties is essential for ensuring the long-term success, functionality, and aesthetics of indirect restorations. Given the complex intraoral environment, characterized by mechanical stress, thermal variations, chemical exposure and microbial activity, materials used in dental restorations must exhibit excellent wear resistance, adhesive performance, and optical stability — i.e., the ability to retain color, translucency, and surface gloss over time despite exposure to staining agents, thermal changes, and mechanical wear. This is especially critical for ultra-thin zirconia veneers, where reduced thickness significantly increases the vulnerability to degradation, making the accurate assessment of their mechanical and optical behavior even more relevant.

2.7.1 Tribological evaluation

The oral cavity is a highly complex environment where various wear phenomena continuously occur due to physiological actions, such as mastication, and personal habits, including oral hygiene and parafunctional behaviors. This wear affects both natural teeth and the materials used in dental restorations. To ensure the clinical success and longevity of restorations, it is essential to evaluate the wear resistance of these materials under simulated clinical conditions. Understanding these mechanisms is critical for improving the long-term effectiveness and durability of dental restorations [138].

Oral biotribology traditionally distinguishes three main types of dental wear: abrasion, erosion, and attrition, with abfraction recently proposed as a fourth category. Each phenomenon results from distinct mechanisms. Abrasion refers to mechanical wear caused by external objects or excessive oral hygiene practices. Erosion is the result of chemical exposure to acidic substances, while attrition arises from direct contact between teeth during mastication and mandibular movements. Abfraction, in turn, is related to excessive biomechanical stresses in the cervical region of the tooth [138,139]. Saliva plays a significant protective role, acting as a lubricant and reducing

wear effects. However, pathological conditions such as bruxism or xerostomia can markedly increase wear rates, requiring special attention in the development and selection of restorative materials [140].

To simulate the wear of dental restorative materials, laboratory-based tribological tests are commonly employed and generally categorized into three main types: two-body wear tests, three-body wear tests, and toothbrushing simulation tests. Two-body wear tests include methods such as pin-on-disc, unidirectional sliding abrasion, oscillatory abrasion, and fretting tests, which simulate direct contact between two solid surfaces [64,138]. Three-body wear tests simulate conditions more representative of mastication by incorporating abrasive particles or toothpaste between the test surfaces. Toothbrushing simulation tests directly replicate wear due to daily oral hygiene, using standardized experimental setups involving applied load, frequency, and repeated motions [69]. Several material characteristics have been associated with wear resistance in dental restorative materials. Recent studies [141] indicate that characteristics such as hardness, surface roughness, surface finishing (polished vs. glazed), microstructure, and coefficient of friction directly influence wear resistance and the impact on opposing enamel. For example, although zirconia is among the hardest materials, surface roughness has proven to be the most significant factor in enamel wear. Specifically, polished zirconia has been shown to cause less enamel wear than less hard materials with rougher surfaces, such as lithium disilicate glass ceramics [141]. Yttria-stabilized zirconia (3Y-TZP) is of particular interest due to its high fracture toughness and stable surface integrity, standing out as an ideal material for long-lasting restorations with minimal antagonistic enamel wear [141].

Despite the advantages of laboratory tribological testing—such as rigorous control of experimental variables and direct comparison between materials under standardized conditions—important limitations must be acknowledged. These include the challenge of replicating exact biological and environmental conditions of the oral cavity, such as dynamic changes in temperature, pH, biofilm presence, chewing cycles, and patient-specific clinical factors. These discrepancies can affect the clinical relevance of lab results and therefore require careful interpretation alongside data from controlled clinical studies [142].

Given this thesis's specific focus on the development and CAD/CAM production of ultra-thin 3Y-TZP zirconia veneers, the relevance of these tribological methods becomes evident. Ultra-thin veneers, due to their extremely reduced thickness, are particularly sensitive to surface wear, which can rapidly compromise their aesthetic and functional performance. Therefore, the application of

robust and validated wear testing methodologies is essential to ensure that the selected zirconia maintains its surface and structural properties over time.

2.7.2 Bond strength evaluation

The adhesion between restorative materials and dental substrates plays a critical role in the longevity and clinical success of indirect restorations. Various methods have been developed to assess the bond strength between dental substrates and restorative materials, which are generally classified according to the type of stress applied during the test.

The most commonly used methods can be divided into static and dynamic tests. Static tests are further categorized into macrotests (adhesive areas greater than 3 mm²) and microtests (adhesive areas less than or equal to 1 mm²) [143,144]. Among the macrotests, the most prominent are shear bond strength (SBS), tensile bond strength (TBS), and push-out (PO) tests. The SBS test is widely used due to its technical simplicity and quick execution; however, it presents limitations related to non-uniform stress distribution, which often leads to cohesive failures in the dental substrate [144]. The TBS test, on the other hand, provides a more uniform stress distribution, allowing more accurate estimation of the stress required to initiate fracture, though it is highly susceptible to slippage effects that can affect the results. The push-out test is particularly relevant for assessing adhesion in root canals and better reflects real clinical failures, despite being more complex to perform [144].

Microtests, such as micro-shear bond strength (μ SBS) and micro-tensile bond strength (μ TBS), allow for the evaluation of smaller areas, reducing premature failures caused by high internal stresses. However, μ SBS also faces challenges with uniform stress distribution, particularly in fragile substrates, while μ TBS offers improved stress distribution and more precise evaluation of exclusively adhesive failures [144].

Dynamic tests (fatigue testing) better simulate the real conditions of repetitive masticatory loading in the oral cavity. Examples of these include macro and micro push-out, dynamic shear, rotary fatigue, and three- or four-point bending tests [144].

In addition, the test machine's crosshead speed significantly affects the results. Higher speeds (e.g., 5.0 and 10.0 mm/min) result in greater bond strength values compared to lower speeds (0.1 to 0.75 mm/min). This factor is important in defining clinically relevant laboratory protocols [143,145].

Bonding between substrates and restorative materials involves various mechanisms, including mechanical, physical, chemical, or a combination of these. Mechanical bonding occurs through the interlocking of materials in surface irregularities. Physical bonding is based on weak intermolecular forces, such as Van der Waals⁶ forces, while chemical bonding involves covalent, ionic, or hydrogen bonds between surfaces [68].

To ensure effective bonding, various surface treatments are applied in the dental laboratory. These include abrasive particle blasting (typically aluminum oxide - Al_2O_3), tribochemical silica coatings (e.g., Rocatec® or Cojet®), chemical treatments such as acids and bases, and advanced physical treatments such as chemical vapor deposition (CVD) and laser treatment [68].

In the specific case of zirconia ceramics, mechanochemical treatments are highly effective. These include sandblasting with Al_2O_3 particles (30–50 μm at pressures between 0.2–0.3 MPa), followed by application of primers containing functional monomers such as 10-MDP [146] his treatment significantly enhances the adhesion of resin cements to zirconia.

In bond strength tests, it is essential to recognize that different types of failures may occur: adhesive failure (occurring strictly at the interface between materials), cohesive failure (occurring within one of the substrates, indicating that adhesion exceeded the intrinsic strength of the material), and mixed failure (a combination of the two). Differentiating these failure types is crucial for accurately interpreting test results and identifying the weakest link in the adhesive interface [143].

Thus, laboratory methods for bond strength evaluation must be continuously optimized to faithfully reflect clinical conditions, considering different testing methodologies and appropriate surface treatments. This approach ensures better prediction of the clinical performance of restorative dental materials, especially in ceramic systems like zirconia. Specifically, in the development and fabrication of ultra-thin zirconia veneers, detailed understanding of these adhesive mechanisms and surface treatments becomes essential, as it guarantees a strong and durable bond between the ceramic veneers and the underlying dental structures, directly contributing to the aesthetic and functional success of the restorations.

⁶ Van der Waals forces are weak intermolecular forces that arise from the interaction between neutral molecules.

2.7.3 Optical properties evaluation

Optical properties play a critical role in determining the clinical and aesthetic success of ceramic dental restorations, particularly in cases such as ultra-thin veneers, where accurately replicating the natural characteristics of teeth is essential. Among the most relevant optical parameters are translucency, reflectance, absorption, and light scattering, all of which are fundamental for reproducing the natural and harmonious appearance of dental structures [147].

The most commonly employed methods to characterize these optical properties are spectrophotometry and spectroradiometry, using standardized parameters such as the Translucency Parameter (TP) and the Contrast Ratio (CR) within the CIE Lab* color system. The TP is calculated as the color difference of the material when measured over black and white backgrounds—higher values indicate greater translucency. For translucent dental ceramics, TP values typically range between 10 and 25 units. In turn, the CR ranges from 0 (completely transparent) to 1 (completely opaque), with translucent ceramics typically exhibiting CR values below 0.7 [148,149].

The translucency of zirconia-based ceramics is highly influenced by their microstructure and chemical composition. Ceramics containing 3 mol% Y_2O_3 (3Y-TZP) are predominantly composed of tetragonal phase with small grain sizes (average 0.3 to 0.5 μm), resulting in lower translucency compared to ceramics with higher yttria content (e.g., 5 mol% Y_2O_3 – 5Y-PSZ), which exhibit larger grain sizes and increased cubic phase content, thus achieving higher translucency values (TP near or above 20 units) [150].

Sintering is another key factor that significantly impacts optical properties. Recent studies [151,152] have evaluated the effects of speed-sintering compared to conventional slow sintering cycles. Rapid processes, typically lasting between 30 and 60 minutes, have shown similar translucency outcomes to traditional protocols (which last around 6 to 8 hours), without significant compromise of the crystalline structure. Nevertheless, some reports indicate a slight reduction in translucency (up to 5%) depending on the parameters used during the fast sintering process.

Artificial aging, commonly performed in an autoclave at 134 °C and 2 bar pressure for periods ranging from 5 to 50 hours, may lead to notable changes in the optical properties of zirconia ceramics due to the tetragonal-to-monoclinic phase transformation at the surface. This transformation may result in a decrease in initial translucency by approximately 10 to 15% in 3Y-

TZP ceramics, while ceramics with higher cubic content (such as 4Y- or 5Y-PSZ) show greater optical resistance to aging, with translucency losses under 5% over comparable timeframes [150].

Surface treatments, such as mechanical polishing or glaze application, also influence the optical behavior of ceramics. Polished surfaces generally present higher translucency compared to glazed ones, as glaze layers increase surface reflectance and may slightly reduce perceived translucency (typically by 5% to 10%). Proper polishing provides a smoother, more translucent surface and is especially recommended in highly aesthetic restorations such as ultra-thin veneers [153].

From a clinical perspective, these optical properties are decisive in the selection of ceramic materials for ultra-thin veneers. Ceramics with high translucency—such as 5Y-PSZ-based or glass ceramics—are often preferred for cases requiring ideal aesthetic reproduction. However, the use of 3Y-TZP ceramics with extremely reduced thicknesses (≤ 0.5 mm) has also been explored due to their high mechanical strength combined with satisfactory optical performance, especially when appropriately sintered and polished [149].

Thus, it is essential to thoroughly clearly understand the variables influencing the optical properties of dental ceramics to ensure the proper selection, preparation, and handling of materials for specific clinical applications—particularly for ultra-thin zirconia veneers, where aesthetic and functional integration is key to clinical success.

References

- [1] R.G. Phulari, Textbook of Dental Anatomy, Physiology and Occlusion, 2016. [https://doi.org/10.25241/stomaeduj.2016.3\(1-2\).bookreview.1](https://doi.org/10.25241/stomaeduj.2016.3(1-2).bookreview.1).
- [2] F. Patrícia, M. Carvalhais, Saúde Oral em Idade Escolar: O Papel Fundamental do Farmacêutico, (2014). http://bdigital.ufp.pt/bitstream/10284/4877/1/PPG_18005.pdf.
- [3] P.J. Taub, S.R. Buchman, P.K. Patel, M.N. Cohen, Ferraro's fundamentals of maxillofacial surgery, 2015. <https://doi.org/10.1007/978-1-4614-8341-0>.
- [4] J.L. Ferracane, U. Lohbauer, W.M. Palin, The mechanical behavior of the material-tissue and material-material interface in dental reconstructions, *Int. J. Adhes. Adhes.* 69 (2016) 2–14. <https://doi.org/10.1016/J.IJADHADH.2016.03.021>.
- [5] D. Arola, D. Bajaj, J. Ivancik, H. Majd, D. Zhang, FATIGUE OF BIOMATERIALS: HARD TISSUES., *Int. J. Fatigue.* 32 (2010) 1400–1412. <https://doi.org/10.1016/j.ijfatigue.2009.08.007>.
- [6] R.M. Pereira, R.G. Ribas, T.L.D.A. Montanheiro, V.M. Schatkoski, K.F. Rodrigues, L.T. Kito, L.K. Kobo, T.M.B. Campos, E.A. Bonfante, P.C. Gierthmuehlen, F.A. Spitznagel, G.P. Thim, An engineering perspective of ceramics applied in dental reconstructions, *J. Appl. Oral Sci.* 31 (2023) 1–20. <https://doi.org/10.1590/1678-7757-2022-0421>.

- [7] G.W. Marshall, M. Balooch, R.R. Gallagher, S.A. Gansky, S.J. Marshall, Mechanical properties of the dentinoenamel junction: AFM studies of nanohardness, elastic modulus, and fracture, *J. Biomed. Mater. Res.* 54 (2001) 87–95. [https://doi.org/10.1002/1097-4636\(200101\)54:1<87::AID-JBM10>3.0.CO;2-Z](https://doi.org/10.1002/1097-4636(200101)54:1<87::AID-JBM10>3.0.CO;2-Z).
- [8] Y.L. Chan, A.H.W. Ngan, N.M. King, Nano-scale structure and mechanical properties of the human dentine-enamel junction, *J. Mech. Behav. Biomed. Mater.* 4 (2011) 785–795. <https://doi.org/10.1016/j.jmbbm.2010.09.003>.
- [9] P. Bayle, M.C. Dean, New ways to think about enamel and dentine thickness in longitudinal tooth sections, *Bulletin Int. Assoc. Paleodont.* 7 (2013) 3–10.
- [10] D.M. Daubert, J.L. Kelley, Y.G. Udod, C. Habor, C.G. Kleist, I.K. Furman, I.N. Tikonov, W.J. Swanson, F.A. Roberts, Human enamel thickness and ENAM polymorphism, *Int. J. Oral Sci.* 8 (2016) 93–97. <https://doi.org/10.1038/ijos.2016.1>.
- [11] G. Freedman, *CONTEMPORARY ESTHETIC DENTISTRY*, Elsevier Inc., 2012.
- [12] P.D. Battersby, S.J. Battersby, Measurements and modelling of the influence of dentine colour and enamel on tooth colour, *J. Dent.* 43 (2015) 373–381. <https://doi.org/10.1016/j.jdent.2014.11.003>.
- [13] D. Bartlett, D. Ricketts, *Inlays, onlays and veneers*, Elsevier Ltd, 2011. <https://doi.org/10.1016/B978-0-7020-3126-7.00012-0>.
- [14] A. Mulic, G. Svendsen, S.E. Kopperud, A retrospective clinical study on the longevity of posterior Class II cast gold inlays/onlays, *J. Dent.* 70 (2018) 46–50. <https://doi.org/10.1016/j.jdent.2017.12.010>.
- [15] F. Angeletaki, A. Gkogkos, E. Papazoglou, D. Kloukos, Direct versus indirect inlay/onlay composite restorations in posterior teeth. A systematic review and meta-analysis, *J. Dent.* 53 (2016) 12–21. <https://doi.org/10.1016/j.jdent.2016.07.011>.
- [16] I. Naert, 6.623 - *Materials in Fixed Prosthodontics for Indirect Dental Restorations*, Elsevier Ltd., 2011. <https://doi.org/http://dx.doi.org/10.1016/B978-0-08-055294-1.00222-1>.
- [17] A. Aspros, *Inlays & Onlays Clinical Experiences and Literature Review*, *J. Dent. Heal. Oral Disord. Ther.* 2 (2015) 1–7. <https://doi.org/10.15406/jdhodt.2015.02.00038>.
- [18] I. Klineberg, S. Eckert, *Functional Occlusion in Restorative Dentistry and Prosthodontics*, Elsevier Health Sciences UK, 2015.
- [19] Randal Watson, *Inlays and Onlays: A Conservative Alternative to Dental Crowns*, (n.d.). <https://www.rwatsondds.com/restorative/inlays-onlays/> (accessed January 14, 2025).
- [20] Coroaas dentárias permanentes ou temporárias., (n.d.). <https://www.vitacentro.com/coroas-dentarias> (accessed March 20, 2018).
- [21] A.M./ S. Yolanda Smith, Liji Thomas, *Dental Crown Uses*, (n.d.). <https://www.news-medical.net/health/Dental-Crown-Uses.aspx> (accessed January 14, 2025).
- [22] Harmony Dental, *Bridges*, (n.d.). <https://www.lvharmonydental.com/bridges/>.
- [23] Smile Science Dental SPA, *Dental Veneers: The Pros, Cons, and Surprising Realities*, (n.d.). <https://smilescience.com/dental-veneers-the-pros-cons-and-surprising-realities/> (accessed January 14, 2025).
- [24] S.Y. Chai, V. Bennani, J.M. Aarts, K. Lyons, Incisal preparation design for ceramic veneers: A critical review, *J. Am. Dent. Assoc.* 149 (2018) 25–37. <https://doi.org/10.1016/j.adaj.2017.08.031>.
- [25] J.A. Cardoso, P.J. Almeida, S. Fernandes, C.L. Silva, A. Pinho, A. Fischer, L. Simões, Co-existence of crowns and veneers in the anterior dentition: case report., *Eur. J. Esthet. Dent.* 4 (2009) 12–26.

- [26] M. Peumans, B. Van Meerbeek, P. Lambrechts, G. Vanherle, Porcelain veneers: A review of the literature, *J. Dent.* 28 (2000) 163–177. [https://doi.org/10.1016/S0300-5712\(99\)00066-4](https://doi.org/10.1016/S0300-5712(99)00066-4).
- [27] A. Ben-Amar, Porcelain laminate veneers—for improved aesthetics of anterior teeth., *Refuat Hashinayim.* 7 (1989) 17–23. <http://www.ncbi.nlm.nih.gov/pubmed/2605818> (accessed March 20, 2018).
- [28] M. GRESNIGT, *Clinical and Laboratory Evaluation of Laminate Veneers*, 2011.
- [29] R.K. Morita, M.F. Hayashida, Y.M. Pupo, G. Berger, R.D. Reggiani, E.A.G. Betiol, Minimally Invasive Laminate Veneers: Clinical Aspects in Treatment Planning and Cementation Procedures, *Case Rep. Dent.* 2016 (2016). <https://doi.org/10.1155/2016/1839793>.
- [30] B. MG, A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces, *J Dent Res.* 34 (1955) 53. <https://doi.org/10.1177/00220345550340060801>.
- [31] C. JR, Etched porcelain facial veneers: a new treatment modality based on scientific and clinical evidence, *N Y J Dent.* 53 (1983) 9.
- [32] H. HR, Porcelain laminate veneers bonded to etched enamel, *Dent Clin North Am.* 4 (1983) 84.
- [33] E.A. McLaren, B. LeSage, Feldspathic veneers: what are their indications?, *Compend. Contin. Educ. Dent.* 32 (2011) 44–49.
- [34] M. Peumans, B. Van Meerbeek, P. Lambrechts, G. Vanherle, Porcelain veneers: a review of the literature., *J. Dent.* 28 (2000) 163–77. <http://www.ncbi.nlm.nih.gov/pubmed/10709338> (accessed March 20, 2018).
- [35] G. Gurel, *The Science and Art of Porcelain Laminate Veneers*, Quintessence Publishing Co. Ltd, 2003.
- [36] C.J. Tredwin, S. Naik, N.J. Lewis, C. Scully Cbe, Hydrogen peroxide tooth-whitening (bleaching) products: Review of adverse effects and safety issues, *Br. Dent. J.* 200 (2006) 371–376. <https://doi.org/10.1038/sj.bdj.4813423>.
- [37] C.E. de O. Lima, L.S. Carreiro, P.E. Baggio, P.M. Tondelli, R. Takahashi, A.C.G. da Silva, Avaliação dos Efeitos da Remoção da Resina Residual e do Polimento na Superfície do Esmalte após Descolagem do Braquete, *Rev. Nav. Odontol.* 50 (2023) 5–14. <https://doi.org/10.22491/25149.50-2-1>.
- [39] K.J. Anusavice, *PHILLIPS' SCIENCE OF DENTAL MATERIALS*, 12th ed., 2013.
- [40] M.S. Zafar, F. Amin, M.A. Fareed, H. Ghabbani, S. Riaz, Z. Khurshid, N. Kumar, Biomimetic aspects of restorative dentistry biomaterials, *Biomimetics.* 5 (2020) 1–42. <https://doi.org/10.3390/biomimetics5030034>.
- [41] R. ME, A thousand years of dental implants: a definitive history—part 1, *Compend Contin Educ Dent.* 16 (1995) 1060,1062,1064.
- [42] U. Eduok, Microbiologically induced intergranular corrosion of 316L stainless steel dental material in saliva, *Mater. Chem. Phys.* 313 (2024) 128799. <https://doi.org/10.1016/j.matchemphys.2023.128799>.
- [43] S. Ali, M. Irfan, U.M. Niazi, A.M.A. Rani, A. Rashedi, S. Rahman, M.K.A. Khan, M.A. Alsaiari, S. Legutko, J. Petrů, A. Trefil, Microstructure and Mechanical Properties of Modified 316L Stainless Steel Alloy for Biomedical Applications Using Powder Metallurgy, *Materials (Basel).* 15 (2022). <https://doi.org/10.3390/ma15082822>.
- [44] MatWeb - Material Property Data, AISI Type 316L Stainless Steel, annealed bar, (n.d.). <https://www.matweb.com/search/DataSheet.aspx?MatGUID=a2d0107bf958442e9f8db6dc9933fe31> (accessed January 22, 2025).

- [45] M. Roach, Base Metal Alloys Used for Dental Restorations and Implants, *Dent. Clin. North Am.* 51 (2007) 603–627. <https://doi.org/10.1016/j.cden.2007.04.001>.
- [46] J. Loch, A. Łukaszczyk, J. Augustyn-Pieniążek, H. Krawiec, Electrochemical behaviour of Co-Cr and Ni-Cr dental alloys, *Solid State Phenom.* 227 (2015) 451–454. <https://doi.org/10.4028/www.scientific.net/SSP.227.451>.
- [47] M. Kassapidou, V.F. Stenport, C.B. Johansson, M. Syverud, P. Hammarström Johansson, J. Börjesson, L. Hjalmarsson, Cobalt chromium alloys in fixed prosthodontics: Investigations of mechanical properties and microstructure, *J. Prosthet. Dent.* 130 (2023) 255.e1-255.e10. <https://doi.org/10.1016/j.prosdent.2023.05.005>.
- [48] B. Henriques, Bond Strenght enhancement of metal-ceramic dental restorations by FGM design, University of Minho, 2012.
- [49] B. Grosgeat, A. Vaicelyte, R. Gauthier, C. Janssen, M. Le Borgne, Toxicological Risks of the Cobalt–Chromium Alloys in Dentistry: A Systematic Review, *Materials (Basel)*. 15 (2022) 1–16. <https://doi.org/10.3390/ma15175801>.
- [50] H. Heller, I. Beitlitz, T. Goldberger, A. Emodi-Perlman, S. Levartovsky, Outcomes and Complications of 33 Soft-Milled Cobalt-Chromium-Ceramic Full-Arch Screw-Retained Implant-Supported Prostheses: A Retrospective Study with up to 10-Year Follow-Up, *J. Funct. Biomater.* 14 (2023). <https://doi.org/10.3390/jfb14030157>.
- [51] H.J. Rack, J.I. Qazi, Titanium alloys for biomedical applications, *Mater. Sci. Eng. C*. 26 (2006) 1269–1277. <https://doi.org/10.1016/j.msec.2005.08.032>.
- [52] A. Arieira, S. Madeira, F. Rodrigues, F. Silva, Tribological Behavior of TiO₂ PEEK Composite and Stainless Steel for Pediatric Crowns, *Materials (Basel)*. 16 (2023). <https://doi.org/10.3390/ma16062420>.
- [53] S. Madeira, A.M.P. Pinto, L.C. Rodrigues, O. Carvalho, G. Miranda, R.L. Reis, J. Caramês, F.S. Silva, Effect of sintering pressure on microstructure and mechanical properties of hot-pressed Ti6Al4V-ZrO₂ materials, *Mater. Des.* 120 (2017) 394–403. <https://doi.org/10.1016/j.matdes.2017.02.038>.
- [54] C.N. Elias, D.J. Fernandes, C.R.S. Resende, J. Roestel, Mechanical properties, surface morphology and stability of a modified commercially pure high strength titanium alloy for dental implants, *Dent. Mater.* 31 (2015) e1–e13. <https://doi.org/10.1016/j.dental.2014.10.002>.
- [55] S.C.S. Madeira, Development of hybrid titanium-zirconia based materials by pressure-assisted sintering for dental implants, Universidade do Minho, 2019.
- [56] Erpro Group, Materialdatenblatt Stainless Steel 316L / 1.4404 / A276, (2018).
- [57] B. Stawarczyk, M. Özcan, A. Trottmann, F. Schmutz, M. Roos, C. Hämmerle, Two-body wear rate of CAD/CAM resin blocks and their enamel antagonists, *J. Prosthet. Dent.* 109 (2013) 325–332. [https://doi.org/10.1016/S0022-3913\(13\)60309-1](https://doi.org/10.1016/S0022-3913(13)60309-1).
- [58] B. Stawarczyk, A. Liebermann, M. Eichberger, J.F. Güth, Evaluation of mechanical and optical behavior of current esthetic dental restorative CAD/CAM composites, *J. Mech. Behav. Biomed. Mater.* 55 (2016) 1–11. <https://doi.org/10.1016/j.jmbbm.2015.10.004>.
- [59] L.C. Ardelean, Additive Manufactured Polymers in Dentistry, Current, Currente State of the Art and Future Perspectives-A Review, *Polymers (Basel)*. 14 (2022) 1–24. <https://doi.org/https://doi.org/10.3390/polym14173658>.
- [60] S. Najeeb, M.S. Zafar, Z. Khurshid, F. Siddiqui, Applications of polyetheretherketone (PEEK) in oral implantology and prosthodontics, *J. Prosthodont. Res.* 60 (2016) 12–19. <https://doi.org/10.1016/j.jpor.2015.10.001>.

- [61] H. Kaur, A. Thakur, Applications of poly(methyl methacrylate) polymer in dentistry: A review, *Mater. Today Proc.* 50 (2021) 1619–1625. <https://doi.org/10.1016/j.matpr.2021.09.125>.
- [62] V. Alt, M. Hannig, B. Wöstmann, M. Balkenhol, Fracture strength of temporary fixed partial dentures: CAD/CAM versus directly fabricated restorations, *Dent. Mater.* 27 (2011) 339–347. <https://doi.org/10.1016/j.dental.2010.11.012>.
- [63] Z.É. Vincze, L. Nagy, K. Kelemen, B.G.N. Cavalcante, N. Gede, P. Hegyi, D. Bányai, L. Köles, K. Márton, Milling has superior mechanical properties to other fabrication methods for PMMA denture bases: A systematic review and network meta-analysis, *Dent. Mater.* (2025). <https://doi.org/10.1016/j.dental.2024.12.017>.
- [64] A. Carvalho, P. Pinto, S. Madeira, F.S. Silva, O. Carvalho, J.R. Gomes, Tribological Characterization of Dental Restorative Materials, *Biotribology*. 23 (2020) 100140. <https://doi.org/10.1016/J.BIOTRI.2020.100140>.
- [65] M. Alqahtani, Mechanical properties enhancement of self-cured PMMA reinforced with zirconia and boron nitride nanopowders for high-performance dental materials, *J. Mech. Behav. Biomed. Mater.* 110 (2020) 103937. <https://doi.org/10.1016/j.jmbbm.2020.103937>.
- [66] P. Sikder, A comprehensive review on the State of the Art in the research and development of poly-ether-ether-ketone (PEEK) biomaterial-based implants, *Acta Biomater.* 191 (2024) 29–52. <https://doi.org/10.1016/j.actbio.2024.11.033>.
- [67] K.P. Parate, N. Naranje, R. Vishnani, P. Paul, Polyetheretherketone Material in Dentistry, *Cureus*. 15 (2023) 1–7. <https://doi.org/10.7759/cureus.46485>.
- [68] O. Abdullah Alsadon, Adhesion concepts and techniques for laboratory-processed indirect dental restorations, *Saudi Dent. J.* 34 (2022) 661–668. <https://doi.org/10.1016/j.sdentj.2022.09.007>.
- [69] P. Pinto, A. Carvalho, F.S. Silva, J.R. Gomes, O. Carvalho, S. Madeira, Comparative toothbrush abrasion resistance and surface analysis of different dental restorative materials, *Tribol. Int.* 175 (2022) 107799. <https://doi.org/10.1016/j.triboint.2022.107799>.
- [70] Y. Alothman, M.S. Bamasoud, The success of dental veneers according to preparation design and material type, *Open Access Maced. J. Med. Sci.* 6 (2018) 2402–2408. <https://doi.org/10.3889/oamjms.2018.353>.
- [71] C.R.F. Yanikian, F. Yanikian, D. Sundfeld, R.B.E. Lins, L.R.M. Martins, Direct composite resin veneers in nonvital teeth: A still viable alternative to mask dark substrates, *Oper. Dent.* 44 (2019) E159–E166. <https://doi.org/10.2341/18-220-T>.
- [72] G.C. Souza, R.K. Silva, Facetas diretas em resina composta ou indiretas em cerâmica : qual é a melhor opção ? Direct veneers in composite resin or indirect veneers in ceramics : which is the best option ? Carillas directas en resina compuesta o carillas indirectas en cerámica : , 2022 (2022) 1–9.
- [73] J.L. Ferracane, Resin composite - State of the art, *Dent. Mater.* 27 (2011) 29–38. <https://doi.org/10.1016/j.dental.2010.10.020>.
- [74] T. Alluhaidan, M. Qaw, I.M. Garcia, C. Montoya, S. Orrego, M.A. Melo, Seeking Endurance: Designing Smart Dental Composites for Tooth Restoration, *Designs*. 8 (2024) 1–18. <https://doi.org/10.3390/designs8050092>.
- [75] J. Da Costa, A. Adams-Belusko, K. Riley, J.L. Ferracane, The effect of various dentifrices on surface roughness and gloss of resin composites, *J. Dent.* 38 (2010) e123–e128. <https://doi.org/10.1016/j.jdent.2010.02.005>.

- [76] D.H. Lozano, J. Miguel, A. Vera, S. Alfredo, A. Vera, Facetas diretas em resina composta : Uma revisão abrangente Direct resin composite veneers : An extensive review Carillas de resina compuesta directa : Una revisión exhaustiva, 2024 (2024) 1–12.
- [77] N.H. Fares, H.N. Filho, I.B. Pacheco, K.Q. Coutinho, H.D. Nagem, Resistência Flexural E Módulo De Elasticidade Da Resina Composta, *Rev. Clín. Pesq. Odontol.* 2 (2005) 55–58.
- [78] J.L. Ferracane, T.J. Hilton, Polymerization stress - Is it clinically meaningful?, *Dent. Mater.* 32 (2016) 1–10. <https://doi.org/10.1016/j.dental.2015.06.020>.
- [79] I. Nedeljkovic, W. Teughels, J. De Munck, B. Van Meerbeek, K.L. Van Landuyt, Is secondary caries with composites a material-based problem?, *Dent. Mater.* 31 (2015) e247–e277. <https://doi.org/10.1016/j.dental.2015.09.001>.
- [80] W.F. Liberato, N. Silikas, D.C. Watts, L.M. Cavalcante, L.F.J. Schneider, Luting laminate veneers: Do resin-composites produce less polymerization stress than resin cements?, *Dent. Mater.* 39 (2023) 1190–1201. <https://doi.org/10.1016/j.dental.2023.09.010>.
- [81] M.L. Faria, M.A. e Sousa, Reabilitação estética em dentes anteriores com facetas em cerâmica e em resina composta, Universidade de Lisboa, 2024.
- [82] L.A.V. de Paula, L.A.N. Araújo, A.M.G. Matos, M.A. Mussolin, J.V. de O. Pereira, G. dos R. Rodrigues, L.T.A. de Souza, FALHAS EM FACETAS DENTÁRIAS: UMA PERSPECTIVA INTEGRAL SOBRE SAÚDE E ESTÉTICA BUCAL, (2024) 90–109.
- [83] F. Takeshige, M.T. Y Kinomoto, Additional heat-curing of light-cured composite resin for inlay restoration, *J Osaka Univ Dent Sch.* 35 (1995) 59–66.
- [84] B. Hussain, M.K. Le Thieu, G.F. Johnsen, J.E. Reseland, H.J. Haugen, Can CAD/CAM resin blocks be considered as substitute for conventional resins?, *Dent. Mater.* 33 (2017) 1362–1370. <https://doi.org/10.1016/j.dental.2017.09.003>.
- [85] S. Abu Alhuda, G.A. Arossi, F. Anagnostopoulos-King, Q. Alkhubaizi, L.S. Mokeem, M.A.S. de Melo, Current Evidence and Advances in CAD-CAM Resin Composite Blocks for Chairside Dental Restorations: Where Are We Now? A Scoping Review, *Appl. Sci.* 14 (2024). <https://doi.org/10.3390/app142210423>.
- [86] L. Ling, Y. Ma, R. Malyala, A novel CAD/CAM resin composite block with high mechanical properties, *Dent. Mater.* 37 (2021) 1150–1155. <https://doi.org/10.1016/j.dental.2021.03.006>.
- [87] G.W. Ho, J.P. Matinlinna, Insights on Ceramics as Dental Materials. Part I: Ceramic Material Types in Dentistry, *Silicon.* 3 (2011) 109–115. <https://doi.org/10.1007/s12633-011-9078-7>.
- [88] J. Lin, V. Bennani, J.M. Aarts, P. Brunton, J. Ratnayake, Factors influencing success rate of ceramic veneers on endodontically treated anterior teeth: A systematic review, *J. Prosthet. Dent.* 133 (2023) 62–70. <https://doi.org/10.1016/j.prosdent.2023.10.031>.
- [89] F. Komine, M. Furuchi, J. Honda, K. Kubochi, H. Takata, Clinical performance of laminate veneers: A review of the literature, *J. Prosthodont. Res.* 68 (2024) 368–379. https://doi.org/10.2186/JPR.JPR_D_23_00151.
- [90] B. Henriques, Bond strength enhancement of metal-ceramic dental restorations by FGM design, Universidade do Minho, 2012.
- [91] E.B. Benalcazar Jalkh, K.N. Monteiro, P.F. Cesar, L.A. Genova, E.T.P. Bergamo, A.C. de O. Lopes, E. Lima, P.N. Lisboa-Filho, T.M.B. Campos, L. Witek, P.G. Coelho, A.F.S. Borges, E.A. Bonfante, Aging resistant ZTA composite for dental applications: Microstructural, optical and mechanical characterization, *Dent. Mater.* 36 (2020) 1190–1200. <https://doi.org/10.1016/j.dental.2020.05.011>.

- [92] P.F. Cesar, R.B. de P. Miranda, K.F. Santos, S.S. Scherrer, Y. Zhang, Recent advances in dental zirconia: 15 years of material and processing evolution, *Dent. Mater.* 40 (2024) 824–836. <https://doi.org/10.1016/j.dental.2024.02.026>.
- [93] P. Pinto, Influence of Applied Pressure and Thickness Variation on the Bond Strength Between 3Y- - TZP Zirconia and Adhesive Resin Cement, (2025) 1–10. <https://doi.org/10.1002/jbm.b.35563>.
- [94] A.L. Kozlovskiy, M. Alin, D.B. Borgekov, Study of Polymorphic Transformation Processes and Their Influence in Polycrystalline ZrO₂ Ceramics upon Irradiation with Heavy Ions, *Ceramics*. 6 (2023) 686–706. <https://doi.org/10.3390/ceramics6010042>.
- [95] J. Chevalier, B. Cales, J.M. Drouin, Low-Temperature Aging of Y-TZP Ceramics, *J. Am. Ceram. Soc.* 82 (2004) 2150–2154. <https://doi.org/10.1111/j.1151-2916.1999.tb02055.x>.
- [96] K.L. & M.S. B. AL-AMLEH, Clinical trials in zirconia: a systematic review, *J. Oral Rehabil.* 37 (2010) 641–652.
- [97] I. Denry, J.R. Kelly, State of the art of zirconia for dental applications, *Dent. Mater.* 24 (2008) 299–307. <https://doi.org/10.1016/J.DENTAL.2007.05.007>.
- [98] G.K.R. Pereira, A.B. Venturini, T. Silvestri, K.S. Dapieve, A.F. Montagner, F.Z.M. Soares, L.F. Valandro, Low-temperature degradation of Y-TZP ceramics: A systematic review and meta-analysis, *J. Mech. Behav. Biomed. Mater.* 55 (2016) 151–163. <https://doi.org/10.1016/j.jmbbm.2015.10.017>.
- [99] J. Chevalier, S. Deville, E. Münch, R. Jullian, F. Lair, Critical effect of cubic phase on aging in 3 mol% yttria-stabilized zirconia ceramics for hip replacement prosthesis, *Biomaterials*. 25 (2004) 5539–5545. <https://doi.org/10.1016/j.biomaterials.2004.01.002>.
- [100] V. Lughi, V. Sergo, Low temperature degradation -aging- of zirconia: A critical review of the relevant aspects in dentistry, *Dent. Mater.* 26 (2010) 807–820. <https://doi.org/10.1016/j.dental.2010.04.006>.
- [101] B. Huang, M. Chen, J. Wang, X. Zhang, Advances in zirconia-based dental materials: Properties, classification, applications, and future prospects, *J. Dent.* 147 (2024) 105111. <https://doi.org/10.1016/j.jdent.2024.105111>.
- [102] P. Shelar, S. Butler, H. Abdolvand, On the behaviour of zirconia-based dental materials: A review, *J. Mech. Behav. Biomed. Mater.* 124 (2021) 104861. <https://doi.org/10.1016/j.jmbbm.2021.104861>.
- [103] G.-J. Oh, K.-D. Yun, K.-M. Lee, H.-P. Lim, S.-W. Park, Sintering behavior and mechanical properties of zirconia compacts fabricated by uniaxial press forming, *J. Adv. Prosthodont.* 2 (2010) 81. <https://doi.org/10.4047/jap.2010.2.3.81>.
- [104] L. Yunbing, Z. Kaiyin, X. Jinsheng, W. Dongsheng, Thermal stresses analysis of ceramic/metal functionally gradient material cylinder, *Appl. Math. Mech.* 20 (1999) 413–417. <https://doi.org/10.1007/BF02458568>.
- [105] R.H.W. Brodbelt, W.J. O'Brien, P.L. Fan, Translucency of Dental Porcelains, *J. Dent. Res.* 59 (1980) 70–75. <https://doi.org/10.1177/00220345800590011101>.
- [106] S.F. Wang, J. Zhang, D.W. Luo, F. Gu, D.Y. Tang, Z.L. Dong, G.E.B. Tan, W.X. Que, T.S. Zhang, S. Li, L.B. Kong, Transparent ceramics: Processing, materials and applications, *Prog. Solid State Chem.* 41 (2013) 20–54. <https://doi.org/10.1016/J.PROGSOLIDSTCHEM.2012.12.002>.
- [107] R. Shahmiri, O.C. Standard, J.N. Hart, C.C. Sorrell, Optical properties of zirconia ceramics for esthetic dental restorations: A systematic review, *J. Prosthet. Dent.* 119 (2018) 36–46. <https://doi.org/10.1016/j.prosdent.2017.07.009>.
- [108] F. Tabatabaian, Color in Zirconia-Based Restorations and Related Factors: A Literature Review, *J. Prosthodont.* 27 (2018) 201–211. <https://doi.org/10.1111/jopr.12740>.

- [109] J.Y. Thompson, B.R. Stoner, J.R. Piascik, R. Smith, Adhesion/cementation to zirconia and other non-silicate ceramics: Where are we now?, *Dent. Mater.* 27 (2011) 71–82. <https://doi.org/10.1016/j.dental.2010.10.022>.
- [110] M. Kern, S.M. Wegner, Bonding to zirconia ceramic: adhesion methods and their durability., *Dent. Mater.* 14 (1998) 64–71. <http://www.ncbi.nlm.nih.gov/pubmed/9972153> (accessed March 20, 2018).
- [111] J.R. Piascik, S.D. Wolter, B.R. Stoner, Development of a novel surface modification for improved bonding to zirconia, *Dent. Mater.* 27 (2011) e99–e105. <https://doi.org/10.1016/j.dental.2011.01.005>.
- [112] M.N. Aboushelib, J.P. Matinlinna, Z. Salameh, H. Ounsi, Innovations in bonding to zirconia-based materials: Part I, *Dent. Mater.* 24 (2008) 1268–1272. <https://doi.org/10.1016/J.DENTAL.2008.02.010>.
- [113] Z. Ozkurt, E. Kazazoglu, A. Unal, In vitro evaluation of shear bond strength of veneering ceramics to zirconia., *Dent. Mater. J.* 29 (2010) 138–46. <http://www.ncbi.nlm.nih.gov/pubmed/20379023> (accessed March 20, 2018).
- [114] M. Amaral, R. Belli, P.F. Cesar, L.F. Valandro, A. Petschelt, U. Lohbauer, The potential of novel primers and universal adhesives to bond to zirconia, *J. Dent.* 42 (2014) 90–98. <https://doi.org/10.1016/j.jdent.2013.11.004>.
- [115] S.M. Reddy, D. Vijitha, T. Deepak, R. Balasubramanian, A. Satish, Evaluation of shear bond strength of zirconia bonded to dentin after various surface treatments of zirconia, *J. Indian Prosthodont. Soc.* 14 (2014) 38–41. <https://doi.org/10.1007/s13191-012-0198-6>.
- [116] R. Amaral, M. Özcan, M.A. Bottino, L.F. Valandro, Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning, *Dent. Mater.* 22 (2006) 283–290. <https://doi.org/10.1016/j.dental.2005.04.021>.
- [117] P. Thammajaruk, M. Inokoshi, S. Chong, M. Guazzato, Bonding of composite cements to zirconia: A systematic review and meta-analysis of in vitro studies, *J. Mech. Behav. Biomed. Mater.* 80 (2018) 258–268. <https://doi.org/10.1016/J.JMBBM.2018.02.008>.
- [118] L. Yin, Y. Nakanishi, A.R. Alao, X.F. Song, J. Abduo, Y. Zhang, A Review of Engineered Zirconia Surfaces in Biomedical Applications, *Procedia CIRP.* 65 (2017) 284–290. <https://doi.org/10.1016/j.procir.2017.04.057>.
- [119] F.A. Mudado, Cimentação Adesiva De Cerâmicas À Base De Zircônia, (2012) 60. http://www.bibliotecadigital.ufmg.br/dspace/bitstream/handle/1843/BUOS-9E9HGG/monografiafl_viaamatamudado.pdf?sequence=1.
- [120] C. Monaco, A. Tucci, L. Esposito, R. Scotti, Microstructural changes produced by abrading Y-TZP in presintered and sintered conditions, *J. Dent.* 41 (2013) 121–126. <https://doi.org/10.1016/J.JDENT.2012.06.009>.
- [121] H.L. de Castro, P.H. Corazza, T. de A. Paes-Júnior, A. Della Bona, Influence of Y-TZP ceramic treatment and different resin cements on bond strength to dentin, *Dent. Mater.* 28 (2012) 1191–1197. <https://doi.org/10.1016/j.dental.2012.09.003>.
- [122] C.P. Zucuni, L.F. Guilardi, S. Fraga, L.G. May, G.K.R. Pereira, L.F. Valandro, CAD/CAM machining Vs pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: Effects on their mechanical fatigue behavior, *J. Mech. Behav. Biomed. Mater.* 71 (2017) 201–208. <https://doi.org/10.1016/J.JMBBM.2017.03.013>.
- [123] S. KURIYAMA, Y. TERUI, D. HIGUCHI, D. GOTO, Y. HOTTA, A. MANABE, T. MIYAZAKI, Novel fabrication method for zirconia restorations: Bonding strength of machinable ceramic to zirconia with resin cements, *Dent. Mater. J.* 30 (2011) 419–424. <https://doi.org/10.4012/dmj.2010-213>.

- [124] M. Inokoshi, A. Kameyama, J. De Munck, S. Minakuchi, B. Van Meerbeek, Durable bonding to mechanically and/or chemically pre-treated dental zirconia, *J. Dent.* 41 (2013) 170–179. <https://doi.org/10.1016/j.jdent.2012.10.017>.
- [125] K. Takeuchi, A. Fujishima, A. Manabe, S. Kuriyama, Y. Hotta, Y. Tamaki, T. Miyazaki, Combination treatment of tribochemical treatment and phosphoric acid ester monomer of zirconia ceramics enhances the bonding durability of resin-based luting cements., *Dent. Mater. J.* 29 (2010) 316–23. <http://www.ncbi.nlm.nih.gov/pubmed/20484831> (accessed March 20, 2018).
- [126] T.F. Alghazzawi, Advancements in CAD/CAM technology: Options for practical implementation, *J. Prosthodont. Res.* 60 (2016) 72–84. <https://doi.org/10.1016/j.jpor.2016.01.003>.
- [127] R.O. Pilecco, R.V. Machry, A. Baldi, J.P.M. Tribst, R. Sarkis-Onofre, L.F. Valandro, C.J. Kleverlaan, N. Scotti, G.K.R. Pereira, Influence of CAD-CAM milling strategies on the outcome of indirect restorations: A scoping review, *J. Prosthet. Dent.* 131 (2024) 811.e1-811.e10. <https://doi.org/10.1016/j.prosdent.2024.02.021>.
- [128] Scanners Intraorais, (n.d.). <https://www.3shape.com/pt-br/products/trios/intraoral-scanners> (accessed March 20, 2018).
- [129] P. González de Villambrosia, F. Martínez-Rus, A. García-Orejas, M.P. Salido, G. Pradies, In vitro comparison of the accuracy (trueness and precision) of six extraoral dental scanners with different scanning technologies, *J. Prosthet. Dent.* 116 (2016) 543-550.e1. <https://doi.org/10.1016/j.prosdent.2016.01.025>.
- [130] M. Imburgia, S. Logozzo, U. Hauschild, G. Veronesi, C. Mangano, F.G. Mangano, Accuracy of four intraoral scanners in oral implantology: A comparative in vitro study, *BMC Oral Health.* 17 (2017) 1–13. <https://doi.org/10.1186/s12903-017-0383-4>.
- [131] C. Tokita, M. Maeno, Y. Nara, The effect of space setting values and restorative block materials on the bonding of metal-free CAD/CAM onlay restorations, *Dent. Mater. J.* 40 (2021) 994–1006. <https://doi.org/10.4012/dmj.2020-293>.
- [132] U. Today, Milling Materials, (n.d.).
- [133] D. Sirona, CAD/CAM Materials for the dental lab, (n.d.).
- [134] I. Naert, 7.24 Materials in fixed prosthodontics for indirect dental restorations, Elsevier Ltd., 2017. <https://doi.org/10.1016/B978-0-08-100691-7.00244-5>.
- [135] T. Miyazaki, Y. Hotta, J. Kunii, S. Kuriyama, Y. Tamaki, A review of dental CAD/CAM: current status and future perspectives from 20 years of experience., *Dent. Mater. J.* 28 (2009) 44–56. <http://www.ncbi.nlm.nih.gov/pubmed/19280967> (accessed March 20, 2018).
- [136] A. Demarbaix, E. Rivière-Lorphèvre, F. Ducobu, E. Filippi, F. Petit, N. Preux, Behaviour of pre-sintered Y-TZP during machining operations: Determination of recommended cutting parameters, *J. Manuf. Process.* 32 (2018) 85–92. <https://doi.org/10.1016/j.jmapro.2018.01.020>.
- [137] S. Papadiochou, A.L. Pissiotis, Marginal adaptation and CAD-CAM technology: A systematic review of restorative material and fabrication techniques, *J. Prosthet. Dent.* (2017) 1–7. <https://doi.org/10.1016/j.prosdent.2017.07.001>.
- [138] Y. Zheng, K. Bashandeh, A. Shakil, S. Jha, A.A. Polycarpou, Review of dental tribology: Current status and challenges, *Tribol. Int.* 166 (2022) 107354. <https://doi.org/10.1016/j.triboint.2021.107354>.
- [139] R.P.S.; M. Addy, The Interactions between Attrition, Abrasion and Erosion in Tooth Wear, in: A.L.C. Ganss (Ed.), *Erosive Tooth Wear From Diagnosis to Ther.*, 2014: pp. 32–45. <https://doi.org/https://doi.org/10.1159/isbn.978-3-318-02553-8>.

- [140] H. Bronkhorst, S. Kalaykova, M.C. Huysmans, B. Loomans, T. Pereira-Cenci, Tooth wear and bruxism: A scoping review, *J. Dent.* 145 (2024) 104983. <https://doi.org/10.1016/j.jdent.2024.104983>.
- [141] Okkar Kyaw, M. Inokoshi, M. Kanazawa, Tribological aspects of enamel wear caused by zirconia and lithium disilicate: A meta-narrative review, *Jpn. Dent. Sci. Rev.* 60 (2024) 258–270. <https://doi.org/10.1016/j.jdsr.2024.11.001>.
- [142] M. Masen, P. Cann, Tribology test design for friction measurements with application to oral medicines, *Biotribology*. 35–36 (2023) 100260. <https://doi.org/10.1016/j.biotri.2023.100260>.
- [143] R.R. Braga, J.B.C. Meira, L.C.C. Boaro, T.A. Xavier, Adhesion to tooth structure: A critical review of “macro” test methods, *Dent. Mater.* 26 (2010) 38–49. <https://doi.org/10.1016/j.dental.2009.11.150>.
- [144] A.M. El Mourad, Assessment of Bonding Effectiveness of Adhesive Materials to Tooth Structure using Bond Strength Test Methods: A Review of Literature, *Open Dent. J.* 12 (2018) 664–678. <https://doi.org/10.2174/1745017901814010664>.
- [145] H. Hassan Elandif, B.W. Darvell, Effect of cross-head speed on the bond strength of dental resin to tooth structure – A review and re-analysis, *Dent. Mater.* 40 (2024) 2085–2100. <https://doi.org/10.1016/j.dental.2024.09.020>.
- [146] A. Mazaheri Tehrani, M. Nami, A. Zarbakhsh, A. Moscowchi, E. Jalalian, Impact of surface pre-treatment on bond strength between cement and zirconia: A systematic review and network meta-analysis, *J. Prosthet. Dent.* (2025) 1–13. <https://doi.org/10.1016/j.prosdent.2024.12.014>.
- [147] R. Shahmiri, O.C. Standard, J.N. Hart, C.C. Sorrell, Optical properties of zirconia ceramics for esthetic dental restorations: A systematic review, *J. Prosthet. Dent.* 119 (2018) 36–46. <https://doi.org/10.1016/j.prosdent.2017.07.009>.
- [148] H.N. Lim, B. Yu, Y.K. Lee, Spectroradiometric and spectrophotometric translucency of ceramic materials, *J. Prosthet. Dent.* 104 (2010) 239–246. [https://doi.org/10.1016/S0022-3913\(10\)60131-X](https://doi.org/10.1016/S0022-3913(10)60131-X).
- [149] T.F. Alghazzawi, J. Lemons, P.R. Liu, M.E. Essig, G.M. Janowski, Evaluation of the optical properties of CAD-CAM generated yttria-stabilized zirconia and glass-ceramic laminate veneers, *J. Prosthet. Dent.* 107 (2012) 300–308. [https://doi.org/10.1016/S0022-3913\(12\)60079-1](https://doi.org/10.1016/S0022-3913(12)60079-1).
- [150] B. Hajhamid, N. Alfrisany, E. Somogyi-Ganss, The effect of accelerated aging on crystalline structures and optical properties of different monolithic zirconia: A qualitative systematic review, *Dent. Mater.* 38 (2022) 569–586. <https://doi.org/10.1016/j.dental.2022.02.005>.
- [151] H. Liu, M. Inokoshi, K. Xu, W. Tonprasong, S. Minakuchi, B. Van Meerbeek, J. Vleugels, F. Zhang, Does speed-sintering affect the optical and mechanical properties of yttria-stabilized zirconia? A systematic review and meta-analysis of in-vitro studies, *Jpn. Dent. Sci. Rev.* 59 (2023) 312–328. <https://doi.org/10.1016/j.jdsr.2023.08.007>.
- [152] M.H. Abed, A.J. Kadhim, Effect of speed sintering process on the microstructure, flexural strength and translucency of zirconia, *Heliyon*. 10 (2024) e37848. <https://doi.org/10.1016/j.heliyon.2024.e37848>.
- [153] R. Sorrentino, G. Ruggiero, M.I. Di Mauro, L. Breschi, S. Leuci, F. Zarone, Optical behaviors, surface treatment, adhesion, and clinical indications of zirconia-reinforced lithium silicate (ZLS): A narrative review, *J. Dent.* 112 (2021) 103722. <https://doi.org/10.1016/j.jdent.2021.103722>.
- [154] N. Kumar, N. Maher, F. Amin, H. Ghabbani, M.S. Zafar, F.J. Rodríguez-Lozano, R.E. Oñate-Sánchez, Biomimetic Approaches in Clinical Endodontics, *Biomimetics*. 7 (2022). <https://doi.org/10.3390/biomimetics7040229>.

- [155] O.S. J. Schweiger, D. Edelhoff, 3D printing of ultra-thin veneers made of lithium disilicate using the LCM method in a digital workflow: A feasibility study, (2023) 588–594. <https://doi.org/https://doi.org/10.1111/jerd.1315>

C H A P T E R 3.

Comparative toothbrush abrasion resistance and surface analysis of different dental restorative materials

Published in Tribology International, Volume 175, November 2022, 107799

P. Pinto ^{*a,b,c}, A. Carvalho ^{a,b}, F. S. Silva ^{a,b,c}, J. R. Gomes ^{a,b,c}, O. Carvalho ^{a,b,c}, S. Madeira ^{a,b}

^a Center for Micro-Electro Mechanical Systems (CMEMS-UMinho), University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

^b LABBELS –Associate Laboratory, Braga, Guimarães, Portugal

^c Department of Mechanical Engineering, University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

Abstract

Different materials have been used for dental restorations, and, like a natural tooth, they are subject to wear due to abrasion produced by toothbrushing during oral hygiene routine. The purpose of the present study was to evaluate the toothbrush abrasion resistance of four restorative dental CAD/CAM materials available on the market: an acrylic polymer - PMMA (DD TempMED HI, Dental Direkt), a composite resin (BRILLIANT CRIOS cerec HT A1 14, Coltene, CH) and two ceramics (polychromatic feldspathic porcelain (VITABLOCS RealLife 1M1C, Vita Zahnfabrik) and 3Y-TZP zirconia (DD Bio ZW iso color, Dental Direkt), under the same conditions. The specimens were immersed in a mixture of toothpaste and deionized water and subjected to simulated toothbrushing tests following ISO/TR14569-1:2007. Tests were performed under 3.5 N load, 5 Hz frequency, and 10 mm stroke length. The toothbrush abrasion resistance of each tested material was evaluated through the specific wear rate. The surface roughness and color difference were assessed for all materials before and after 16 years of simulated toothbrushing tests. Statistical analysis was performed by applying one-way ANOVA. The results revealed that the surface characteristics of restorative dental materials were modified by the toothbrushing, except for zirconia. The polymer was the material more susceptible to toothbrushing wear, presenting the highest surface roughness, specific wear rate, and surface change. On the other hand, zirconia showed to be about 10 times more resistant to abrasion than feldspathic porcelain and composite and about 100 times more resistant than polymer. Thus, it is a promising solution for the promotion of longevity restorations.

Keywords: *Dental restorative materials; Toothbrushing; Abrasion; Surface roughness.*

3.1 Introduction

The oral cavity is a very complex wear environment as several mechanisms occur due to both mechanical and chemical interactions. Besides the wear from occlusion during chewing, teeth are subject to wear due to abrasion produced by oral hygiene, namely toothbrushing [1]. People suffer from certain common dental issues, such as dental caries, which damage the tooth structure despite oral care. For the treatment of the damaged tooth, different dental restorative materials, such as polymers, composites, and ceramics, have been used, and, like teeth, they are also submitted to wear conditions.

Surface quality is extremely important for its clinical success and longevity in any restoration. A smooth surface can improve the longevity of restoration as it provides both low plaque accumulation and good aesthetics, mimicking the tooth's natural appearance. In contrast, a rougher surface causes discomfort to the patient, increases bacterial retention and compromises

the aesthetic quality of restoration [2–4]. Despite the intrinsic roughness of the material conferred by finishing and polishing processes, daily toothbrushing changes the surface condition [5].

Dental professionals generally recommend toothbrushing for at least 2 min, twice daily, as the most effective practice for removing plaque and preventing caries and dental diseases [3,6]. The type of toothbrush (electric or manual)[7]; the stiffness of the toothbrush [8]; the abrasiveness of the toothpaste [9]; the brushing time [5] and load [5,7], and the dental restorative material properties [10] have been reported as factors that influence toothbrushing wear.

The stiffness of a toothbrush (soft, medium, and hard) depends on factors such as the diameter and length of the bristle and the number of bristles per unit area packed into a tuft hole [11]. There is a general opinion that hard toothbrushes cause more abrasion than soft brushes [8,11] however, there are also studies supporting the opposite [6,12,13]. The wear produced by toothpaste is influenced by the type of constituents, including the amount of abrasive agent, the particle size, and the surface structure of the particles. It has been reported that the use of toothpaste with higher abrasivity leads to increased wear [2,9,14]. Relative Dentin Abrasivity (RDA) is the abrasiveness measurement of the toothpaste, and the recommended limit by American Dental Association is 250 [2][3].

The effects of toothbrushing wear have been assessed by the surface roughness and gloss measurements [15]. Some in vitro studies [2,4,10] of resin-based composites have shown that toothbrushing leads to an increase in surface roughness and a decrease in gloss due to the wear of filler particles and degradation of the resin matrix or weakening of resin-filler bonding [16]. Consequently, the aesthetic properties of restoration are strongly affected. W. H.Mörmann et al. [17] reported a significant reduction in gloss and increased surface roughness of acrylic polymers, silicate ceramics, hybrid ceramics, and nanocomposites caused by abrasive toothbrushing except for zirconium dioxide (zirconia) ceramics. The same study stated that zirconia showed the best gloss retention. In contrast, the lowest gloss retention was attained by polymers being, therefore, the most susceptible material to toothbrushing deterioration. In another study conducted by L. A. Garza et al. [3], it was verified that leucite glass-ceramics showed no significant differences in gloss and surface roughness after 12 years of simulated toothbrushing however, the same was not confirmed for lithium disilicate glass-ceramics.

Several studies show the effect of toothbrushing of various types of restorative materials separately, differing on the abrasiveness of toothpaste, applied brushing load and time, or even toothbrushing apparatus. Limited information is available comparing the toothbrush abrasion

resistance of different restorative materials under the same test conditions, thus allowing a direct comparison.

Therefore, this study aimed to evaluate and compare the effect of toothbrushing on abrasive wear and surface modification of four different dental CAD/CAM restorative materials (polymer, composite, and ceramics: feldspathic porcelain and zirconia), under the same toothbrushing conditions, through assessment of specific wear rate, surface roughness, and color stability.

3.2 Materials and methods

3.2.1 Specimen preparation

Four commercial CAD/CAM dental restorative materials were used to replicate toothbrush abrasion damage in teeth: polymer, composite, and two ceramics (feldspathic porcelain and TZP zirconia) commonly used in clinical dentistry applications. The feldspathic porcelain used in this study was polychromatic, therefore, two-color grades (O - opaque shade and T - translucent shade) were considered, as in previous work [18]. The composition of these materials is shown in

Table 3.1. Circular discs of each material with a diameter of 10 mm, and a thickness of 2 mm, were cut from the commercial blocks. Each sample was polished with silicon carbide (SiC) papers, from 800 to 4000 grit, following the procedure widely used in the literature [4,5,19] to obtain similar initial surface roughness. Then, the samples were washed in an ultrasonic bath in isopropyl alcohol for 5 min.

Table 3.1 - Dental restorative materials tested in this study

Type of material	Brand Name	Manufacturer	Composition
Polymer	DD TempMED HI	Dental Direkt GmbH, Spenge, BRD	PMMA > 99.8%; Colouring pigments ≤ 0.2%
Composite	BRILLIANT CRIOS cerec HT A1 14	Coltene, CH	Cross-linked methacrylates; 70.7 wt% barium glass (< 1.0 µm) and silica particles (< 20 nm)
Feldspathic Porcelain	VITABLOCS RealLife 1M1C	Vita Zahnfabrik, Bad Säckingen, BRD	SiO ₂ : 56 - 64%; Al ₂ O ₃ : 20 - 23%; Na ₂ O: 6 - 9%; K ₂ O: 6 - 8%; CaO: 0.3 - 0.6%; TiO ₂ : 0.0 - 0.1% and other oxides in very low concentrations
Zirconia	DD Bio ZW iso color	Dental Direkt GmbH, Spenge, BRD	ZrO ₂ + HfO ₂ + Y ₂ O ₃ ≥ 99%; Y ₂ O ₃ < 6; Al ₂ O ₃ < 0.5%; Other oxides < 1.0%

3.2.2 Simulated toothbrushing

The simulated toothbrushing tests were performed in a custom and homemade toothbrushing simulator, as schematically represented in Figure 3.1. A toothbrush head with round parallel bristles with medium stiffness (Colgate® Extra Clean) was fixed at the top, while each specimen was fixed on a specimen holder under the toothbrush head. Up and down linear strokes completely covered the outer sample's surface.

A slurry of 70 RDA toothpaste (Colgate® Max fresh) and deionized water in a ratio of 1:2, following the ISO/TR 14569_1:2007 guideline [20], was prepared and added to the device till the cover sample. The tests were carried out under a vertical load of 3.5 N [5][10] applied at the toothbrush head and a brushing frequency of 5 Hz with a linear brushing distance of 10 mm (stroke length) to completely cover the length specimen's surface. The simulation period of 8h (corresponding to 16 years of toothbrushing, respectively) was calculated based on a brushing time estimation of 120 seconds twice-daily of all teeth (correspondent to ~5 seconds for one tooth surface per day), as reported by some studies [7]. Three samples were tested for each material. The toothbrush and the slurry were replaced every two hours. After testing, the samples were ultrasonically cleaned in distilled water for 5 min.

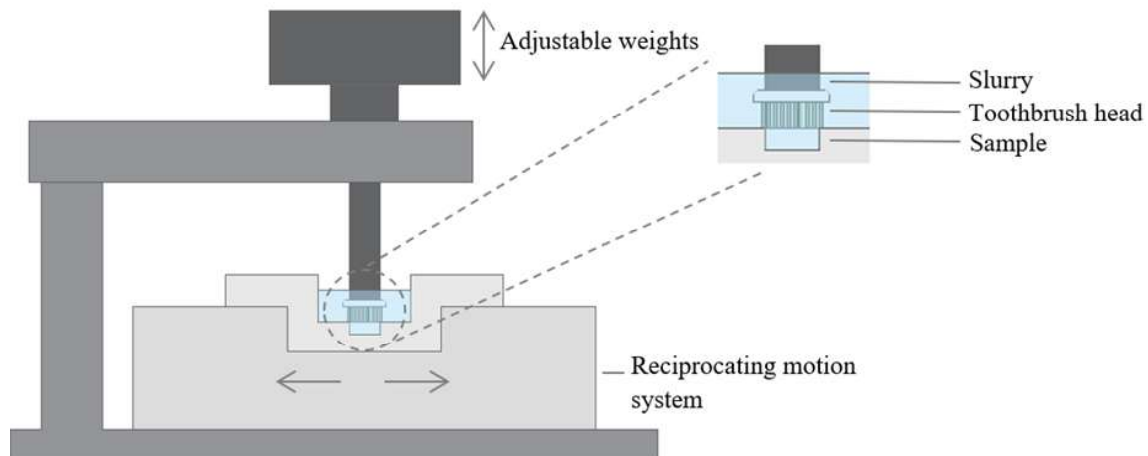


Figure 3.1 - Schematic representation of toothbrushing tests.

3.2.3 Surface characterization

The surface of the specimens was analyzed, before and after 8h of toothbrushing, using Scanning Electron Microscopy (SEM - Nova NanoSEM 200, FEI, Netherlands) equipped with an Energy Dispersive Spectrometer (EDS) at an accelerating voltage of 10 kV and a magnification of 2000x. Previously, the samples were sputter-coated with gold.

3.2.4 Surface roughness analysis

The influence of toothbrushing on the surface was evaluated by assessing average surface roughness (R_a , μm) using a 3D optical profilometer (Filmetrics Inc., USA). Five measurements were performed for each sample in the central area before and after 8h of the simulated toothbrushing abrasion test. In the case of brushed samples, the roughness was measured perpendicularly relative to the toothbrushing test. The R_a value was calculated as the average of the five measurements of each specimen. Three samples were tested for each material.

3.2.5 Color difference evaluation

The color measurement was performed before and after toothbrushing abrasion using a portable spectrophotometer (CM-2600d, Konica Minolta, Tokyo, Japan) according to Commission International de l'Eclairage $L^*a^*b^*$ (CIE $L^*a^*b^*$) system. For each material, three samples and five color measurements on the sample were performed in the initial state and after 8h of toothbrushing. The color was also measured at three intermediate periods of simulated toothbrushing, i.e., 1, 2, and 4h, to analyze the evolution of color change and lightness with brushing over time.

CIElab is a method that expresses the color in three coordinates: L^* , which represents the lightness/darkness, a^* the red/green value, and b^* the blue/yellow value. These coordinates are then used to obtain the color change, which is often calculated in the literature using the CIELab color difference formula ΔE_{ab} . However, over time, new formulas were developed, namely, the CIEDE2000 color difference formula ΔE_{00} , to improve the accuracy of the obtained values. This formula has been proved to be the most suitable for evaluating the color of dental materials [21–23]. In association with these formulas, the perceptibility and acceptability thresholds are also mentioned. The perceptibility level corresponds to the minimal perceptual difference that a human observer can detect, whereas the acceptability is the maximal perceptual difference acceptable [24]. In this study, the perceptibility and acceptability thresholds used were 1.25 and 2.23, respectively [21].

The measurements were carried out under standardized D65 light illumination, which represents the average daylight, including the ultraviolet wavelength region, and based on ISO 3664:2000 is the one that should be opted [24]. As an observer, an angle of 10° was settled, which corresponds to the observation of an object in normal conditions. The spectrophotometer settings were adjusted to a small aperture size (SAV) and specular component excluded (SCE) mode, which reflects the surface condition of specimens and is considered the most suitable geometry for dental restorative materials [25]. Previous to each measurement, the equipment was calibrated according to the instructions of the manufacturer using the white calibration standard. The color change was calculated by CIEDE2000 color difference/change (ΔE_{00}) according to equation (1) [26]:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right) \left(\frac{\Delta H'}{K_H S_H}\right)} \quad (1)$$

where ΔL^* , ΔC^* , and ΔH^* are the differences in lightness, chroma, and hue, respectively; S_L , S_C , and S_H are weighting functions for the lightness, chroma, and hue components, respectively; R_T is a rotation factor and K_L , K_C , and K_H are parametric factors that in this study were set to 1 [27].

3.2.6 Toothbrushing abrasion resistance

The toothbrushing abrasion resistance of each tested material was investigated through the specific wear rate (k). The specific wear rate was evaluated by the weight loss suffered by the material during 8h of toothbrushing, and it was calculated according to equation (2):

$$k = \frac{\Delta V}{W \times S} \quad (2)$$

where ΔV is the worn volume (mm^3), W is the normal applied load (N), and S is the total sliding distance (m). The weight loss of each specimen was evaluated through Archimedes' principle. The five-weight measurements were performed before and after 8 h of the simulated toothbrushing test.

3.2.7 Surface characterization

Statistical analysis of data was performed by one-way ANOVA for surface roughness, color stability, and wear rate, followed by Tukey HSD multiple comparison tests. Such analysis were performed by IBM SPSS statistical software, version 26 for windows, to assess the statistically significant differences at $\alpha=0.05$.

3.3 Results and Discussion

3.3.1. Surface morphological characterization

The surface morphological features of the different materials was analyzed by scanning electron microscopy (SEM) before and after 8h of simulated toothbrushing test, and the respective images are presented in Figure 3.2. Since the surface morphology of both shades of the feldspathic porcelain (opaque and translucent) were very similar, only representative images of this type of material are presented (Figure 3.2 (c) and (c1)).

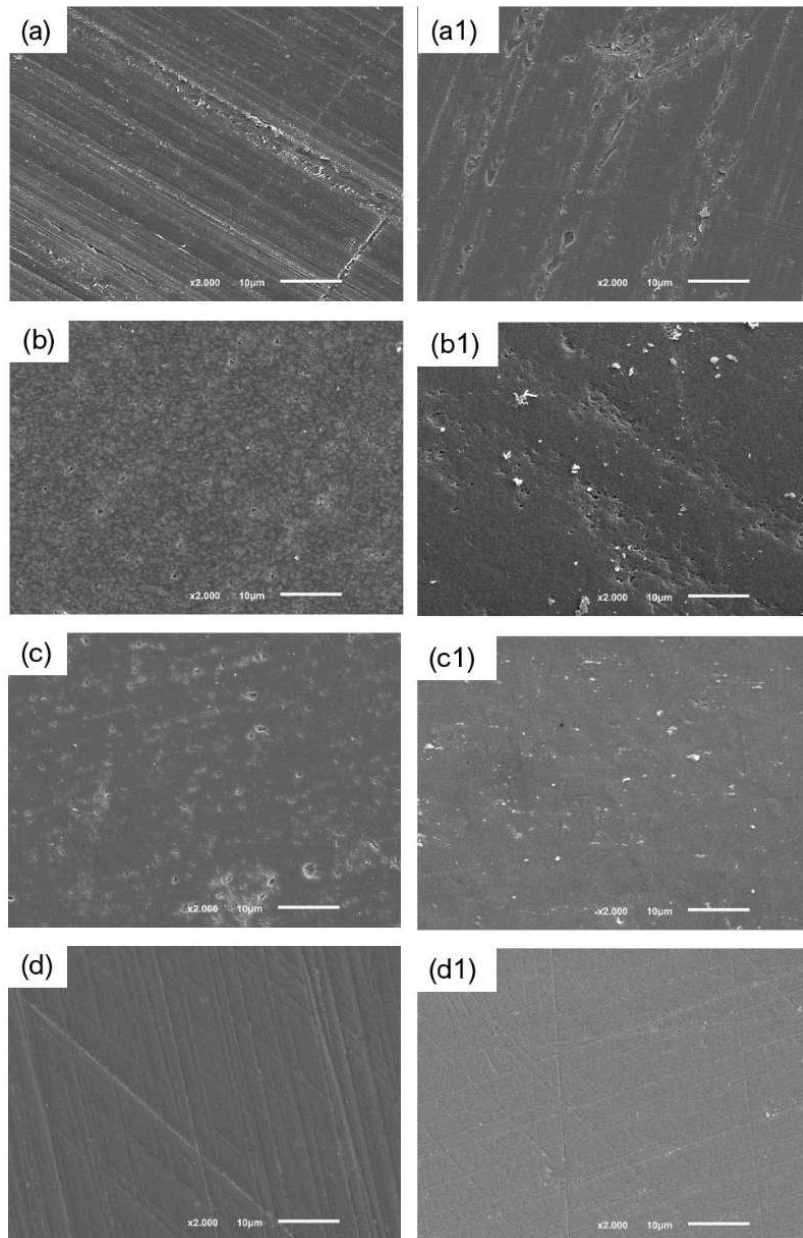


Figure 3.2 - SEM images of the surface topography of specimens before (left column) and after 8h of toothbrushing (right column). Polymer (a, a1), composite (b, b1), feldspathic porcelain (c, c1) and zirconia (d, d1).

Although all materials were subjected to the same polishing procedure, shallow scratches were observed on the surface of polymer and zirconia. After polishing, the scratches are more pronounced for polymer due to its ductility and zirconia due to its high hardness, making it a difficult polishing procedure. After toothbrushing (right column in Figure 3.2) surface roughening occurred over time, more pronounced for polymer followed by composite and ceramics, except for zirconia. Moreover, some debris on the composite and porcelain surfaces can be observed (Figure 3.2 (b1) and (c1)). Zirconia morphological surface appearance was not significantly affected by the

toothbrush abrasion, while polymer was more susceptible to increased roughing appearance. This surface morphological features are in line with surface roughness results presented in the next section.

3.3.2. Surface roughness

As previously mentioned, the surface roughness of any restoration plays an essential role in the aesthetic outcomes and the adhesion of bacteria, which influences its success and durability. In this sense, the surface roughness of four different restorative dental materials was evaluated before and after 8h of simulated toothbrushing. With this analysis, the authors intended to assess the effect of toothbrushing on the surface modification for each type of material and determine which material presented the highest toothbrush abrasion resistance.

The initial Ra surface roughness measured after polishing is listed in Table 3.2 - Initial Ra surface roughness (mean $\pm$ SD) after polishing., and designed as a reference.

Table 3.2 - Initial Ra surface roughness (mean $\pm$ SD) after polishing.

	Reference	
	Mean	SD
Polymer	0.0076	0.0006
Composite	0.0083	0.0008
Feldspathic Porcelain T	0.0073	0.0007
Feldspathic Porcelain O	0.0075	0.0011
Zirconia	0.0184	0.0010

Analyzing the results, it is possible to observe that, despite being polished with the same procedure, polymer, composite and feldspathic porcelain present similar initial average surface roughness ($R_a = 0.0073\text{--}0.0083\ \mu\text{m}$), except zirconia which exhibited the highest initial average roughness ($R_a = 0.0184\ \mu\text{m}$). This difference may be related to the difficulty of polishing the zirconia surface due to its high hardness. These results are in accordance with the SEM images (Figure 3.2 (a), (b), (c), and (d)), where some scratches are visible on the zirconia surface (Figure 3.2 (d)). Regarding the average variation of surface roughness with toothbrushing (depicted in Figure 3.3), it is possible to conclude that the surface roughness of materials was significantly affected by toothbrushing, evidenced by the increase in surface roughness values, except zirconia where roughness was not significantly affected. Polymer exhibited the highest increase ($R_a = 0.026\ \mu\text{m}$), being the most susceptible to abrasion, followed by the composite ($0.007\ \mu\text{m}$) and both shades of the feldspathic porcelain values ($R_a = 0.003 - 0.004\ \mu\text{m}$), while zirconia roughness remained

practically unchanged. Furthermore, it can be seen that there is a significant difference between zirconia and polymer as well as between zirconia and composite. These results are in accordance with the SEM images (Figure 3.2 (a1), (b1), (c1) and (d1)). Furthermore, all roughness measurements obtained in this study are below 0.2 μm which, according to the literature [2,17], indicates that the surface abrasion promoted by toothbrushing during 8h is not enough to reach the critical surface roughness threshold for plaque formation.

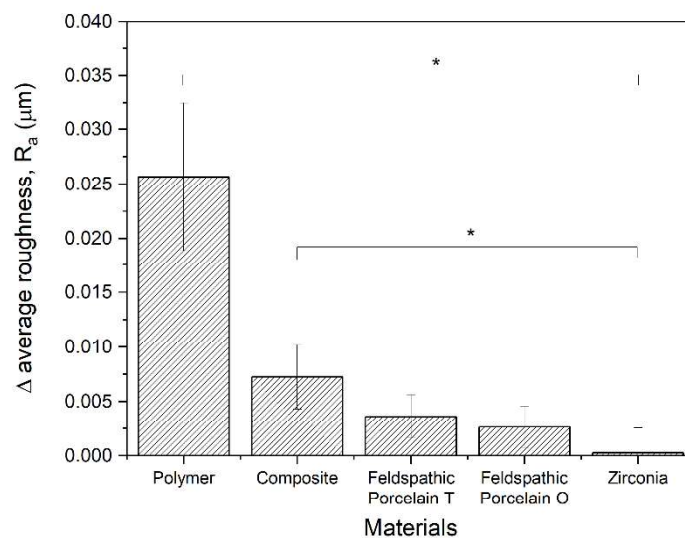


Figure 3.3 - Variation of surface roughness, R_a after 8h of toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).

The roughness results suggest that zirconia surfaces are promising solutions for dental restorations and even for dental veneers since their surface nature is expected to resist toothbrush abrasion and consequently maintain long-term aesthetic outcomes and low bacteria adhesion. The results also suggest that the shade (Opaque and Translucent) of the feldspathic porcelain material has no significant effect on the toothbrush abrasion resistance.

3.3.3. Color and lightness difference

An essential aspect of the aesthetic outcomes of any restoration is the modification of surface color with toothbrushing. Color measurements on the material's surface were performed to quantify the color change caused by toothbrushing. Table 3.3 summarizes the initial color values, while color difference (ΔE_{00}) and differences in lightness (ΔL^*) for each type of material measured after 8h of simulated toothbrushing are depicted in Figure 3.4-Figure 3.7. In order to evaluate the

clinical significance of color difference, it is important to understand color tolerances, namely perceptibility and acceptability thresholds. Perceptibility thresholds (PT) have been defined as the amount of color difference that might be detected visually by an observer. In contrast, acceptability thresholds (AT) correspond to the color difference that is clinically acceptable [3,28].

Table 3.3 - Initial color (E_{00}) values of each type of tested material.

Materials	Reference	
	Mean	SD
Polymer	70.79	0.117
Composite	64.84	0.099
Feldspathic Porcelain T	63.32	0.271
Feldspathic Porcelain O	70.60	0.357
Zirconia	76.02	0.615

Analyzing the results, it is possible to observe that the color of materials is affected by the toothbrushing as expected and the mean (ΔE_{00}) values after 8h of simulated toothbrushing range from 0.278 to 0.524 (Figure 3.4). The polymer was the most affected by the toothbrushing being the one with the most significant color change - ΔE_{00} value (0.527), followed by composite (0.418), feldspathic porcelain T (0.299), zirconia (0.297), and feldspathic porcelain O presenting the lowest ΔE_{00} value (0.278) among all the materials tested. Furthermore, it can be seen from Figure 5 that this color change increases significantly with time, especially for polymer and composite. This effect is more negligible in the case of both shade porcelains. On the other hand, the color of zirconia changes essentially during the first hour and then remains practically constant over time.

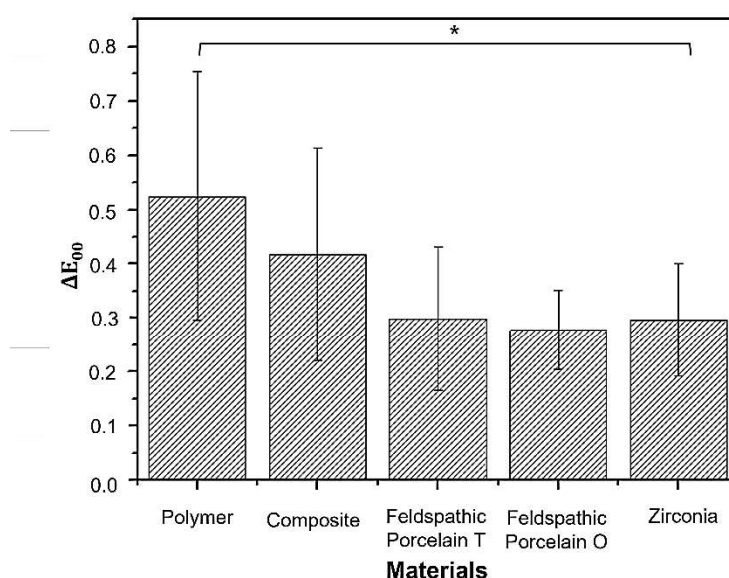


Figure 3.4 - Color change (ΔE_{00}) for each material after 8h of simulated toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).

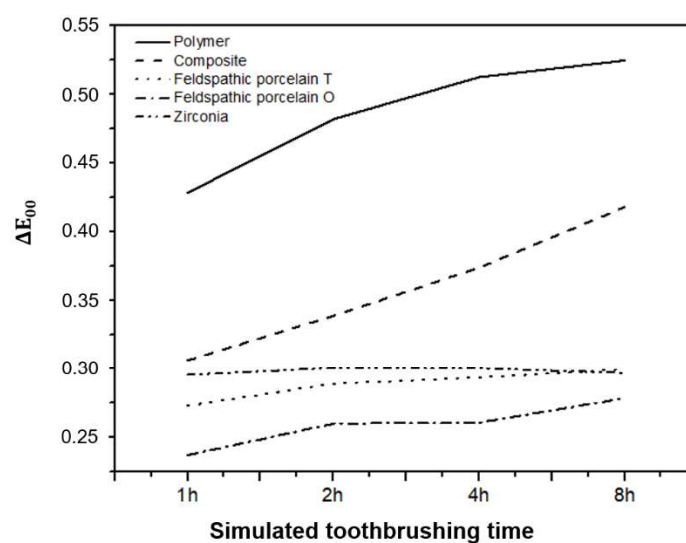


Figure 3.5 - Evolution of color change for each dental material caused by toothbrushing with time.

According to the literature [28,29], the obtained values are below the color tolerances ($\Delta E_{00} = 3.3$) used within color science in dentistry, which means that human eye cannot detect the color differences and, therefore, they are considered clinically insignificant. Despite this, it can be concluded from results that polymer is the one more susceptible to discoloration. Although the surface gloss was not measured in the present study, it can be assumed that the polymer would be the material more susceptible to a greater loss of gloss related to the increased surface roughness surface ($\Delta Ra = 0.0257 \mu m$), while a high gloss retention is expected to zirconia

restorative material [17]. The materials constituents and their ability to water absorption, or even other fluids, are other determining factors of color stability. In general, materials with higher absorption ability are more susceptible to discoloration [29]. Based on the manufacturers' information, the water absorption for the present materials is for Polymer (DD temp MED) about $25 \mu\text{g}/\text{mm}^3$, for composite (Brilliant Crios) is about $19,5 \mu\text{g}/\text{mm}^3$, and null for both ceramics due to its high densification. These values are in accordance with color differences results.

Regarding the $\Delta L'$ values (Figure 3.6), it is possible to observe that lightness of materials is affected by toothbrushing, namely, there is a reduction in L^* (lightness) values for all materials with toothbrushing. This reduction is more pronounced on polymer and is related to the increase in surface roughness promoted by toothbrushing. According to the literature [30], rough surfaces reflect less light than smooth surfaces. Compared to zirconia, the results indicate only statistically significant differences ($p < 0.05$) between the lightness of zirconia and polymer.

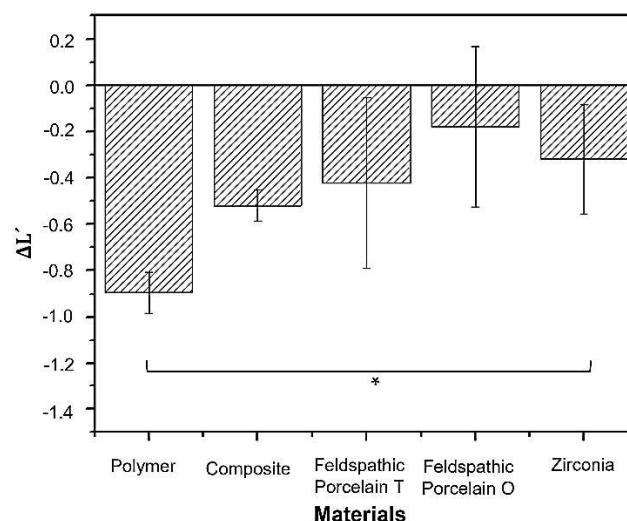


Figure 3.6 - Lightness difference for each material after 8h of simulated toothbrushing. The asterisks (*) indicate statistically significant differences ($p < 0.05$).

Furthermore, although there are no statistically significant differences, the results suggest that the reduction in lightness caused by toothbrushing is different for both shade (Opaque and Translucent) of the feldspathic porcelain material, being more pronounced for porcelain translucent. Additionally, it can be seen from Figure 3.7 that there is a more significant reduction between 2h and 4h, especially evidenced in polymer and composite materials. On the other hand, the results indicate that toothbrushing does not significantly affect the brightness of zirconia and porcelain, as a slow reduction was measured.

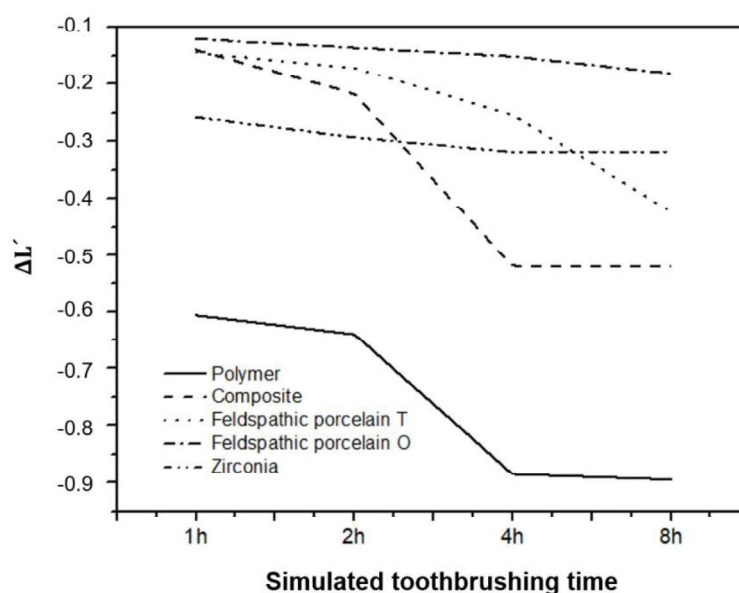


Figure 3.7 - Evolution of lightness difference for each material caused by toothbrushing with time.

It was statistically proved ($p < 0.05$) that the effect of toothbrushing on both lightness and color difference between the polymer and the zirconia is statistically significant. On the other hand, the impact of toothbrushing on lightness and color difference in composite, porcelain, and zirconia is not statistically significant.

3.3.4. Toothbrushing abrasion resistance

Abrasion resistance is an essential factor regarding the durability of dental restorations [31] and even of veneers. To assess abrasion resistance of each restorative material type, the specific wear rate promoted during 8h of simulated toothbrushing was evaluated. Figure 8 denotes the abrasion wear rate for all tested materials.

Although several works are studying the abrasion resistance of different composites, polymers, and ceramics, the absolute values can not directly be compared since the toothbrushing tests are carried out under other conditions, varying in the toothpaste slurries; press-on forces of the toothbrush; the number of toothbrushing cycles; hardness and stiffness of toothbrush bristles; methods used to prepare specimens and the simulated toothbrushing set-up, among others [5]. However, the results obtained in this study make possible the comparison of the different commercial types of restorative materials currently used in the clinical practice since the toothbrushing set-up configuration, including specimen preparation and the simulated conditions, were the same.

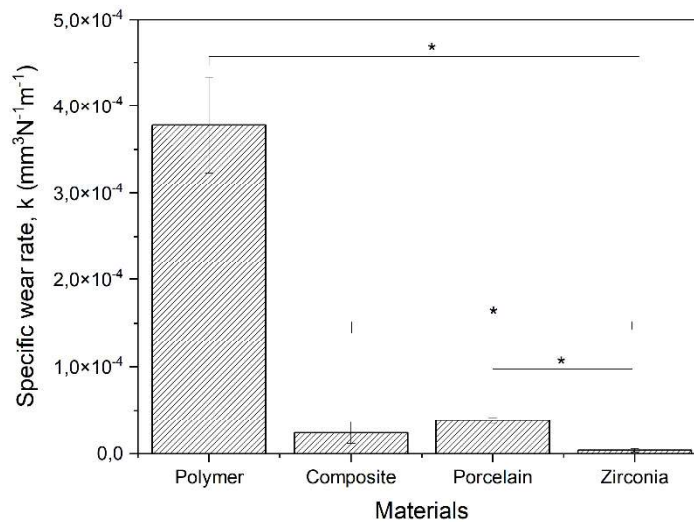


Figure 3.8 - Specific wear rate of the tested restorative dental materials. The asterisks (*) indicate statistically significant differences ($p < 0.05$).

Analyzing the results presented in Figure 3.8, it is possible to verify that polymer is the material more susceptible to toothbrushing wear. It presented the highest specific wear rate ($k = 3.8 \times 10^{-4} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$), followed by porcelain ($k = 3.9 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$), composite ($k = 2.4 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$) and zirconia exhibiting the lowest value ($k = 4.0 \times 10^{-6} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$). All materials presented a significant material loss after the toothbrushing test compared to zirconia, which was revealed to be about 10 times more resistant to abrasion than feldspathic porcelain and composite, and about 100 times more resistant than composite polymer. These findings are in accordance with surface roughness results, where no significant modification of surface roughness was found for zirconia after 8h simulated toothbrushing. In contrast, increased surface roughness was found for porcelain, composite, and polymer surfaces, more pronounced in the last one.

3.4 Conclusions

This study showed that selecting material to use in the restoration is essential since its surface characteristics, such as roughness and color, can be changed over time with a simple diary oral hygiene routine like toothbrushing.

In this study, the surface roughness, color difference, lightness, specific wear rate, and surface morphology of four dental CAD/CAM restorative materials after 8h (equivalent to 16 years) of simulated toothbrushing were evaluated, and the following conclusions can be drawn:

- Except for zirconia, all materials revealed an increase in surface roughness caused by toothbrushing, more pronounced for polymer.
- Color differences and lightness were observed for all materials over time as a consequence of toothbrushing. However, the obtained values are below the perceptibility threshold.
- The polymer was the most affected by toothbrushing, the one with the greatest color change and highest lightness, which can be related to the increase in surface roughness. On the other hand, the aesthetic outcomes of zirconia were the least affected by toothbrushing.

The results demonstrated that zirconia is about 10 times more resistant to abrasion than feldspathic porcelain and composite and about 100 times more resistant than polymer, thus being a promising solution for promoting the longevity of restorations.

Acknowledgements

This work was supported by Fundação para a Ciência e Tecnologia (FCT, Portugal) through the grant SFRH/BD/145421/2019 and the projects FunImp, POCI-01-0145-FEDER-030498 and FunFibRAI – Multi-functional bioinspired root analogue zirconia implant for smart fiber-integration, reference PTDC/EME-EME/4197/2021.

Cofinanciado por:



Fundação para a Ciência e a Tecnologia
MINISTÉRIO DA EDUCAÇÃO E CIÊNCIA



UNIÃO EUROPEIA
Fundo Europeu
de Desenvolvimento Regional

References

- [1] Tellefsen G, Liljeborg A, Johannsen G. How Do Dental Materials React On Tooth brushing? Dentistry 2015;5:11. <https://doi.org/10.4172/2161-1122.1000341>.
- [2] Da Costa J, Adams-Belusko A, Riley K, Ferracane JL. The effect of various dentifrices on surface roughness and gloss of resin composites. J Dent 2010;38:e123–8. <https://doi.org/10.1016/j.jdent.2010.02.005>.
- [3] Garza LA, Thompson G, Cho SH, Berzins DW. Effect of toothbrushing on shade and surface roughness of extrinsically stained pressable ceramics. J Prosthet Dent 2016;115:489–94. <https://doi.org/10.1016/j.prosdent.2015.09.013>.

- [4] Takahashi R, Jin J, Nikaido T, Tagami J, Hickel R, Kunzelmann KH. Surface characterization of current composites after toothbrush abrasion. *Dent Mater J* 2013;32:75–82. <https://doi.org/10.4012/dmj.2012-160>.
- [5] Heintze SD, Forjanic M, Ohmiti K, Rousson V. Surface deterioration of dental materials after simulated toothbrushing in relation to brushing time and load. *Dent Mater* 2010;26:306–19. <https://doi.org/10.1016/j.dental.2009.11.152>.
- [6] Joshi CP, Patil AG, Karde PA, Mahale SA, Dani NH. Comparative evaluation of cemental abrasion caused by soft and medium bristle hardness toothbrushes at three predetermined toothbrushing forces: An in vitro study. *J Indian Soc Periodontol* 2017;21:10–5. https://doi.org/10.4103/jisp.jisp_118_17.
- [7] Bizhang M, Schmidt I, Patricia Y-H, 2017 C, Arnold WH, Zimmer S. Toothbrush abrasivity in a long-term simulation on human dentin depends on brushing mode and bristle arrangement 2017. <https://doi.org/10.1371/journal.pone.0172060>.
- [8] Tellefsen G, Liljeborg A, Johannsen A, Johannsen G. The role of the toothbrush in the abrasion process. *Int J Dent Hyg* 2011;9:284–90. <https://doi.org/10.1111/j.1601-5037.2011.00505.x>.
- [9] Johannsen G, Tellefsen G, Johannsen A, Liljeborg A. The importance of measuring toothpaste abrasivity in both a quantitative and qualitative way. *Acta Odontol Scand* 2013;71:508–17. <https://doi.org/10.3109/00016357.2012.696693>.
- [10] Tanoue N, Matsumura H, Atsuta M. Wear and surface roughness of current prosthetic composites after toothbrush/dentifrice abrasion. *J Prosthet Dent* 2000;84:93–7. <https://doi.org/10.1067/mpr.2000.107560>.
- [11] Wiegand A, Kuhn M, Sener B, Roos M, Attin T. Abrasion of eroded dentin caused by toothpaste slurries of different abrasivity and toothbrushes of different filament diameter. *J Dent* 2009;37:480–4. <https://doi.org/10.1016/j.jdent.2009.03.005>.
- [12] Zairani O, Irawan B, Damiyanti M. The effect of toothbrush bristle stiffness on nanohybrid surface roughness. *J. Phys. Conf. Ser.*, vol. 884, Institute of Physics Publishing; 2017, p. 12008. <https://doi.org/10.1088/1742-6596/884/1/012008>.
- [13] Bizhang M, Riemer K, Arnold WH, Domin J, Zimmer S. Influence of bristle stiffness of manual toothbrushes on eroded and sound human dentin - an in vitro study. *PLoS One* 2016;11. <https://doi.org/10.1371/journal.pone.0153250>.
- [14] Monteiro B, Spohr AM. Surface Roughness of Composite Resins after Simulated Toothbrushing with Different Dentifrices. *J Int Oral Heal JIOH* 2015;7:1–5.
- [15] Rajan VV, Ganapathy DM, Sheeba PS, Kannappan N. Effects of tooth brushing on wear, surface roughness, and color stability of composite resins-A review 2018.
- [16] Jassé FF, De Campos EA, Lefever D, Di Bella E, Salomon JP, Krejci I, et al. Influence of filler charge on gloss of composite materials before and after in vitro toothbrushing. *J Dent* 2013;41:2–5. <https://doi.org/10.1016/j.jdent.2013.04.011>.
- [17] Mörmann WH, Stawarczyk B, Ender A, Sener B, Attin T, Mehl A. Wear characteristics of current aesthetic dental restorative CAD/CAM materials: Two-body wear, gloss retention, roughness and Martens hardness. *J Mech Behav Biomed Mater* 2013;20:113–25. <https://doi.org/10.1016/j.jmbbm.2013.01.003>.
- [18] Carvalho A, Pinto P, Madeira S, Silva FS, Carvalho O, Gomes JR. Tribological Characterization of Dental Restorative Materials. *Biotribology* 2020;23:100140. <https://doi.org/10.1016/J.BIOTRI.2020.100140>.

- [19] Kamonkhantikul K, Arksornnukit M, Lauvahutanon S, Takahashi H. Toothbrushing alters the surface roughness and gloss of composite resin CAD/CAM blocks. *Dent Mater J* 2016;35:225–32. <https://doi.org/10.4012/dmj.2015-228>.
- [20] ISO - ISO/TR 14569-1:2007 - Dental materials — Guidance on testing of wear — Part 1: Wear by toothbrushing n.d.
- [21] Ghinea R, Pérez MM, Herrera LJ, Rivas MJ, Yebra A, Paravina RD. Color difference thresholds in dental ceramics. *J Dent* 2010;38:e57–64. <https://doi.org/10.1016/J.JDENT.2010.07.008>.
- [22] Stevenson B, Ibbetson R. The effect of the substructure on the colour of samples/restorations veneered with ceramic: A literature review. *J Dent* 2010;38:361–8. <https://doi.org/10.1016/J.JDENT.2010.01.009>.
- [23] Salas M, Lucena C, Herrera LJ, Yebra A, Della Bona A, Pérez MM. Translucency thresholds for dental materials. *Dent Mater* 2018;34:1168–74. <https://doi.org/10.1016/J.DENTAL.2018.05.001>.
- [24] Ghinea R, Pérez MM, Herrera LJ, Rivas MJ, Yebra A, Paravina RD. Color difference thresholds in dental ceramics. *J Dent* 2010;38 Suppl 2. <https://doi.org/10.1016/J.JDENT.2010.07.008>.
- [25] Lee YK, Yu B, Lee SH, Cho MS, Lee CY, Lim HN. Variation in instrument-based color coordinates of esthetic restorative materials by measurement method—A review. *Dent Mater* 2010;26:1098–105. <https://doi.org/10.1016/J.DENTAL.2010.07.010>.
- [26] de Moraes Rego Roselino L, Alexandra Chinelatti M, Cecilia Alandia-Román C, de Carvalho Panzeri Pires-de-Souza F, Dra Fernanda P. Effect of Brushing Time and Dentifrice Abrasiveness on Color Change and Surface Roughness of Resin Composites n.d. <https://doi.org/10.1590/0103-6440201300399>.
- [27] Luo MR, Cui G, Rigg B. The development of the CIE 2000 colour-difference formula: CIEDE2000. *Color Res Appl* 2001;26:340–50. <https://doi.org/10.1002/COL.1049>.
- [28] Khashayar G, Bain PA, Salari S, Dozic A, Kleverlaan CJ, Feilzer AJ. Perceptibility and acceptability thresholds for colour differences in dentistry. *J Dent* 2014;42:637–44. <https://doi.org/10.1016/j.jdent.2013.11.017>.
- [29] Bezgin T, Özer L, Tulga Öz F, Özkan P. Effect of toothbrushing on color changes of esthetic restorative materials. *J Esthet Restor Dent* 2015;27:S65–73. <https://doi.org/10.1111/jerd.12136>.
- [30] Kim HK, Kim SH, Lee JB, Han JS, Yeo IS. Effect of polishing and glazing on the color and spectral distribution of monolithic zirconia. *J Adv Prosthodont* 2013;5:296–304. <https://doi.org/10.4047/jap.2013.5.3.296>.
- [31] WANG L, GARCIA FCP, DE ARAÚJO PA, FRANCO EB, MONDELLI RFL. Wear Resistance of Packable Resin Composites after Simulated Toothbrushing Test. *J Esthet Restor Dent* 2004;16:303–14. <https://doi.org/10.1111/j.1708-8240.2004.tb00058.x>.

CHAPTER 4.

Influence of applied pressure and thickness variation on the bond strength between 3Y-TZP zirconia and self-adhesive resin cement

Published in Journal of Biomedical Materials Research Part B: Applied Biomaterials, Volume 113, March 2025, e35563

P. Pinto ^{a,b,c}, O. Carvalho ^{a,b,c}, R. Ferreira ^{a,b,c}, S. Madeira ^{a,b,c}, Filipe Silva ^{a,b,c}

^a Center for Micro-Electro Mechanical Systems (CMEMS-UMinho), University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

^b LABBELS –Associate Laboratory, Braga, Guimarães, Portugal

^c Department of Mechanical Engineering, University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

Abstract

This study evaluated the influence of applied pressure on the shear bond strength (SBS) between commercial 3Y-TZP zirconia and self-adhesive resin cement. Five test groups were created, each subjected to different applied pressures: 195.15 kPa, 97.47 kPa, 48.74 kPa, 9.75 kPa, and 0.20 kPa. A piston-type shear device was used to assess the effect of these pressures on cement layer thickness and SBS. The results demonstrated that the highest applied pressure (195.15 kPa), produced the thinnest cement layers ($36.60 \pm 1.67 \mu\text{m}$) and the highest SBS values ($58.8 \pm 1.6 \text{ MPa}$), whereas the lower applied pressure (0.20 kPa), resulted in the thickest cement layers ($73.20 \pm 1.64 \mu\text{m}$) and significantly lower SBS ($30.8 \pm 2.0 \text{ MPa}$). Regression analysis confirmed a strong correlation ($R^2 = 0.82$, $p < 0.05$) between cement thickness and SBS, indicating that cement thickness is a critical factor influencing adhesion. These findings highlight the importance of controlling applied pressure during cementation to optimize bonding performance. Standardizing pressure application through calibrated pressure devices may improve the durability of zirconia-based restorations and minimize variability during the cementation process.

Keywords: Zirconia; Shear Bond Strength; Self-Adhesive Resin Cement; Thickness; Applied pressure; Dental Restoration

4.1 Introduction

Dental restorations play a fundamental role in preserving oral health, preventing structural damage, and enhancing both the function and appearance of teeth. Due to the high aesthetic standards required in modern dentistry and the advancement of CAD/CAM technologies, which enable more precise and efficient fabrication of restorations, ceramics have gained significant prominence [1–6].

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) is a highly valued dental material, due to its flexural strength (900 to 1200 MPa) [7,8], biocompatibility [9–11], and aesthetic properties, particularly translucency [2,12,13], which enables a natural tooth-like appearance. This material is commonly used in the fabrication of dental restorations such as crowns, bridges, and implant abutments.

When applying a zirconia dental restoration, like a crown or bridge, onto a natural tooth or implant abutment, it is common practice to utilize a bonding agent to ensure a stable and strong bond. Self-adhesive resin cements are meticulously formulated to meet the requirements of

materials such as zirconia, assuring a resilient and enduring attachment [14,15]. According to the literature, self-adhesive resin cements are a popular choice for bonding zirconia restorations, as they offer a simplified and effective bonding approach, ensuring reliable adhesion [14,16,17]. However, achieving strong adhesion to zirconia remains challenging [18,19]. The high degree of surface energy of this ceramic can cause resin bonding agents to slide off or fail to adhere. Additionally, its smooth and dense surface structure can lead to limited mechanical interlocking with the bonding resin. The bond strength between self-adhesive resin cement and zirconia can be affected by several factors, such as how the surface is prepared, the way the cement is applied, and the properties of the materials used [20,21].

Several surface treatment techniques have been explored to enhance the bond strength between zirconia and resin cement, including acid etching, air-abrasion (sand and grit-blasting), laser treatments, plasma treatments, glazing, silanization among others [9,12,17,22–26].

Of the available surface treatments, grit-blasting with alumina (Al_2O_3) particles is one of the most commonly used methods [9,16,26,27]. In this process Al_2O_3 particles are blasted onto the zirconia surface under controlled pressure, distance, and period of time, modifying the surface roughness [16]. This technique enhances micromechanical retention, increases surface area, and improves the surface wettability [24].

In addition to surface treatments, the applied pressure during cementation plays a critical role in determining the stability of the bond between zirconia and resin cements. The pressure applied during the curing process influences cement layer thickness, adhesive penetration, and overall bond strength [3,15].

Kagaoan et al. conducted a systematic review on the factors influencing the bond strength of additively manufactured crown materials in dentistry. They analyzed variables such as airborne-particle abrasion, cement type, post-washing methods, bond strength testing methodology, and artificial aging effects [6]. However, their study did not address the influence of applied pressure during cementation, which remains an unstandardized variable in clinical practice and may significantly affect long-term adhesion. Maneenacarith et al. [16] reported that resin cement thickness significantly influenced the shear bond strength. In contrast, Petkosit et al. [3] found that pre-curing pressure (negative and positive) had no significant effect on the shear bond strength between zirconia and resin cement.

Despite advances in bonding protocols, the optimal applied pressure for zirconia restorations remains unclear [28]. Further research is needed to establish standardized pressure parameters,

as well as to investigate the combined effects of surface treatments and pressure variations on the bond strength of self-adhesive resin cements to zirconia.

This study aims to evaluate the influence of applied pressure and cement layer thickness on the shear bond strength (SBS) of self-adhesive resin cement to zirconia. Conversely to previous studies that set predefined cement thicknesses, this research uniquely isolates the effect of applied pressure as a controlling factor of cement thickness.

The null hypothesis of this study states that applied pressure and cement layer thickness have no significant effect on the shear bond strength between zirconia and self-adhesive resin cement.

4.2 Materials and methods

4.2.1 Specimen preparation

One hundred cylindrical specimens (N=100) were obtained from a commercial pre-sintered block 3Y-TZP zirconia (DD Bio ZWiso zirconia, Dental Direct, Germany). The specimens were machined using a DWX-50 milling machine (by Roland DG Corporation of Hamamatsu, Japan) with a 2 mm flat-end mill at a spindle speed of 25000 rpm. The feed rate was set to 900 mm/min in the X/Y directions and 300 mm/min in the Z direction with a layer depth of 0.8 mm. Table 1 presents the chemical composition of the commercial 3Y-TZP zirconia used in this study, as provided by the manufacturer.

Table 4.1 - Chemical composition of the commercial 3Y-TZP zirconia used in this study (wt%).

Component	ZrO₂+HfO₂+Y₂O₃	Y₂O₃	Al₂O₃	Other oxides
Composition (wt%)	≥ 99.0	< 6	<0.5	< 1.0

Cylinders were then sintered in a furnace (Zirkonofen 700 Ultra-Vakuum from Zirkonzahn) with the following sintering cycle: heating up to 900 °C at 8 °C/min with a holding time of 30 min., then heating up to the final temperature of 1450 °C at 3 °C/min with a holding time of 120 min., and finally cooling to 200 °C at 10 °C/min. After sintering, the samples resulted in cylinders with a diameter of 8 mm and a height of 3.2 mm. Subsequently, the surfaces of all samples intended for joining were polished using silicon carbide papers in the following sequence 600, 800, 1200,

2500 and 4000 grit, ensuring a uniform surface finishing across the samples. The cylinders production and polishing procedure are presented in Figure 4.1.



Figure 4.1 - Sequential depiction of the 3Y-TZP zirconia cylinders' production process, encompassing machining from a pre-sintered block, sintering under specified conditions, and subsequent polishing to achieve a smooth finish.

4.2.2 Surface treatment on zirconia surfaces

To improve adhesion between the zirconia and resin cement, all cylinders were abraded using grit-blasting with Al_2O_3 particles at 2 bar of pressure with a particle size of $50 \mu\text{m}$. The same distance of 15 mm was used for all specimens for 20 seconds. This surface treatment protocol was based on previous studies [6,17,27,29] which demonstrate its effectiveness in increasing surface roughness, thereby enhancing micromechanical interlocking and improving the wettability of the resin cement.

Following the surface treatment, the specimens were ultrasonically cleaned in isopropyl alcohol for 5 minutes to remove any residual particles. The treated surface roughness was assessed using a Mitutoyo SJ-210 profilometer (Mitutoyo Corporation, Japan), with five measurements taken per specimen. The arithmetic mean roughness (R_a) was recorded for surface characterization.

4.2.3 Physical bonding process to resin-matrix cement

The cylinders were divided into five groups ($n = 20$) according to the applied pressure used. Before the cementation procedure, all discs were ultrasonically cleaned in isopropyl alcohol for 5 minutes, and then a cleaning paste (Ivoclean; Ivoclar Vivadent AG) was applied to the bonding surface. For bonding, the self-adhesive and autopolymerizing resin cement SpeedCEM Plus (Ivoclar, Vivadent, Liechtenstein) was used, which is commonly employed for the cementation of high-strength, all-ceramic indirect restorations. The standard composition in wt% is shown in Table 2 based on the data provided by the manufacturer.

Table 4.2 - Standard composition (wt%) of the self-adhesive and autopolymerizing resin cement SpeedCEM Plus, as provided by the manufacturer (Ivoclar Vivadent, Liechtenstein)

Component	Standard Composition (wt%)	
	Base	Catalyst
Dimethacrylates	23.3	26.0
Ytterbium trifluoride	-	45.2
Co-polymer	-	22.6
Glass filler (base only), silicon dioxide	75.0	2.2
Adhesive monomer	-	3.1
Initiators, stabilizers and pigments	1.7	0.9

The application of this cement was strictly in accordance with the manufacturer's instructions, a standard approach confirmed by previous studies [14,30]. The entire bonding procedure was performed as schematized in Figure 4.2.

In step I, a zirconia cylinder was placed on a precision balance. Then, in step II, 0.03 g of self-adhesive resin cement was applied (a slightly excessive amount to ensure full coverage of the bonding surface) and another zirconia cylinder with the same surface treatment was placed on top of the first one. In step III, the assembled pair of cylinders was subjected to a controlled applied pressure in a custom-designed bonding device, which applied the predefined load for 2 minutes, ensuring consistent pressure application across all test groups.

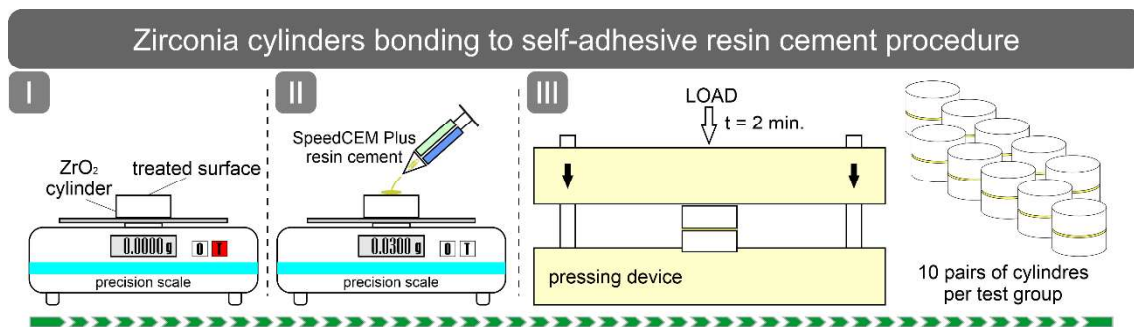


Figure 4.2 - Sequential representation of the bonding procedure, detailing the application of the self-adhesive resin cement and controlled assembly of the paired zirconia cylinders for shear bond strength testing.

Figure 4.3 illustrates the custom-designed bonding device for zirconia cylinders, composed of two cylindrical plates, each with a diameter of 100 mm. The lower plate is fixed at the base, while the upper plate is guided by three calibrated shafts housed in linear ball bearings, ensuring precise alignment and smooth movement. The device includes the following components: three end stoppers located at the top of the device, which act as movement limiters to ensure the correct distance so that the ΔL displacement is always the same; three calibrated shafts with a diameter

of 6 mm that provide alignment and stability to the device, allowing smooth movement of the upper plate; three ball bearings located at the base to reduce friction and ensure precise and uniform movement; the pair of zirconia cylinders positioned in the center between the upper and lower plates, for bonding; and a spring located in the center of the device, responsible for applying the required load to the zirconia cylinders. The specific load for each test group is determined by the spring constant ($F = k * \Delta l$) for a displacement of 15 mm, allowing for different loads to be applied precisely. For the minimal load condition, the applied pressure resulted solely from the weight of the upper zirconia cylinder, without additional force. After each bonding procedure, the excess resin was removed.

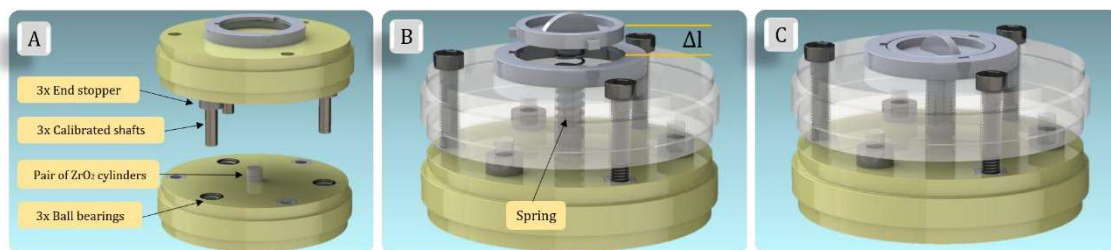


Figure 4.3 - Custom-designed bonding device for zirconia cylinders. (A) Components; (B) Unload position (C) Load position.

Following a one-hour post-preparation period, the adhered specimens were immersed in distilled water at 37°C, for 24 hours (for oral environment simulation) [9,19,31]. It is important to note that SpeedCEM Plus is a self-adhesive, autopolymerizing cement, meaning that the applied pressure was maintained during the initial curing phase. This differs from photopolymerized cements, where pressure is usually released before activation.

Each test group was composed of 10 pairs of cylinders, according to the applied pressure, as follows: G9.81, G4.90, G2.45, G0.49, and G0.01 (Table 4.3). The 'G' denotes the group designation, while the numerical values correspond to the applied force (N) during bonding (9.81 N, 4.90 N, 2.45 N, 0.49 N, and 0.01 N, respectively). These applied forces are within the range reported in previous studies [32,33] and are below 20 N, where it is noted that applying a constant force of this magnitude in clinical practice can result in operator discomfort and fingers pain [34].

Table 4.3 - Experimental groups categorized according to applied force (N) and corresponding applied pressure (kPa), calculated based on a constant contact area of 50.27 mm². Each group consisted of 10 pairs of zirconia cylinders ($n_1 = 10$).

Study group	Applied force (Newton)	Contact area (mm ²)	Applied pressure (kPa)	Pairs of cylinders Subtotal (n_1)
G9.81	9.81	50.27	195.15	10
G4.90	4.90	50.27	97.47	10
G2.45	2.45	50.27	47.74	10
G0.49	0.49	50.27	9.75	10
G0.01	0.01	50.27	0.20	10

This study focused on immediate bond strength evaluation, without incorporating long-term aging conditions such as thermocycling or prolonged water storage. Since self-adhesive resin cements undergo hygroscopic expansion over time, this phenomenon may influence long-term bond durability [35,36]. Future studies should explore how applied pressure interacts with the long-term performance of the zirconia-cement interface under hydrothermal aging conditions.

4.2.4 Characterization methods

4.2.4.1 Surface morphology characterization

Surface morphological analysis was analyzed using Scanning Electron Microscopy (SEM, Model JSM-6010 LV, JEOL, Japan). The resin cement thickness was determined through a cross-sectional SEM analysis. Thickness measurements were performed ImageJ software (National Institutes of Health, USA), with five measurements per condition. The mean and standard deviation were calculated for each group to quantify thickness variations under different applied pressures.

4.2.4.2 Shear bond strength test

Shear bond strength testing was conducted using a universal testing machine (Instron 8874, MA, EUA), equipped with a 25kN capacity load cell. Experiments were conducted using a custom-designed steel apparatus (Figure 4.4), analogous to the system described by Henriques et al. [37] and previously used in other studies to evaluate the shear bond strength of dental materials to zirconia [38,39].

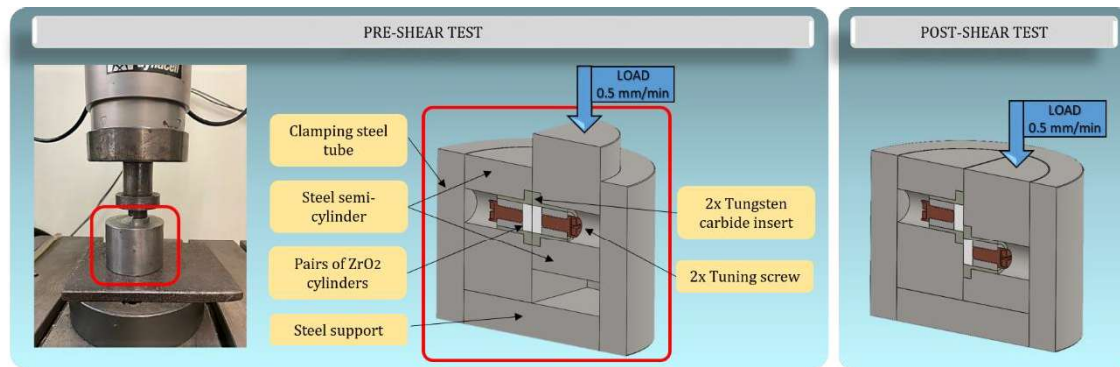


Figure 4.4 - Schematic of the shear test device

The testing apparatus consists of two semi-cylinders (radius: 15 mm). Each semi-cylinder has a hole fitted with a small, high-strength tungsten carbide insert, which holds the pair of zirconia cylinders to be tested. After precise alignment of the semi-cylinders and fine-tuning of the interface using the calibration screws, the assembly was inserted into the clamping steel tube and positioned on the steel support. The setup was then placed on the base of the universal testing machine, where a compressive force is applied at a constant speed of 0.5 mm/min until fracture or debonding occurred. Shear bond strength (MPa) was calculated as the maximum fracture load (N) divided by the cross-sectional area (mm²).

4.2.4.3 Statistical analysis

The null hypothesis stated that variations in applied pressure and cement layer thickness would have no significant effect on the shear bond strength of the zirconia-resin cement system.

To test this hypothesis, one-way analysis of variance (ANOVA) was performed, followed by Tukey's honestly significant difference (HSD) post-hoc test for multiple comparisons. Data normality was assessed using the Shapiro-Wilk test prior to ANOVA. A p-value < 0.05 was considered statistically significant. Additionally, a quadratic regression analysis was conducted to evaluate the relationship between cement layer thickness and shear bond strength (SBS). The coefficient of determination (R^2) was used to assess the goodness of fit of the model. The statistical significance of the regression model was evaluated using the F-test, and regression coefficients were analyzed to quantify the influence of cement thickness variation on SBS. All statistical analyses were conducted using IBM SPSS Statistics v.20 (IBM Corp., USA), and graphical representations were generated using OriginPro 2021 (OriginLab, USA).

4.3 Results

4.3.1 Surface and roughness characterization

In this study, the zirconia surface was treated using grit-blasting with Al_2O_3 particles ($50\text{ }\mu\text{m}$) at 2 bar of pressure to increase roughness and improve adhesion to the resin cement. This surface modification method is widely employed in adhesive dentistry to create a consistent and reproducible microtopography, which facilitates micromechanical interlocking and promotes stronger adhesive interactions.

Figure 4.5, presents scanning electron microscopy (SEM) images at magnification of x1000, x2000 and x10000, providing a detailed visualization of the surface morphology after grit-blasting. The treated surface exhibits randomly distributed peaks and valleys, characteristic of the abrasive impact of Al_2O_3 particles on the zirconia substrate.

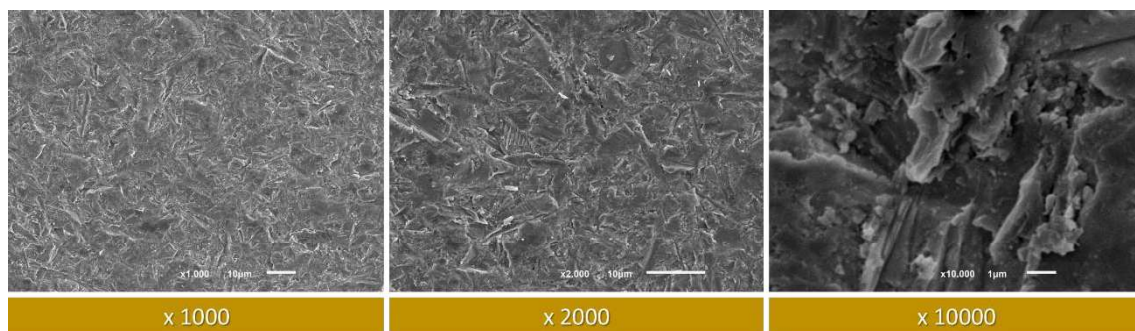


Figure 4.5 - Scanning electron microscopy (SEM) images of zirconia surfaces after grit-blasting with Al_2O_3 particles ($50\text{ }\mu\text{m}$) at 2 bar, at magnifications of x1000, x2000 and x10000.

Surface roughness analysis revealed an average roughness (R_a) of $0.471 \pm 0.005\text{ }\mu\text{m}$. The SEM images confirm that the roughened surface generates multiple micro-anchoring features, which are crucial for enhancing mechanical interlocking and optimizing adhesion to the resin cement.

4.3.2 Zirconia to resin-matrix cement interface thickness analysis

Cross-sectional SEM images of the zirconia-resin cement interface under different applied pressures are presented in Figure 4.6. These images clearly illustrate the variations in resin cement thickness as a function of the applied pressure, with quantitative thickness data provided below each image.

The applied pressure ranged from 195.15 kPa to 0.20 kPa, demonstrating a direct correlation between increased pressure and reduced cement layer thickness. The resin cement

layer appears homogeneously distributed across the interface in all samples, with no visible porosities, indicating a consistent and effective bonding process across different load conditions.

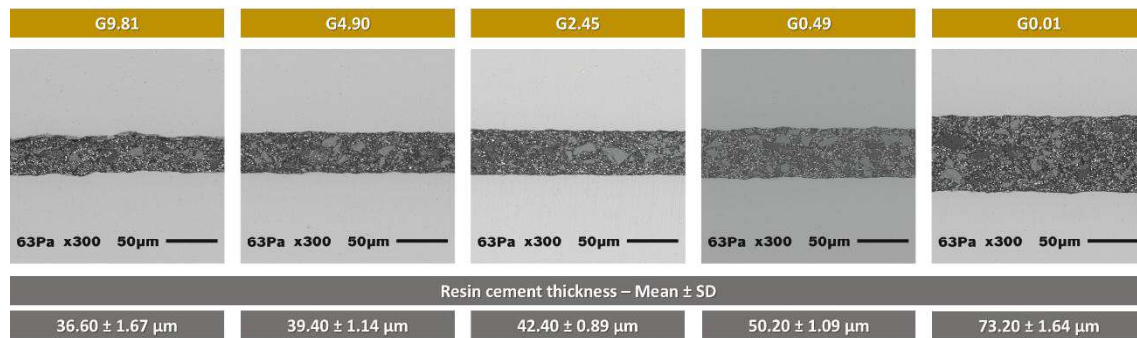


Figure 4.6 - Cross-Sectional scanning electron microscopy (SEM) images of zirconia-resin cement interface under different applied pressures. The quantitative thickness values (mean $\pm$ SD) are provided below each image.

The mean resin cement thickness and corresponding standard deviation (SD) are provided for each group in Figure 6. Group G0.01 (0.20 kPa), exhibited the greatest cement thickness (73.20 $\pm$ 1.64 μ m), whereas group G9.81 (195.15 kPa) showed the thinnest layer (36.60 $\pm$ 1.67 μ m). An expected trend was observed: higher applied pressure resulted in a thinner adhesive layer. When the applied pressure was reduced by half (from 195.15 kPa to 97.47 kPa and from 97.47 kPa to 48.74 kPa), the cement thickness increased by approximately 7.6 %, with group G4.90 showing an average resin cement thickness of 39.40 $\pm$ 1.14 μ m and group G2.45 having an average thickness of 42.40 $\pm$ 0.89 μ m. Group G0.49 exhibited an average cement thickness of 50.20 $\pm$ 1.09 μ m, further confirming the inverse relationship between applied pressure and cement layer thickness.

4.3.3 Shear Bond Strength

The shear bond strength (SBS) of zirconia bonded with self-adhesive resin cement was evaluated under different applied pressures, as presented in Figure 7. The group subjected to the highest applied pressure (G9.81: 195.15 kPa) exhibited the highest SBS value (58.8 $\pm$ 1.6 MPa). In contrast, the lowest SBS value was observed in G0.01 (0.20 kPa), which showed a statistically significant reduction ($p < 0.05$) compared to all other groups, registering an SBS of 30.8 $\pm$ 2.0 MPa. The results indicate a clear trend: as the applied pressure decreases from 195.15 kPa to any of the other applied pressures, the SBS values significantly decrease ($p < 0.05$), with measurements of 45.3 $\pm$ 1.6 MPa for G4.90, 40.3 $\pm$ 2.3 MPa for G2.45, and 38.8 $\pm$ 1.5 MPa for G0.49.

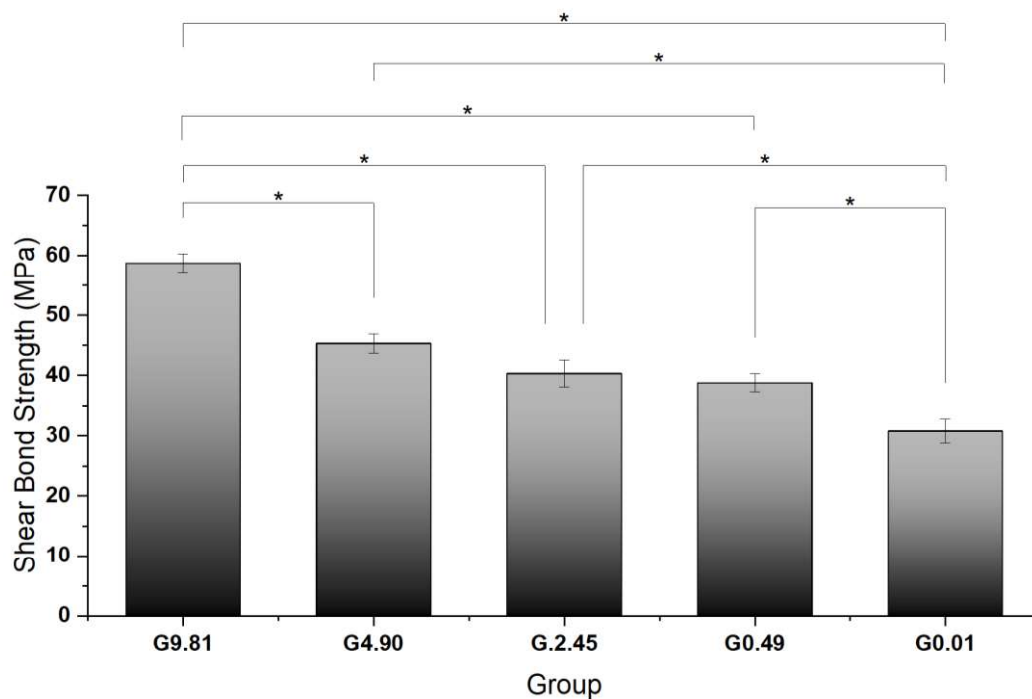


Figure 4.7 - Mean shear bond strength (SBS) and standard error of the mean (SEM) across different applied pressure conditions. The asterisks (*) indicate statistically significant differences ($p < 0.05$).

4.3.4 Correlation between cement layer thickness and shear bond strength

To further investigate the influence of cement layer thickness on shear bond strength (SBS), a quadratic regression analysis was performed. The results demonstrated a strong correlation between these variables ($R^2 = 0.82$, $p < 0.0001$), indicating that approximately 82% of the variation in SBS can be explained by the cement thickness. The regression equation obtained was: $SBS = 112.27 - 2.10 * t + 0.01 * t^2$, where t represents the cement layer thickness (μm). This model suggests that the relationship is not strictly linear, but rather follows a curvilinear trend, where SBS initially decreases with increasing cement thickness but start to stabilize at higher thickness values. The statistical significance of the model ($F(2,47) = 110.28$, $p < 0.0001$) reinforces the strength of this relationship. Figure 4.8 illustrates this correlation, showing the best-fit polynomial curve along with the experimental data points.

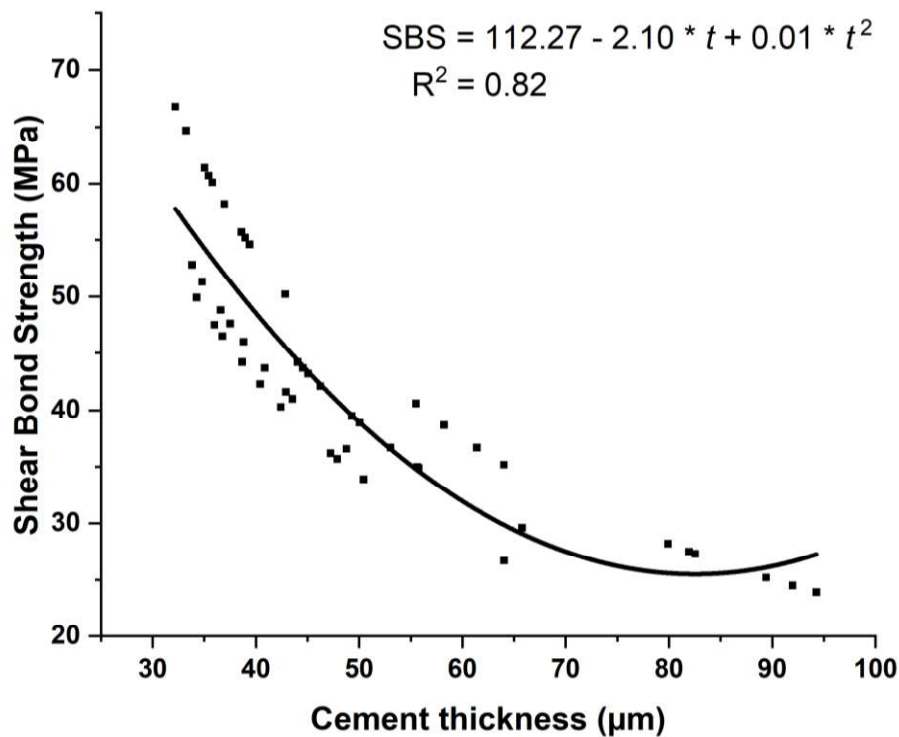


Figure 4.8 - Scatter plot showing the correlation between cement layer thickness (μm) and shear bond strength (MPa). The fitted quadratic regression curve ($R^2=0.82$) indicates a non-linear relationship.

4.4 Discussion

Based on the results obtained, it is evident that the bond strength between zirconia and resin cement is significantly influenced by the applied pressure ($p < 0.05$). Therefore, the null hypothesis of this study is rejected.

Regardless of the applied pressure all shear bond strength (SBS) values exceeded 20 MPa, a threshold considered sufficient for clinically reliable adhesion [20], demonstrating the effectiveness of air abrasion surface treatment.

The scanning electron microscopy (SEM) analysis revealed surfaces characterized by irregular peaks and valleys resulting from Al_2O_3 particle blasting, which significantly increases the available surface area for adhesion and provides additional mechanical anchorage. The observed average surface roughness ($0.471 \mu m$) are within the optimal range [$0.4 - 0.8 \mu m$] for enhancing wettability and cement adhesion facilitating better cement penetration into microgrooves and thereby improving bond effectiveness, as supported by previous studies [3,40,41]. This surface treatment technique is widely used [4,6,17,39,42] and provides very satisfactory results in improving adhesion compared to other conventional techniques.

The applied pressures tested (195.15, 97.47, 48.74, 9.75, and 0.20 kPa) were selected based on previous research investigating factors affecting the bond strength between zirconia and self-adhesive resin cement [4,14,34,43]. By isolating the effect of pressure, this research contributes a new perspective to the understanding of the zirconia-resin cement bonding process. From a clinical perspective, pressure values up to 0.4 MPa have been reported in studies related to the cementation of veneers and other anterior restorations [44]. Additionally, these values are considered clinically appropriate to avoid discomfort or pain, as excessive pressures during cementation can cause sensitivity issues, making this range both effective and comfortable for patients during procedures [34].

The results of the mean shear bond strength (SBS) between zirconia and self-adhesive resin cement reveal an expected trend: higher applied pressure leads to thinner resin cement layers and, consequently, higher SBS values. The authors believe that the thickness of the resin cement plays a crucial role in these outcomes, as it directly correlates with the SBS values obtained. The highest applied pressure resulted in the thinnest cement layers and the highest SBS values, while lower applied pressures progressively led to thicker layers and significantly reduced SBS values.

These findings align with the theoretical understanding that in thinner cement layers, typically in the 20-50 μm range, stress distribution along the adhesive interface tends to be more uniform compared to thicker layers [45]. This occurs because there is less material to deform, resulting in a more direct transmission of force between the substrates. With reduced thickness, the probability of concentrated tensile and shear stresses decreases, potentially enhancing shear resistance. Additionally, thinner layers typically exhibit fewer defects and porosities, resulting in a homogeneous material with fewer potential points of failure under load. This homogeneity also contributes to better marginal adaptation, reducing discontinuities between the cement and the substrate and improving intimate contact and adhesion across the interface [16].

Conversely, thicker cement layers introduce challenges such as a higher probability of stress concentration points, leading to crack formation and premature failure under shear loads. Additionally, they also have a higher propensity for internal porosities, which act as fracture initiators and weaken the adhesive bond. Poor marginal adaptation is an additional risk in thick layers due to the difficulty of cement flow, creating gaps that accumulate stress [46].

Maneenacarith et al. [16] investigated the influence of resin cement thickness on the shear bond strength (SBS) of the cement-zirconia interface, with predefined cement thicknesses of 50, 80, 160, and 240 μm , rather than as a result of an applied pressure as in our study. The results

exhibited a similar trend to our findings, with the 50 μm thickness showing significantly higher bond strength compared to the other thicknesses. However, the absolute SBS values obtained were approximately 50% lower than those in our study. This discrepancy may be attributed, among other factors, to the different shear testing methods used. While Maneenacarith et al. employed a knife-shaped shear blade that applied concentrated force at a single point, our study utilized a device that distributed the load more evenly across the interface, potentially leading to higher SBS values.

To further investigate the relationship between cement thickness and shear bond strength, a polynomial regression analysis was conducted. The results demonstrated a strong negative correlation ($R^2 = 0.82$, $p < 0.05$), indicating that 82% of the variation in SBS can be explained by cement thickness. This model highlights that SBS decreases as cement thickness increases; however, it is important to emphasize that this correlation was observed under the specific conditions tested in this study, which include the type of zirconia, the self-adhesive resin cement used, and its viscosity, as well as the methodology employed for cementation and shear bond strength testing. Different materials, variations in cement formulations, or alternative testing methodologies could potentially yield different correlations. Furthermore, an interesting observation in the regression model was the slight upward trend at higher thickness values. This behavior may be a limitation of the quadratic model, as theoretical expectations suggest that SBS should continue decreasing rather than stabilizing or increasing. This trend should be interpreted with caution, as it may not represent a real physical phenomenon but rather an adjustment limitation of the polynomial model used. Future studies should explore whether this trend persists experimentally or if an alternative regression model provides a better fit for predicting SBS based on cement thickness.

Additionally, the rheological properties of the resin cement may also play a role in this behavior. Self-adhesive resin cements are known to undergo hygroscopic expansion over time, which may alter the initial bond strength [35,36]. Although this study did not evaluate these effects, future studies should explore the interaction between applied pressure, cement viscosity, and hygroscopic expansion to determine their long-term impact on adhesion durability.

Furthermore, the assumption that maintaining uniform pressure throughout cementation improves adhesion must be critically evaluated. In clinical practice, while manual pressure is typically applied during seating, it is often released before photopolymerization in light-cured cements. In contrast, for self-adhesive, autopolymerizing cements, maintaining pressure during the

setting reaction may be beneficial [47–49]. Therefore, clinical protocols should consider the curing mechanism of the cement when deciding on the optimal pressure application technique.

From a clinical perspective, resin cement layers in the 20-50 μm range tend to provide more favorable stress distribution, improved marginal adaptation, enhanced adhesion, and superior mechanical properties compared to thicker layers [15]. Applying uniform pressure during the cementation of dental restorations is crucial for ensuring the integrity and durability of the bond. However, this must be interpreted with caution, as the pressure-release protocols vary depending on the curing mechanism of the cement used. In procedures like veneer cementation, even pressure is essential to prevent the formation of voids and variations in cement thickness, which can lead to concentrated stresses and compromise the bond strength. Studies have shown that manually applied pressure, often by the dentist's fingers, can vary significantly depending on individual technique and strength, potentially leading to inconsistencies in adhesion [47,50]. Moreover, the applied force can vary depending not only on the material of the veneer, such as porcelain, composite resin, or zirconia, but also on the specific technique used by the dentist. Different materials have varying mechanical properties, such as strength and brittleness, which necessitate tailored approaches to ensure optimal adhesion without damaging the restoration. For instance, porcelain veneers are generally less resistant than zirconia, requiring more careful pressure application to avoid fractures. It is also important to note that the initial manual pressure peak can quickly diminish, potentially leading to inadequate curing in areas with less contact, increasing the risk of bond failure [47]. To address these challenges, there is increasing interest in calibrated pressure devices, such as customized applicators and pressure-sensitive pads, which provide real-time feedback to standardize pressure during cementation. These devices help ensure consistent force, improving marginal adaptation, cement thickness, and bond strength while reducing operator variability [15,47]. However, widespread adoption depends on factors like cost, training, and material compatibility. As technology advances, these devices are expected to become more integrated into routine practice, potentially improving success rates in indirect restorations.

Although this study provides significant insights into the bond strength between zirconia and self-adhesive resin cement under different applied pressure, some limitations must be acknowledged. Shear tests were carried out to evaluate ceramic-to-ceramic adhesion, a method previously deemed appropriate for this purpose in other studies [38,39]. However, the shear device used in this study differs from the more commonly used knife-edge device, which applies concentrated force at a single point without a groove. Instead, our study employed a piston-type

shear device with half-loop loading, which helps to reduce stress concentration near the interface [39,51]. This improved loading condition may influence the results, making direct comparisons with other studies more difficult. Although the SBS values in this study are higher than those found in previous research, the focus should remain on the relative differences between the applied pressure conditions rather than absolute SBS values.

Additionally, this study did not include aging conditions such as long-term water storage or thermocycling, which are commonly used to assess long-term durability [3,17,43,52,53]. Future research should incorporate accelerated aging conditions to evaluate how applied pressure influences long-term bond degradation under clinically simulated conditions.

Finally, the composition of the self-adhesive resin cement used may have influenced the results, and future studies should evaluate whether different cement formulations exhibit similar trends under different applied pressures. Expanding the investigation to include varied clinical conditions and alternative adhesive systems may provide further advancements in adhesive dentistry.

4.5 Conclusions

This study demonstrated that applied pressure significantly influences the shear bond strength (SBS) between zirconia and self-adhesive resin cement. Higher applied pressures resulted in thinner cement layers and increased SBS, while lower pressures led to thicker layers and reduced adhesion. These findings reinforce the importance of controlling pressure during cementation to optimize bonding performance. Additionally, the regression analysis revealed a strong correlation between cement thickness and SBS, suggesting that cement thickness is a critical factor influencing adhesion. Future studies should explore the long-term durability of these bonds under aging conditions to better simulate clinical performance.

Acknowledgements

This work was supported by Fundação para a Ciência e Tecnologia (FCT, Portugal) through the grant SFRH/BD/145421/2019 (DOI: <https://doi.org/10.54499/SFRH/BD/145421/2019>) and national funds through the project FunFibRAI, PTDC/EME-EME/4197/2021 (DOI 10.54499/PTDC/EME-EME/4197/2021). Additional strategic funding was provided by CMEMS-UMinho, UIDB/04436/2020 (DOI 10.54499/UIDB/04436/2020), UIDP/04436/2020 (DOI

10.54499/UIDP/04436/2020), & LABBELS – Associate Laboratory in Biotechnology, Bioengineering and Microelectromechanical Systems, LA/P/0029/2020 (DOI 10.54499/LA/P/0029/2020).

Cofinanciado por:



References

- [1] Sulaiman TA, Altak A, Abdulmajeed A, Rodgers B, Lawson N. Cleaning Zirconia Surface Prior To Bonding: A Comparative Study of Different Methods and Solutions. *J Prosthodont* 2022;31:239–44. <https://doi.org/10.1111/jopr.13389>.
- [2] Ghozeizi R, Parsirad R, Tavakolizadeh S, Beyabanaki E. Effect of Different Nd:YAG Laser Power Outputs on Bond Strength of Resin Cement to Zirconia in Comparison to Sandblasting. *J Lasers Med Sci* 2021;12:1–6. <https://doi.org/10.34172/jlms.2021.06>.
- [3] Petkosit P, Sanohkan S. The Effect of Curing Pressure on Shear Bond Strength of Zirconia to Resin Cement. *Open Dent J* 2022;15:410–6. <https://doi.org/10.2174/1874210602115010410>.
- [4] Henriques B, Hammes N, Souza JCM, Özcan M, Mesquita-Guimarães J, Silva FS, et al. Influence of ns-Nd:YAG laser surface treatment on the tensile bond strength of zirconia to resin-matrix cements. *Ceram Int* 2020;46:27822–31. <https://doi.org/10.1016/j.ceramint.2020.07.281>.
- [5] Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. *J Prosthet Dent* 2007;98:379–88. [https://doi.org/10.1016/S0022-3913\(07\)60123-1](https://doi.org/10.1016/S0022-3913(07)60123-1).
- [6] Kagaoan Z, Liu X, Cameron A, Aarts J, Choi JJE. Factors influencing the bond strength of additively manufactured crown materials in dentistry: A systematic review of in vitro studies. *J Dent* 2024;144:104908. <https://doi.org/10.1016/j.jdent.2024.104908>.
- [7] Qeblawi DM, Muñoz CA, Brewer JD, Monaco EA. The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. *J Prosthet Dent* 2010;103:210–20. [https://doi.org/10.1016/S0022-3913\(10\)60033-9](https://doi.org/10.1016/S0022-3913(10)60033-9).
- [8] Akin H, Ozkurt Z, Kirmali O, Kazazoglu E, Ozdemir AK. Shear bond strength of resin cement to zirconia ceramic after aluminum oxide sandblasting and various laser treatments. *Photomed Laser Surg* 2011;29:797–802. <https://doi.org/10.1089/pho.2011.3039>.
- [9] Liu JF, Yang CC, Luo JL, Liu YC, Yan M, Ding SJ. Bond strength of self-adhesive resin cements to a high transparency zirconia crown and dentin. *J Dent Sci* 2022;17:973–83. <https://doi.org/10.1016/j.jds.2021.12.008>.
- [10] Koko M, Takagaki T, Abd El-Sattar NEA, Tagami J, Abdou A. MDP Salts: A New Bonding Strategy for Zirconia. *J Dent Res* 2022;101:769–76. <https://doi.org/10.1177/00220345211070758>.

- [11] Dantas T, Padrão J, da Silva MR, Pinto P, Madeira S, Vaz P, et al. Bacteria co-culture adhesion on different texturized zirconia surfaces. *J Mech Behav Biomed Mater* 2021;123. <https://doi.org/10.1016/j.jmbbm.2021.104786>.
- [12] Seo SH, Kim JE, Nam NE, Moon HS. Effect of air abrasion, acid etching, and aging on the shear bond strength with resin cement to 3Y-TZP zirconia. *J Mech Behav Biomed Mater* 2022;134:105348. <https://doi.org/10.1016/j.jmbbm.2022.105348>.
- [13] da Cruz MB, Marques JF, Fernandes BF, Pinto P, Madeira S, Carvalho Ó, et al. Laser surface treatment on Yttria-stabilized zirconia dental implants: Influence on cell behavior. *J Biomed Mater Res - Part B Appl Biomater* 2022;110:249–58. <https://doi.org/10.1002/jbm.b.34909>.
- [14] Sadid-Zadeh R, Strazzella A, Li R, Makwoka S. Effect of zirconia etching solution on the shear bond strength between zirconia and resin cement. *J Prosthet Dent* 2021;126:693–7. <https://doi.org/10.1016/j.prosdent.2020.09.016>.
- [15] Heboyan A, Vardanyan A, Karobari MI, Marya A, Avagyan T, Tebyaniyan H, et al. Dental Luting Cements: An Updated Comprehensive Review. *Molecules* 2023;28:1–16. <https://doi.org/10.3390/molecules28041619>.
- [16] Maneenacarith A, Rakmanee T, Klaisiri A. The influence of resin cement thicknesses on shear bond strength of the cement-zirconia. *J Stomatol* 2022;75:7–12. <https://doi.org/10.5114/jos.2022.114349>.
- [17] Khanlar LN, Takagaki T, Abdou A, Inokoshi M, Ikeda M, Takahashi A, et al. Effect of Air-Particle Abrasion Protocol and Primer on The Topography and Bond Strength of a High-Translucent Zirconia Ceramic. *J Prosthodont* 2022;31:228–38. <https://doi.org/10.1111/jopr.13372>.
- [18] Müller N, Al-Haj Husain N, Chen L, Özcan M. Adhesion of Different Resin Cements to Zirconia: Effect of Incremental versus Bulk Build Up, Use of Mould and Ageing. *Materials (Basel)* 2022;15:2186. <https://doi.org/10.3390/ma15062186>.
- [19] Sarıkaya I, Hayran Y. Adhesive bond strength of monolithic zirconia ceramic finished with various surface treatments. *BMC Oral Health* 2023;23:1–10. <https://doi.org/10.1186/s12903-023-03630-7>.
- [20] Papia E, Larsson C, Du Toit M, Von Steyern PV. Bonding between oxide ceramics and adhesive cement systems: A systematic review. *J Biomed Mater Res - Part B Appl Biomater* 2014;102:395–413. <https://doi.org/10.1002/jbm.b.33013>.
- [21] Obradovic-Djuricic K, Medić V, Dodić S, Gavrilov D, Antonijević D, Zrilić M. Dilemmas in zirconia bonding: A review. *Srp Arh Celok Lek* 2013;141:395–401. <https://doi.org/10.2298/SARH1306395O>.
- [22] Gerdzhikov I, Radeva E, Uzunov T. Comparative study of the bond strength of ZirCAD Multi and ZirCAD Prime ceramics with different types of cements. *Biotechnol Biotechnol Equip* 2023;37. <https://doi.org/10.1080/13102818.2023.2234501>.
- [23] Rohr N, Martin S, Zitzmann NU, Fischer J. A comprehensive in vitro study on the performance of two different strategies to simplify adhesive bonding. *J Esthet Restor Dent* 2022;34:833–42. <https://doi.org/10.1111/jerd.12903>.
- [24] Okutan Y, Gundogdu Y, Kilic HS, Kandemir B, Yucel MT. Femtosecond laser microstructuring of monolithic zirconia: Effects of laser parameters on resin bond strength. *Int J Appl Ceram Technol* 2022;19:2834–44. <https://doi.org/10.1111/ijac.14073>.
- [25] Quigley NP, Loo DSS, Choy C, Ha WN. Clinical efficacy of methods for bonding to zirconia: A systematic review. *J Prosthet Dent* 2021;125:231–40. <https://doi.org/10.1016/j.prosdent.2019.12.017>.

- [26] Moradi Z, Akbari F, Valizadeh S. Effects of Universal Adhesive on Shear Bond Strength of Resin Cement to Zirconia Ceramic with Different Surface Treatments. *Int J Dent* 2021;2021. <https://doi.org/10.1155/2021/5517382>.
- [27] 2022 - Effects of Hydrofluoric Acid Concentration and Etching Time on the Bond Strength to Ceramic-coated Zirconia.pdf n.d.
- [28] Özcan M, Volpato C. Adhesion to Zirconium Dioxide Used for Dental Reconstructions: Surface Conditioning Concepts, Challenges, and Future Prospects. *Curr Oral Heal Reports* 2015;1–5. <https://doi.org/10.1007/s40496-015-0071-x>.
- [29] Joukhadar C, Osman E, Rayyan M, Shrebaty M. Comparison between different surface treatment methods on shear bond strength of zirconia (in vitro study). *J Clin Exp Dent* 2020;12:e264–70. <https://doi.org/10.4317/jced.56242>.
- [30] Carrabba M, Nagasawa Y, Parrini S, Doldo T, Wood D, Ferrari M. Zirconia Translucency and Cement Systems as Factors Influencing the Zirconia-Titanium and Zirconia-Zirconia Shear Bond Strength. *Int J Oral Maxillofac Implants* 2019;34:1053–8. <https://doi.org/10.11607/jomi.7212>.
- [31] Delgado-Ruiz RA, Calvo-Guirado JL, Moreno P, Guardia J, Gomez-Moreno G, Mate-Sánchez JE, et al. Femtosecond laser microstructuring of zirconia dental implants. *J Biomed Mater Res Part B Appl Biomater* 2011;96B:91–100. <https://doi.org/10.1002/jbm.b.31743>.
- [32] Chai H, Kaizer M, Chughtai A, Tong H, Tanaka C, Zhang Y. On the interfacial fracture resistance of resin-bonded zirconia and glass-infiltrated graded zirconia. *Dent Mater* 2015;31:1304–11. <https://doi.org/10.1016/j.dental.2015.08.161>.
- [33] Lüthy H, Loeffel O, Hammerle CHF. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. *Dent Mater* 2006;22:195–200. <https://doi.org/10.1016/j.dental.2005.04.016>.
- [34] Chen B, Yang L, Lu Z, Meng H, Wu X, Chen C, et al. Shear bond strength of zirconia to resin: The effects of specimen preparation and loading procedure. *J Adv Prosthodont* 2019;11:313–23. <https://doi.org/10.4047/jap.2019.11.6.313>.
- [35] Collares FM, Leitune VCB, Augusto CR, Franken P, Samuel SMW. Swelling of self-adhesive resin cement increases long-term push-out bond strength of fiber post to dentin. *Brazilian J Oral Sci* 2015;14:246–50. <https://doi.org/10.1590/1677-3225v14n3a14>.
- [36] Kirsten M, Matta RE, Belli R, Lohbauer U, Wichmann M, Petschelt A, et al. Hygroscopic expansion of self-adhesive resin cements and the integrity of all-ceramic crowns. *Dent Mater* 2018;34:1102–11. <https://doi.org/10.1016/j.dental.2018.04.008>.
- [37] Henriques B, Soares D, Silva FS. Shear bond strength of a hot pressed Au-Pd-Pt alloy-porcelain dental composite. *J Mech Behav Biomed Mater* 2011;4:1718–26. <https://doi.org/10.1016/j.jmbbm.2011.05.029>.
- [38] Henriques B, Fabris D, Tuyama E, Mesquita-Guimarães J, Silva FS, Souza JCM, et al. Shear bond strength of PEEK and PEEK-30GF cemented to zirconia or titanium substrates. *J Adhes Sci Technol* 2019;33:1090–101. <https://doi.org/10.1080/01694243.2019.1565289>.
- [39] Santos RLP, Silva FS, Nascimento RM, Souza JCM, Motta F V., Carvalho O, et al. Shear bond strength of veneering porcelain to zirconia: Effect of surface treatment by CNC-milling and composite layer deposition on zirconia. *J Mech Behav Biomed Mater* 2016;60:547–56. <https://doi.org/10.1016/j.jmbbm.2016.03.015>.
- [40] Mohit KG, Lakha TA, Chinchwade A, Batul QA, Shaikh M, Kheur SM. Effects of surface modification techniques on zirconia substrates and their effect on bonding to dual cure resin cement - An in- vitro study. *J Indian Prosthodont Soc* 2022;22:179–87. https://doi.org/10.4103/jips.jips_298_21.

- [41] Sarmiento HR, Campos F, Sousa RS, Machado JPB, Souza ROA, Bottino MA, et al. Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia. *Acta Odontol Scand* 2014;72:346–53. <https://doi.org/10.3109/00016357.2013.837958>.
- [42] Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. *J Adhes Dent* 2015;17:7–26. <https://doi.org/10.3290/j.jad.a33525>.
- [43] Zaia WLS, de Figueiredo JLG, Leite LM, Satake A, Medeiros IS. Bond strenght of zirconia submitted to different surface treatments. *Pesqui Bras Odontopediatria Clin Integr* 2015;15:387–98. <https://doi.org/10.4034/PBOCI.2015.151.41>.
- [44] M A Muradov, A N Ryahovsky, V A Chkalin AAK. Study on the pressure on ceramic veneers during cementation. *Stomatologija (Mosk)* 2024;103:26–30.
- [45] Mounica Y, Behera S, Arunachalam S, Srikanth L, Deepthi MN, Susmita M. Influence of Different Cements and Cement Thickness on the Stress Distribution under Occlusal Surfaces of Porcelain-fused-to-metal and Porcelain-fused-to-zirconium Crowns: A Finite Element Analysis. *J Contemp Dent Pract* 2022;23:1224–9. <https://doi.org/10.5005/jp-journals-10024-3448>.
- [46] Sokolowski G, Krasowski M, Szczesio-Wlodarczyk A, Konieczny B, Sokolowski J, Bociong K. The influence of cement layer thickness on the stress state of metal inlay restorations—photoelastic analysis. *Materials (Basel)* 2021;14:1–11. <https://doi.org/10.3390/ma14030599>.
- [47] Zortuk M, Bolpaca P, Kilic K, Ozdemir E, Aguloglu S. Effects of Finger Pressure Applied By Dentists during Cementation of All-Ceramic Crowns. *Eur J Dent* 2010;04:383–8. <https://doi.org/10.1055/s-0039-1697857>.
- [48] Pedreira APRDV, D’Alpino PHP, Pereira PNR, Chaves SB, Wang L, Hilgert L, et al. Effects of the application techniques of self-adhesive resin cements on the interfacial integrity and bond strength of fiber posts to dentin. *J Appl Oral Sci* 2016;24:437–46. <https://doi.org/10.1590/1678-775720150600>.
- [49] Lino JSFB. “Cimentação Adesiva de Restaurações Cerâmicas 2016:118.
- [50] Black S, Amoores JN. Measurement of forces applied during the clinical cementation of dental crowns. *Physiol Meas* 1993;14:387–92. <https://doi.org/10.1088/0967-3334/14/3/018>.
- [51] Vásquez VZC, Özcan M, Kimpara ET. Evaluation of interface characterization and adhesion of glass ceramics to commercially pure titanium and gold alloy after thermal- and mechanical-loading. *Dent Mater* 2009;25:221–31. <https://doi.org/10.1016/j.dental.2008.07.002>.
- [52] Liu D, Pow EHN, Tsoi JKH, Matinlinna JP. Evaluation of four surface coating treatments for resin to zirconia bonding. *J Mech Behav Biomed Mater* 2014;32:300–9. <https://doi.org/10.1016/j.jmbbm.2013.12.011>.
- [53] Lümekemann N, Eichberger M, Stawarczyk B. Different surface modifications combined with universal adhesives: the impact on the bonding properties of zirconia to composite resin cement. *Clin Oral Investig* 2019;23:3941–50. <https://doi.org/10.1007/s00784-019-02825-z>.

C H A P T E R 5.

Can 3Y-TZP Zirconia reach 5Y-PSZ Zirconia translucency?

Submitted to Journal of Biomedical Materials Research Part B: Applied Biomaterials, April, 2025

P. Pinto ^{a,b,c}, O. Carvalho ^{a,b,c}, Filipe Silva ^{a,b,c}, S. Madeira ^{a,b,c}

a Center for Micro-Electro Mechanical Systems (CMEMS-UMinho), University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

b LABBELS –Associate Laboratory, Braga, Guimarães, Portugal

c Department of Mechanical Engineering, University of Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

Abstract

This study investigates whether 3Y-TZP zirconia can achieve the same translucency levels as 5Y-PSZ zirconia at different thicknesses, ranging from 1.00 mm to 0.05 mm. Three types of commercial zirconia ceramics with different Yttria contents were analysed: 3Y-TZP (3 mol% yttria), 4Y-PSZ (4 mol% yttria), and 5Y-PSZ (5 mol% yttria). Optical properties, including Translucency Parameter (TP), Contrast Ratio (CR), and light transmittance, were measured using standardized spectrophotometric techniques. Additionally, grain size measurements were performed using Scanning Electron Microscopy (SEM) to assess the microstructural contribution to light scattering and translucency. The results show that, at conventional thicknesses (≥ 0.45 mm), 3Y-TZP presents significantly lower translucency compared to 4Y-PSZ and 5Y-PSZ due to higher light scattering at its smaller grain boundaries. However, at ultra-thin thicknesses (≤ 0.15 mm), 3Y-TZP reaches or even surpasses the translucency levels of 5Y-PSZ, demonstrating that yttria content has a minimal effect on translucency at these dimensions. An unexpected flexible behaviour was also observed in 3Y-TZP at 0.05 mm, suggesting potential applications beyond dental ceramics. These findings indicate that 3Y-TZP zirconia can achieve comparable translucency to 5Y-PSZ at ultra-thin dimensions while maintaining superior mechanical properties, expanding its potential use in both dental and non-dental applications.

Keywords: Dental ceramics, Yttria-stabilized Zirconia, Ultra-thin zirconia, Translucency, Transmittance, Grain size.

5.1. Introduction

The evolution of dental ceramic materials, particularly zirconia, has marked a milestone in the field of restorative dentistry, offering aesthetic and durable alternatives for dental restorations [1,2]. These restorations are fabricated utilizing computer-aided design and computer-aided manufacturing (CAD/CAM) techniques, typically undergoing milling in a pre-sintered state followed by sintering in a high-temperature furnace [3]. CAD/CAM technology enhances the accuracy and fit of dental prosthetics [4], accelerates their production, and allows for customized designs tailored to each patient's unique dental structure [5], all while reducing material waste and improving efficiency [6].

The introduction of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has revolutionized the use of ceramics in dentistry, providing a metal-free, resilient, and tooth-coloured option [7,8]. However, balancing the mechanical and aesthetic properties, such as translucency and fracture resistance, remains a challenge [9–12]. Translucency is particularly critical for the aesthetic

success of dental restorations, as it influences how light is transmitted and reflected by the ceramic, closely mimicking the natural appearance of tooth enamel [13].

Initially, 3Y-YSZ zirconia, with 3 mol% yttria, provided high strength and fracture toughness through a stress-induced transformation-toughening mechanism [14], but its translucency was limited due to its polycrystalline structure. This made it more suitable for dental prosthesis frameworks that required veneering with feldspathic ceramic for enhanced aesthetics [11,15]. Yttria is incorporated into zirconia to stabilize high-temperature phases (tetragonal and cubic) at room temperature, thereby enhancing the material's mechanical properties and altering its optical characteristics. To overcome the limitation in translucency, newer generations of zirconia, such as 4YSZ (4 mol% yttria-stabilized zirconia) and 5YSZ (5 mol% yttria stabilized zirconia), were developed. These materials offer greater translucency but at the cost of reduced mechanical strength and fracture toughness [12,15]. This balance between translucency and mechanical properties underscores the importance of optimizing material thickness in clinical applications. Malallah et al [16] studied the impact of thickness on zirconia with 3 mol% yttria and found that reducing the thickness from 0.5 mm to 0.3 mm led to a decrease in fracture resistance by approximately 40%. Additionally, Souza et al [17] reported a clinical case where 0.3 mm thick zirconia veneers with 3 mol% yttria were used, and after one year of follow-up, they still exhibited good adhesion.

Other studies [18–20] suggest that thicknesses between 0.8 and 1.5 mm are necessary for dental restorations using 4YSZ and 5YSZ zirconia. The challenge lies in balancing the mechanical integrity of zirconia with the need for thin restorations to achieve the desired optical properties.

The aesthetic properties of zirconia are assessed not only by its translucency but also by its ability to replicate the natural colour of teeth. The Munsell colour space and the CIELAB system are used to define and compare the colour properties of dental materials, including zirconia. Translucency is quantified by the translucency parameter (TP) and the contrast ratio (CR). TP measures the degree of translucency by quantifying the colour difference of a material over black and white backgrounds, while CR assesses the opacity of the material [10,21,22]. These parameters provide a standardized approach to evaluating and comparing the aesthetic properties of dental ceramics, particularly regarding how light interacts with the material.

Previous research has shown that the translucency of zirconia is influenced by several factors, including material thickness [12,15,23,24], grain size [12,25–27], yttria content [17,23,28], and sintering conditions [11,29]. As the thickness of zirconia increases, its

translucency decreases, because thicker materials allow less light to pass through, leading to a more opaque appearance. Smaller grain sizes generally scatter less light, resulting in higher translucency [30]. Yttria content, also plays a significant role: higher yttria concentrations, such as in 4YSZ and 5YSZ, can increase translucency by stabilizing the cubic phase of zirconia, which is less opaque than the tetragonal phase. However, this increase in translucency often comes at the expense of mechanical strength [25]. Sintering conditions, including temperature and duration, further affect translucency. Higher sintering temperatures and longer durations typically promote more complete densification, which can reduce internal defects or porosity and, in some cases, improve translucency. However, this densification often leads to larger grain sizes, which generally reduce translucency due to increased light scattering, while also decreasing the material's mechanical strength [11,21,30].

This investigation aims to address a gap in the existing literature by systematically exploring the translucency of 3Y-TZP, 4YSZ, and 5YSZ zirconias across various thicknesses, including standard sizes of 1 and 0.45 mm, as well as ultra-thin sizes of 0.3, 0.15, and 0.05 mm. Particular emphasis is placed on the latter two thicknesses, which have not previously been attainable or reported in clinical practice. This work seeks to challenge preconceived notions about the aesthetic limitations of 3Y-TZP and to provide a basis for future innovations in dental material science.

5.2 Materials and Methods

5.2.1 Specimens preparation

For this study, three pre-sintered commercial zirconia blocks with varying degrees of translucency were used, namely a 3Y-TZP Bio ZW iso Shade White from Dental Direkt, Germany; a 4Y-PSZ Priti Multidisc Extra Translucent SHADE: A Light Multicolor; and a 5Y-PSZ Priti Multidisc High Translucent SHADE: A Light Multicolor, both purchased from Pritidenta, Germany, indicating higher levels of translucency. Table 5.1 summarizes the chemical composition (specific elements and their weight percentages) and correspondent mechanical properties of different zirconia types (3Y-TZP, 4Y-PSZ, 5Y-PSZ) as provided by the manufacturers.

Table 5.1 - Summary of the chemical composition (wt%) and mechanical properties of Zirconia materials used (3Y-TZP, 4Y-PSZ, 5Y-PSZ) (Manufacturer-provided data).

Chemical Composition (wt%)					Mechanical Properties	
Zircónia	ZrO ₂ + HfO ₂	Y ₂ O ₃	Al ₂ O ₃	Other Oxides	Fracture toughness (MPa√m)	Bending strength (MPa)
3Y-TZP	≥ 93.0	< 6.0	< 0.5	< 1.0	> 9.5	1150±200
4Y-PSZ	92.1 - 90.7	6.6 - 7.9	< 0.2	< 0.7	-	> 1150
5Y-PSZ	89.9 - 90.7	8.6 - 10.1	< 0.2	< 0.7	-	> 650

For each type of zirconia block, three independent specimens were produced for each thickness using CAD/CAM technology. The specimens were then sintered in a Zirkonofen 700 Ultra-Vakuum furnace (Zirkonzahn) following the manufacturer's specified instructions as outlined in Table 5.2. The final dimensions of each sample were a diameter of 13 mm and thicknesses across five different measurements: 1.00 mm, 0.45 mm, 0.30 mm, 0.15 mm, and 0.05 mm. For the thinnest samples, those less than 0.45 mm, a patented CAD/CAM machining method was employed to achieve ultra-thin thicknesses. All samples were polished on the both sides with water cooling with 600, 1200 and 2400 grit sandpaper and thicknesses were verified with a digital caliper. A tolerance of ± 0.01 mm was considered for all thicknesses. The schematic of the experimental procedure is shown in Figure 5.1

Table 5.2 - Manufacturer's specified sintering instructions for different types of zirconia blocks.

Zirconia	Heating	Dwell	Heating	Dwell	Cooling
3Y-TZP	900 °C 8 °C/min	30 min	1450 °C 3 °C/min	120 min	Room Temp. 10 °C/min
4Y-PSZ	1450 °C 10 °C/min	120 min	-	-	
5Y-PSZ	1450 °C 10 °C/min	120 min	-	-	

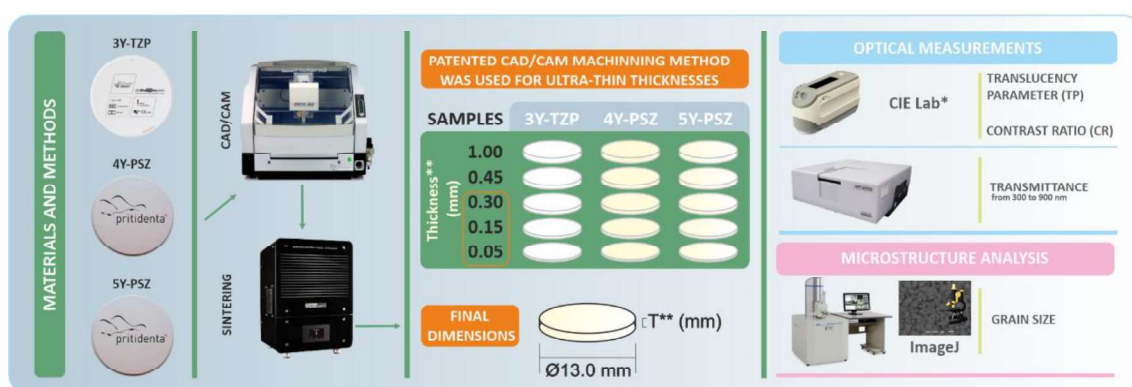


Figure 5.1 - Schematic representation of the experimental procedure used in this study: samples production with different thicknesses, and optical and microstructure analysis.

A nomenclature system was designed to facilitate sample identification, categorized by the type of Zirconia (3Y, 4Y and 5Y and respective thicknesses (1, 0.45, 0.30, 0.15 and 0.05). Details of this nomenclature are presented in Figure 2.

SAMPLES		3Y-TZP	4Y-PSZ	5Y-PSZ
Thickness (mm)	1.00	3Y ₁	4Y ₁	5Y ₁
	0.45	3Y ₀₄₅	4Y ₀₄₅	5Y ₀₄₅
	0.30	3Y ₀₃₀	4Y ₀₃₀	5Y ₀₃₀
	0.15	3Y ₀₁₅	4Y ₀₁₅	5Y ₀₁₅
	0.05	3Y ₀₀₅	4Y ₀₀₅	5Y ₀₀₅

Figure 5.2 - Nomenclature system for sample identification, categorized by Zirconia type and respective thicknesses.

5.2.2 Optical measurements

Three measurements per condition of the optical properties were conducted using a portable spectrophotometer (CM-2600d, Konica Minolta, Tokyo, Japan), in accordance with the Commission Internationale de l'Eclairage Lab* (CIE Lab*) colour system (Figure 1). The CIE Lab* system is a colorimetric method that quantifies colour using three coordinates: L^* , indicating lightness from dark to light; a^* , denoting the red to green spectrum; and b^* , representing the blue to yellow spectrum. This method provides a comprehensive representation of colour that is consistent with human vision [31]. The measurements were conducted under standardized D65

light illumination, which represents average daylight, including the ultraviolet wavelength region, and is recommended according to ISO 3664:2000 [31,32]. A 10° observer angle was set to replicate typical viewing conditions. The spectrophotometer settings were adjusted to a small aperture size (SAV), allowing for precise measurement of small areas and reducing the influence of surface irregularities. Additionally, the specular component was excluded (SCE mode), minimizing the effects of surface gloss and providing a more accurate representation of the material's intrinsic colour characteristics [31,33]. Before each measurement, the equipment was calibrated according to the manufacturer's instructions using the white calibration standard.

The translucency parameter (TP) was calculated using Equation 1 [10,11,26,28], where the sub-indices w and b correspond to measurements taken against white and black backgrounds, respectively. The TP formula is expressed as:

$$\text{Eq. (1)} \quad \text{TP} = [(L_w^* - L_b^*)^2 + (a_w^* - a_b^*)^2 + (b_w^* - b_b^*)^2]^{1/2}$$

Contrast Ratio (CR), a measure frequently utilized to describe the opacity of ceramics [28], was determined based on Equation 2. CR is calculated from the illuminance ratio, comparing readings taken on black (Yb) and white (Yw) backgrounds. The equation for CR is given as [11,28]:

$$\text{Eq. (2)} \quad \text{CR} = \frac{Y_b}{Y_w}$$

where Y is defined as:

$$Y = \left(\frac{L + 16}{116} \right)^3 \times 100$$

This equation accounts for the luminance factor, transforming it into a value that reflects the material's opacity or transparency relative to the specified backgrounds, with 1.0 representing a completely opaque material and 0.0 denoting full transparency [28,30].

Transmittance measurements were conducted to determine the amount of light passing directly through the zirconia samples [34]. These measurements were performed across a spectral range of 400 to 800 nm, using a Shimadzu UV-2450 PC spectrophotometer. The samples were placed in front of the detector in a black polymeric sample holder, with the incident light perpendicular to the sample surface.

5.2.3 Microstructure analysis/Grain Size

The microstructure of all polished samples was analyzed using Scanning Electron Microscopy (SEM), specifically with a JSM-6010 LV, JEOL model. The samples were examined at magnifications of 5,000x and 10,000x. No thermal or chemical etching was performed prior to imaging, and the samples were not sputtered with a metallic coating, as the SEM model employed allows imaging of non-conductive samples with minimal charging effects. Grain size measurements were conducted on the SEM micrographs at 10,000x magnification using the ImageJ software. Initially, the images were calibrated by setting the scale, and then the grain sizes were measured employing the linear intercept method (ASTM E112 2013) [10,27,35]. The analysis was performed using 15 horizontal and 15 vertical. No correction factor was applied, as the grain morphology varied significantly between different zirconia types. The 3Y-TZP zirconia presented equiaxed and more uniform grains, justifying the use of a correction factor if analyzed separately. However, the 4Y-PSZ and 5Y-PSZ had more irregular and elongated grains, making the use of the same correction factor inappropriate, as it would distort the actual grain size values and mask the true microstructural differences between these materials [36].

5.3 Results and Discussion

5.3.1 Optical properties and translucency behaviour

Figure 3 provides a qualitative perspective on the relationship between the 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconia specimens studied and their translucency characteristics. As previously noted, translucency in zirconia materials is directly influenced by a reduction in thickness.

As Figure 5.3 illustrates, at a thickness of 1 mm, all three zirconia types—3Y-TZP, 4Y-PSZ, and 5-PSZ—display a uniformly opaque appearance, reflecting the inherent limitations of light transmission through thicker ceramic layers, even in zirconias with higher yttria (Y_2O_3) content. The opacity at this thickness is consistent with the expected behaviour, where light scattering at the grain boundaries and internal defects of material are the main factors that contribute to the reduction in overall translucency. As the thickness decreases to 0.45 mm or 0.30 mm, and more notably to 0.15 mm and 0.05 mm, a significant increase in translucency is observed across all zirconia types, evidenced by the enhanced visibility of the black horizontal lines. At these ultra-thin dimensions (0.15 and 0.05 mm), the translucency levels of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ begin to

converge, reaching similar values. This convergence suggests that when the material's thickness is reduced, the distance that light has to travel becomes shorter and the number of grain boundaries that light encounters is significantly higher, thereby reducing the cumulative scattering effects that typically differentiate these materials at greater thicknesses. Additionally, this observation suggests that at these thin thickness levels, the differences in yttria (Y_2O_3) content, which affect the degree of cubic phase stabilization and consequently the translucency of zirconia [37], become less significant. Similar observations were found in a study by Tong et al. [8], where the translucency of YSZ ceramics also significantly decreased with the increase of the thickness. In the same study, all tested ceramics exhibited opacity at 1 mm thickness, which is aligned with the qualitative findings of the present study.

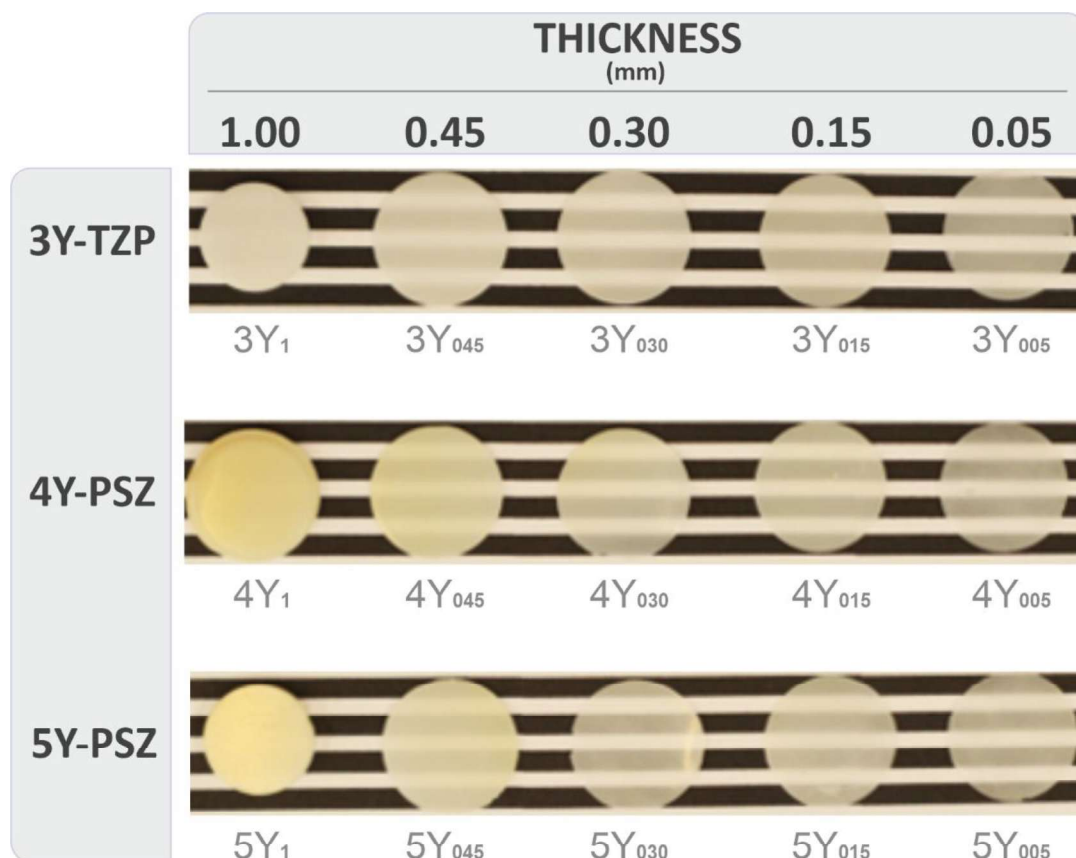


Figure 5.3 - Visual Representation for Qualitative Analysis of Translucency in 3Y-TZP, 4Y-PSZ, and 5Y-PSZ Zirconia Specimens. At thickness levels of 1.00, 0.45, 0.30, 0.15, and 0.05 mm, highlighting the observable translucency variations.

Figure 5.4 and Figure 5.5 depict the quantitative analysis of translucency, represented by the Translucency Parameter (TP) and the Contrast Ratio (CR), respectively. The results reveal significant trends depending on the thickness and composition of the evaluated zirconias. As

expected, TP decreases by increasing sample thickness. Considering the same thickness, TP increases with the increase of the yttria (Y_2O_3) content in the zirconia. Conversely, and as expected, CR follows the opposite trend regarding both thickness and yttria content. This behaviour is aligned with previous studies [38–43] which also reported a reduction in translucency with increasing thickness in dental ceramic materials.

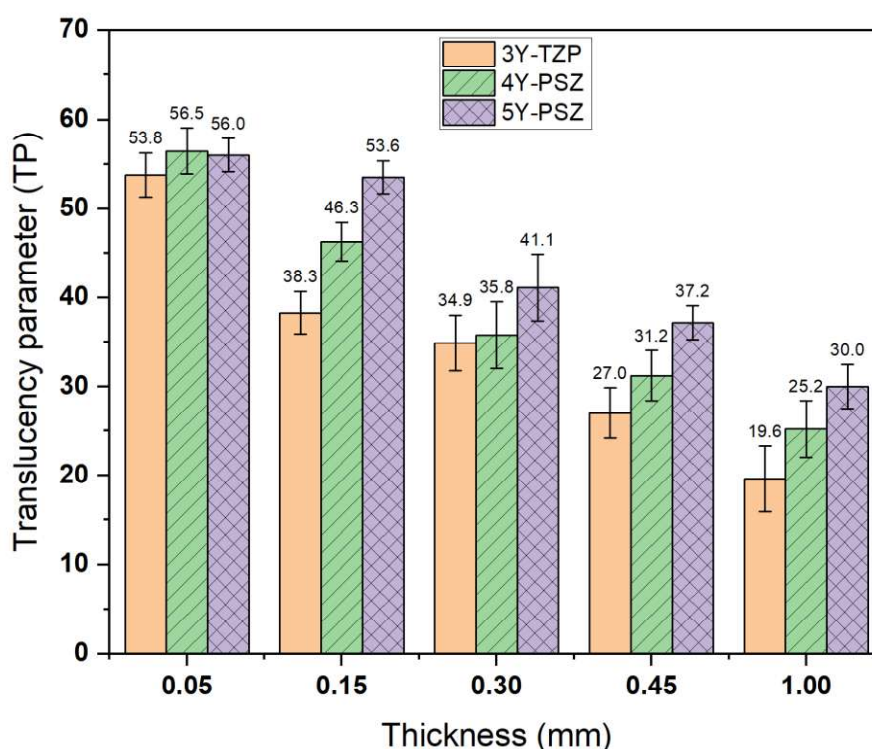


Figure 5.4 - Translucency parameter (TP) as a function of ceramic thickness (mm) for three types of zirconia in this study: 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. Error bars represent the standard deviation (SD) from three independent specimens, with three measurements per specimen.

At thicknesses greater than 0.05 mm, the influence of yttria content on translucency becomes evident. For example, at a thickness of 1.0 mm, the TP values are 19.6 ± 3.7 , 25.2 ± 3.2 , and 30.0 ± 2.5 for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, respectively. As the thickness decreases to 0.45, 0.30, and 0.15 mm, the TP values increase progressively for all zirconia types, clearly demonstrating that a higher yttria content results in greater translucency at each of these thickness levels. This trend is consistent with other studies [16,23,38] and is explained by the stabilization of the cubic phase of zirconia, whose optical properties are superior compared to the tetragonal

phase. An increase on the on the cubic phase reduces light scattering, enhancing optical transmission through the material and thus, promoting higher translucency [28].

On the other hand, it is noticeable that, at the ultra-thin thickness of 0.05 mm, the difference in translucency between the different types of zirconia (3Y-TZP, 4Y-PSZ, and 5Y-PSZ) becomes less pronounced. Under these conditions, the TP values reached a maximum of 53.8 ± 2.5 , 56.4 ± 2.6 , and 56.0 ± 1.9 for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, respectively. These results suggest that, at extremely reduced dimensions, the effect of yttria content on translucency is minimal and this phenomenon can be explained by the significant reduction in the optical path through the material, which minimizes light scattering at the grain boundaries, thus reducing the influence of microstructure on translucency. This observation has not been previously reported in the literature, possibly due to the technical challenges of producing samples with a thickness of 0.15 mm or less, where this work stands out as a differentiating factor.

Another important finding from the results is that, from a thickness of 0.15 mm, 3Y-TZP zirconia achieves translucency levels nearly equal to or higher, than those of zirconias with higher yttria content (4Y-PSZ and 5Y-PSZ) at thicknesses greater than 0.30 mm. This observation is highly relevant, particularly in certain clinical cases, where the more resistant zirconia (3Y-TZP) can be used with the same level of translucency as zirconias with a higher cubic phase content, which are typically less resistant.

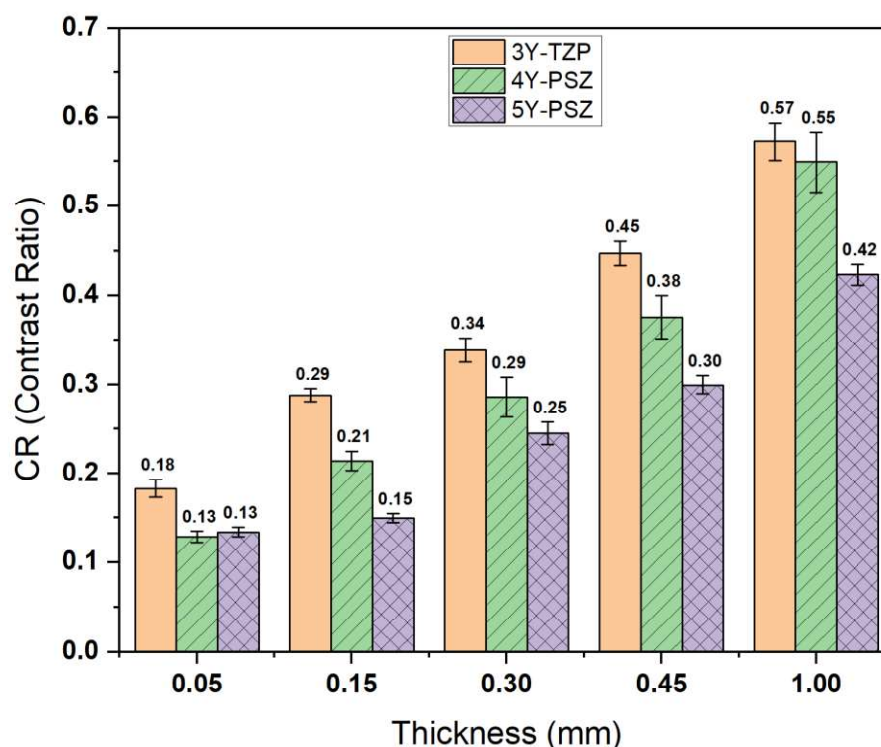


Figure 5.5 - Contrast Ratio (CR) as a function of ceramic thickness (mm) for three types of zirconia in this study: 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. Error bars represent the standard deviation (SD) from three independent specimens, with three measurements per specimen.

In contrast to the TP values, the Contrast Ratio (CR) values (presented in Figure 5) decrease as thickness increases. For the same thickness, the CR increases as the yttria content in the zirconia decreases. For example, the maximum CR values are observed at a thickness of 1.0 mm, with 0.57 ± 0.02 , 0.55 ± 0.03 , and 0.42 ± 0.01 for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, respectively. These results are in accordance with similar studies [11,12,44,45] found in literature. Conversely, the minimum values are observed at the ultra-thin thickness of 0.05 mm, with 0.18 ± 0.01 , 0.13 ± 0.01 , and 0.13 ± 0.01 for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, respectively. At this ultra-thin thickness, the yttria content in the 3Y-TZP zirconia sample still seems to have some relevance, increasing its opacity compared to the 4Y-PSZ and 5Y-PSZ samples. Since the CR is a measure that directly depends on the material's ability to block or absorb light [45,46], even at ultra-thin thicknesses such as 0.05 mm, the yttria content still plays an important role in opacity. This effect is due to the influence of yttria on grain size and material density: lower yttria content (as in 3Y-TZP) results in

smaller grains and higher density, which increase light scattering and overall opacity, while higher yttria content (in 4Y-PSZ and 5Y-PSZ) leads to larger grains and lower density, allowing more light transmission [38,47,48].

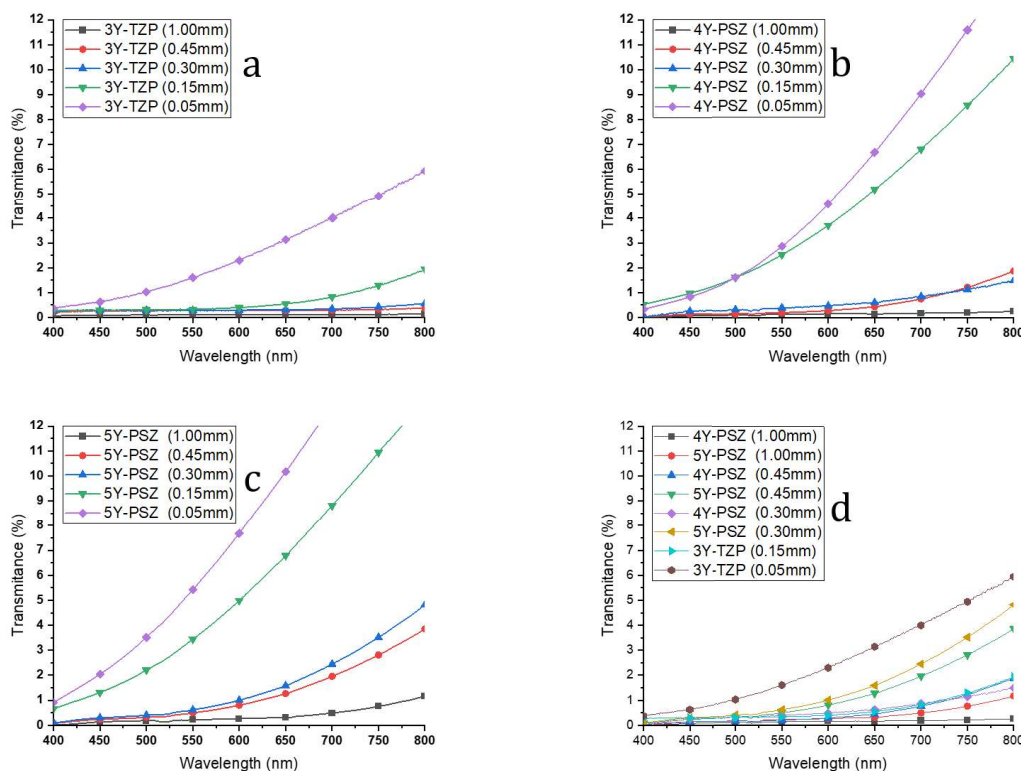


Figure 5.6 - Light transmittance through zirconia ceramics as a function of thickness (0.05 to 1.00 mm) for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconias. Figures 6a, 6b, and 6c illustrate the transmittance behaviour of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ at various thicknesses, respectively. Figure 6d compares the transmittance values of 3Y-TZP (at 0.15 mm and 0.05 mm) with 4Y-PSZ and 5Y-PSZ at higher thicknesses (0.30 mm, 0.45 mm, and 1.00 mm).

Figure 5.6a, 5.6b, and 5.6c illustrate the optical transmittance behaviour at various thicknesses (1.00 mm, 0.45 mm, 0.30 mm, 0.15 mm, and 0.05 mm) for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconia, respectively. For easier visual comparison, all graphs in Figure 6 (a, b, c, and d) maintain the same scale for transmittance percentage and wavelength. The analysis of these graphs supports the TP and CR results, showing a significant increase in light transmission as thickness decreases for all types of zirconia. This behaviour is consistent with the Beer-Lambert Law, which states that the decrease in light intensity is exponentially related to the material's thickness and absorption coefficient [49]. For 3Y-TZP zirconia (Figure 6a), at thicknesses of 1 mm, 0.45 mm, and 0.30 mm, the percentage of transmitted light is almost zero, which can be explained

by the anisotropy of tetragonal phase. The tetragonal grains present different crystallographic orientations, causing a discontinuity in the refractive index at grain boundaries, resulting in a significant reduction in light transmittance [12,28]. For the ultra-thin thicknesses of 0.15 and 0.05 mm, the transmittance increases sharply, which is consistent with what was observed for the TP and CR values. This behaviour can again be explained by the reduced material thickness, which shortens the optical path length and thus, results in lower light scattering at the grain boundaries. The behaviour of 4Y-PSZ zirconia (Figure 5.6b) also follows the trend of increasing transmittance as thickness decreases, and compared to 3Y-TZP, it shows higher transmittance at all thicknesses, particularly at the ultra-thin thicknesses of 0.15 and 0.05 mm. In terms of the 5Y-PSZ zirconia (Figure 6c), which has the highest yttria content, it exhibits the highest transmittance values, which is also in agreement with the TP and CR results already discussed.

The graph in Figure 5.6d aims to compare at which thickness levels 3Y-TZP zirconia can achieve transmittance similar to 4Y-PSZ and 5Y-PSZ zirconia. This graph shows the results for the 1.00 mm, 0.45 mm, and 0.30 mm thicknesses of 4Y-PSZ and 5Y-PSZ zirconia, while also positioning the thinner 0.15 mm and 0.05 mm thicknesses of 3Y-TZP zirconia. It can be observed that for greater thicknesses (1.00 mm and 0.45 mm), both 4Y-PSZ and 5Y-PSZ present very low transmittance values, reinforcing the previously discussed results on the influence of thickness and composition on the optical properties. This trend is aligned with similar studies [8,13,34] that reported near-zero transmittance levels for YSZ with a thickness of 1.00 mm, observing an increase in the percentage of incident light that passes through the sample as thickness decreases. On the other hand, there are studies [50,51] that obtained transmittance percentages approximately 20% higher than ours, which could be due to differences in material density and porosity [8,40,51].

The most interesting behaviour emerges at the ultra-thin thicknesses of 0.15 mm and 0.05 mm for 3Y-TZP zirconia, where it achieves transmittance levels similar to or even higher than those of 4Y-PSZ and 5Y-PSZ zirconia at thicknesses of 0.30, 0.45, and 1.00 mm, despite its lower yttria content. This result challenges the idea that a lower amount of yttria is always associated with lower transmittance.

Although 3Y-TZP zirconia, predominantly composed of the tetragonal phase, is susceptible to low-temperature degradation (LTD) or hydrothermal aging—resulting in a transformation to the monoclinic phase [12] its superior mechanical strength and fracture toughness make it a viable option for thin restorations compared to higher-yttria zirconias. While ultra-thin thicknesses such

as 0.15 mm and 0.05 mm are not yet standard in dental practice, they serve as a proof-of-concept for exploring the boundaries of translucency and mechanical performance in zirconia ceramics. Future advancements in CAD/CAM technology and adhesive systems could expand their applicability in cases requiring minimal or no tooth reduction.

5.3.2 Microstructural characterization and grain size impact

A grain size analysis was also performed, as it is one of the variables directly related to the translucency of dental ceramic materials [8,12,30,44,52]. The graph in Figure 5.7 shows the average grain size for the different zirconias (3Y-TZP, 4Y-PSZ, and 5Y-PSZ) at various thicknesses (0.05 mm, 0.15 mm, 0.30 mm, 0.45 mm, and 1.00 mm). From the results, the analysis of grain sizes for different yttria concentrations (3Y, 4Y, and 5Y) shows a progressive increase in grain size with higher yttria content across all thicknesses. This reflects the stabilizing effect of yttria on the cubic phase of zirconia, promoting grain growth during sintering. The increase in grain size is consistent regardless of thickness, indicating that yttria content is the primary factor influencing microstructure. Larger standard deviations within the same yttria content group suggest that thickness alone has less impact on grain size. This aligns with findings in the literature, where a higher fraction of the cubic phase (stabilized by yttria) reduces grain boundary energy, facilitating grain coalescence and growth [27,30,38,52].

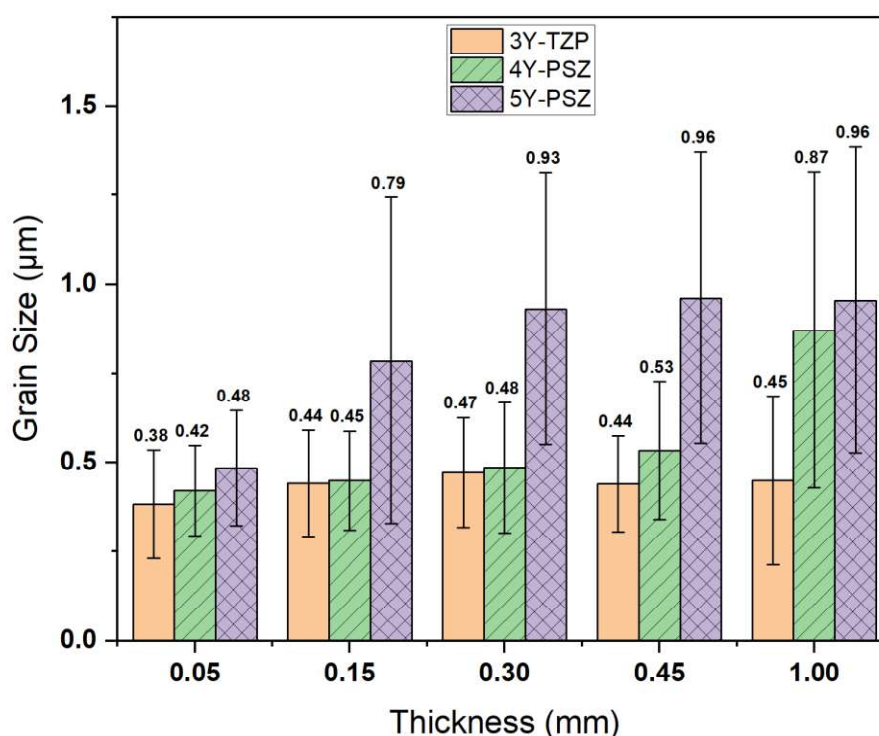


Figure 5.7 - Grain size (μm) as a function of thickness (mm) for 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconias. Error bars represent the standard deviation (SD)

As observed in Figure 5.7, 3Y-TZP zirconia shows the smallest grain sizes, with its average size remaining stable across all analyzed thicknesses, ranging from $0.38 \pm 0.15 \mu\text{m}$ at 0.05 mm to $0.45 \pm 0.24 \mu\text{m}$ at 1.00 mm. This stability is typical of 3Y-TZP, where the predominant tetragonal phase resists grain growth due to its higher microstructural stability and lower grain boundary mobility. The smaller grain size leads to increased light scattering at the grain boundaries, which reduces optical transmittance, particularly at greater thicknesses. As a result, 3Y-TZP exhibits a more opaque appearance compared to zirconias with higher yttria content, where larger grains enable more efficient light transmission.

4Y-PSZ zirconia presents intermediate grain sizes across all thicknesses, ranging from $0.42 \pm 0.13 \mu\text{m}$ (0.05 mm) to $0.87 \pm 0.44 \mu\text{m}$ (1.00 mm). This increase in grain size and variation compared to 3Y-TZP is directly related to the stabilization of the cubic phase, which contributes to higher transmittance, as previously observed in the TP and CR results.

5Y-PSZ zirconia shows the largest grain sizes, varying from $0.48 \pm 0.16 \mu\text{m}$ (0.05 mm) to $0.96 \pm 0.43 \mu\text{m}$ (1.00 mm). The larger grain size and its variation are consistent with the

stabilization of the cubic phase, which enhances transmittance. However, this grain size increase may also contribute to a potential loss of optical uniformity, particularly in thicker samples.

These average grain size results are in agreement with several similar studies [12,44,52], where the influence of grain size on the optical properties of dental zirconias is observed.

Figure 5.8 presents Scanning Electron Microscopy (SEM) images at 10,000x magnification showing the grain structure of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconia ceramics at two thicknesses: 0.05 mm (top row) and 1.00 mm (bottom row). These micrographs serve as examples of the extreme conditions used and demonstrate the results of the average grain sizes, where uniformity is observed in the 3Y-TZP zirconia structure, unlike what is seen in 4Y and 5Y zirconias, which predominantly present larger grains but also small grains, aligning with the standard deviation results.

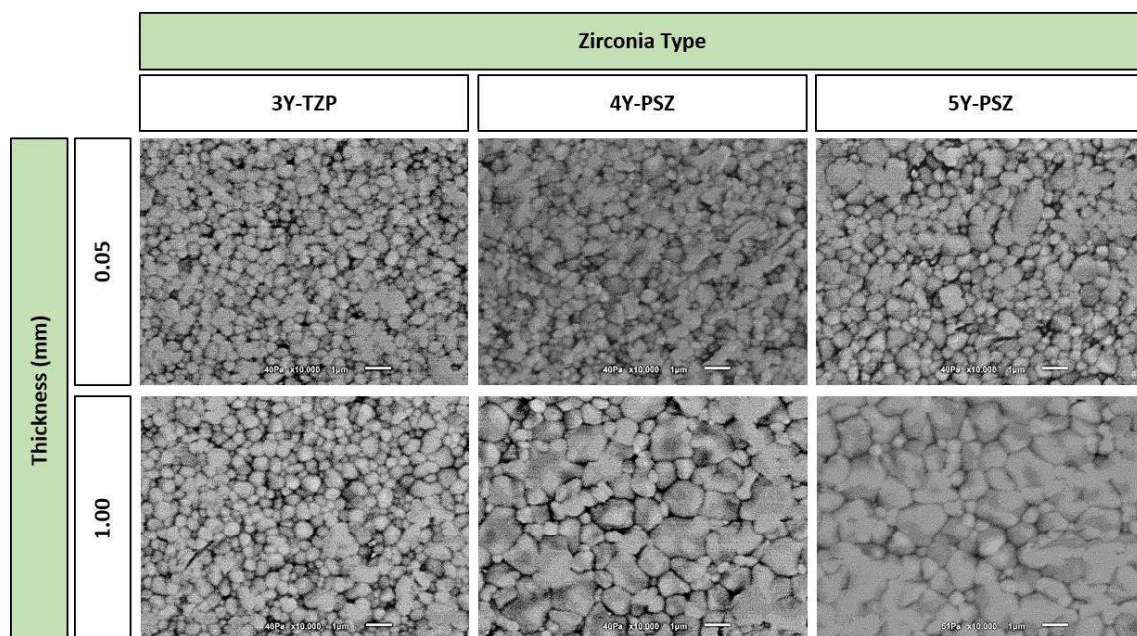


Figure 5.8 - Scanning Electron Microscopy (SEM) images at 10,000x magnification showing the grain structure of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ zirconia ceramics at two thicknesses: 0.05 mm (top row) and 1.00 mm (bottom row).

When comparing our results with the existing literature, we observe that the absolute TP and CR values for conventional thicknesses (1.00 mm and 0.45 mm) are consistent with some previous studies [8,11,46]. However, our study stands out by providing data on ultra-thin thicknesses (0.30 mm, 0.15 mm, and 0.05 mm), which have not been previously reported in the literature.

The clinical implications of these results are significant. The possibility of using zirconia at ultra-thin thicknesses without significantly compromising translucency opens new perspectives for minimally invasive restorations. Zirconia contact lenses refer to ultra-thin zirconia layers designed

for minimally invasive dental restorations, similar in concept to porcelain veneers but requiring significantly less tooth reduction. These ultra-thin layers, often as thin as 0.3 mm or less, adhere directly to the enamel surface and serve as a restorative material for enhancing aesthetics and functionality in the anterior teeth region. Their thickness is comparable to a contact lens for the eye, hence the terminology. By combining excellent aesthetics with superior mechanical strength, zirconia contact lenses present a promising alternative to traditional feldspathic veneers, offering enhanced durability and tooth preservation [53]. However, it is crucial to consider that reducing thickness also implies a decrease in mechanical strength, as demonstrated in previous studies [28,52,54] which reported a significant reduction in fracture resistance when decreasing the thickness of 3Y-TZP zirconia.

With the patented CAD/CAM production method used in this study, we were able to go beyond the minimum thickness of 0.5 mm suggested by CNC milling machine manufacturers [53] achieving thicknesses of 0.3, 0.15, and 0.05 mm. The reduction to these ultra-thin dimensions is not merely a technical achievement but also a significant contribution to clinical practice, as it challenges the current guidelines on the minimum thickness required to maintain both mechanical and aesthetic properties. Current ceramic veneers often require high tooth reduction to accommodate material thickness, which can lead to irreversible damage to healthy tooth structure, increased tooth sensitivity, and the potential need for endodontic treatment. In this context, the use of 3Y-TZP at 0.3 mm and 0.15 mm thicknesses allows for preservation of tooth structure while retaining mechanical strength and durability, thus addressing a critical gap in minimally invasive applications [55–57].

While other studies [18–20] suggest that thicknesses between 0.8 and 1.5 mm are necessary for dental restorations using 4Y-PSZ and 5Y-PSZ zirconia, it can be considered that using 3Y-TZP zirconia at thicknesses of 0.3 and 0.15 mm makes it possible to achieve the desired translucency level without compromising mechanical strength.

5.3.3 Flexible behavior in Ultra-Thin 3Y-TZP Zirconia and potential applications

Another interesting finding that emerged from this study was the 3Y-TZP zirconia sample with a thickness of 0.05 mm, which exhibited a flexible behaviour (as illustrated in Figure 5.9), an

uncommon characteristic for ceramic materials. This observation suggests that ultra-thin zirconia layers may possess a degree of mechanical adaptability, opening new perspectives for applications beyond dentistry. This flexible behaviour is particularly interesting for biomedical and electronic device engineering. In implantable devices, for instance, the combination of mechanical strength with a certain degree of flexibility could be advantageous for creating components that better adapt to biomechanical stresses without fracturing, thereby improving the material's durability and compatibility with the biological environment. In electronics, 3Y-TZP at this thickness could function as a flexible dielectric, isolating circuits in microdevices subjected to mechanical deformations [58]. While further quantitative analyses are required to fully characterize this behaviour, these preliminary findings indicate the potential for ultra-thin 3Y-TZP zirconia to adapt mechanically in contexts where ceramics are typically brittle. This unique characteristic makes 3Y-TZP a promising candidate for novel applications, combining its well-known mechanical strength with an unexpected degree of flexibility.

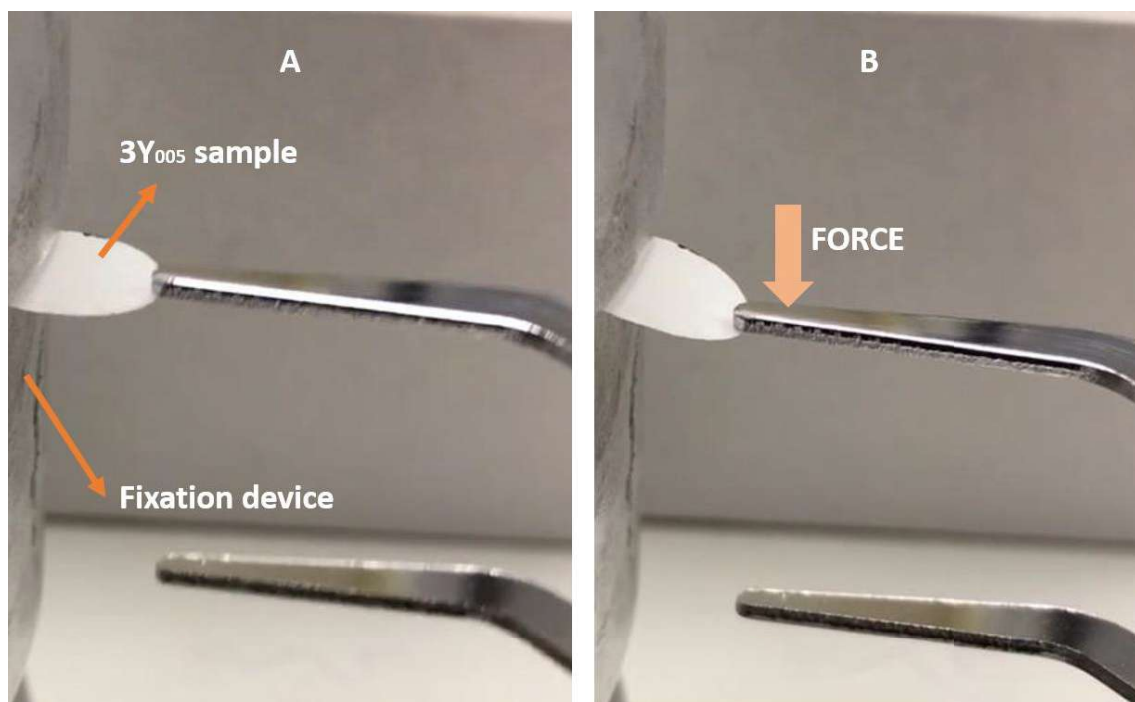


Figure 5.9 - Zirconia sample (3Y-TZP, 0.05 mm thickness) held by a fixation device. In image A, the sample is not subjected to any applied force, while in image B, a force is applied to demonstrate the flexibility of the zirconia, as it returns to its original position after the force is removed.

While the current study provides valuable insights into the translucency and contrast ratio of ultra-thin zirconia specimens, the detection of subtle differences in these parameters at 0.05 mm thickness may have been influenced by the resolution of the measurement techniques employed. Future investigations could benefit from advanced optical characterization methods, such as spectrophotometers with higher sensitivity or imaging-based techniques, to capture more granular differences. Additionally, computational modeling of light transmission through ultra-thin zirconia layers may provide complementary insights, helping to validate and expand upon the findings presented here.

In this study, the results demonstrate that it is possible to achieve comparable translucency levels between different types of zirconia (3Y-TZP, 4Y-PSZ, and 5Y-PSZ) at ultra-thin thicknesses, thus affirmatively addressing the question proposed in the title of the work. This finding has significant implications for the design of dental restorations, potentially allowing the use of zirconias with higher mechanical strength (such as 3Y-TZP) in applications that previously prioritized more translucent materials. However, the clinical application of these findings requires further studies, particularly regarding the long-term durability and performance of ultra-thin zirconia restorations in the oral environment.

5.4 Conclusions

This study investigated whether 3Y-TZP zirconia can reach the translucency levels of 5Y-PSZ zirconia at various thicknesses, ranging from conventional (1.00 mm) to ultra-thin dimensions (0.05 mm). The findings indicate that:

- At conventional thicknesses (≥ 0.45 mm), 3Y-TZP zirconia exhibits significantly lower translucency compared to 4Y-PSZ and 5Y-PSZ due to increased light scattering at the smaller grain boundaries and the dominance of the tetragonal phase;
- However, at ultra-thin thicknesses (≤ 0.15 mm), 3Y-TZP achieves similar or even higher translucency levels than 5Y-PSZ zirconia, demonstrating that the influence of yttria content on translucency becomes negligible at reduced thicknesses.
- The results confirm that 3Y-TZP zirconia can achieve translucency comparable to 5Y-PSZ, but only at ultra-thin dimensions (0.15 mm or below), challenging the common

assumption that a higher cubic phase content is always necessary for high translucency in dental zirconias.

- This finding has significant clinical implications, suggesting that 3Y-TZP, known for its superior mechanical strength, can be used in minimally invasive restorations without compromising aesthetics, thereby expanding its application in aesthetic zones.

Acknowledgements

This work was supported by Fundação para a Ciência e Tecnologia (FCT, Portugal) through the grant SFRH/BD/145421/2019 (DOI: <https://doi.org/10.54499/SFRH/BD/145421/2019>) and national funds through the project FunFibRAI, PTDC/EME-EME/4197/2021 (DOI 10.54499/PTDC/EME-EME/4197/2021). Additional strategic funding was provided by CMEMS-UMinho, UIDB/04436/2020 (DOI 10.54499/UIDB/04436/2020), UIDP/04436/2020 (DOI 10.54499/UIDP/04436/2020), & LABBELS – Associate Laboratory in Biotechnology, Bioengineering and Microelectromechanical Systems, LA/P/0029/2020 (DOI 10.54499/LA/P/0029/2020).



References

- [1] L.H. et al Silva, Critical review Dental Materials/Dentistry Dental ceramics: a review of new materials and processing methods, *Braz. Oral Rest.* 31 (2017) 133–146. <https://doi.org/10.1590/1807-3107BOR-2017.vol31.0058>.
- [2] P. Shelar, S. Butler, H. Abdolvand, On the behaviour of zirconia-based dental materials: A review, *J. Mech. Behav. Biomed. Mater.* 124 (2021) 104861. <https://doi.org/10.1016/j.jmbbm.2021.104861>.
- [3] T. Dantas, J. Padrão, M.R. da Silva, P. Pinto, S. Madeira, P. Vaz, A. Zille, F. Silva, Bacteria co-culture adhesion on different texturized zirconia surfaces, *J. Mech. Behav. Biomed. Mater.* 123 (2021). <https://doi.org/10.1016/j.jmbbm.2021.104786>.
- [4] G. Marchesi, A.C. Piloni, V. Nicolini, G. Turco, R. Di Lenarda, Chairside cad/cam materials: Current trends of clinical uses, *Biology* (Basel). 10 (2021). <https://doi.org/10.3390/biology10111170>.

- [5] M. Suganna, H. Kausher, S. Tarek Ahmed, H. Sultan Alharbi, B. Faraj Alsubaie, A. DS, S. Haleem, A.B. Meer Rownaq Ali, Contemporary Evidence of CAD-CAM in Dentistry: A Systematic Review, *Cureus*. 14 (2022). <https://doi.org/10.7759/cureus.31687>.
- [6] P.F. Gouveia, L.M. Schabbach, J.C.M. Souza, B. Henriques, J.A. Labrincha, F.S. Silva, M.C. Fredel, J. Mesquita-Guimarães, New perspectives for recycling dental zirconia waste resulting from CAD/CAM manufacturing process, *J. Clean. Prod.* 152 (2017) 454–463. <https://doi.org/10.1016/j.jclepro.2017.03.117>.
- [7] A.N. Cavalcanti, R.M. Foxton, T.F. Watson, M.T. Oliveira, M. Giannini, G.M. Marchi, Y-TZP ceramics: Key concepts for clinical application, *Oper. Dent.* 34 (2009) 344–351. <https://doi.org/10.2341/08-79>.
- [8] H. Tong, C.B. Tanaka, M.R. Kaizer, Y. Zhang, Characterization of three commercial Y-TZP ceramics produced for their High-Translucency, High-Strength and High-Surface Area, *Ceram. Int.* 42 (2016) 1077–1085. <https://doi.org/10.1016/j.ceramint.2015.09.033>.
- [9] N. Heidari, R. Amawi, P. Seweryniak, F. Bakitian, P.V. von Steyern, Fracture Resistance and Fracture Behaviour of Monolithic Multi-Layered Translucent Zirconia Fixed Dental Prostheses with Different Placing Strategies of Connector: An in vitro Study, *Clin. Cosmet. Investig. Dent.* 14 (2022) 61–69. <https://doi.org/10.2147/CCIDE.S344941>.
- [10] S.M. Čokić, M. Córdor, J. Vleugels, B. Van Meerbeek, H. Van Oosterwyck, M. Inokoshi, F. Zhang, Mechanical properties–translucency–microstructure relationships in commercial monolayer and multilayer monolithic zirconia ceramics, *Dent. Mater.* 38 (2022) 797–810. <https://doi.org/10.1016/j.dental.2022.04.011>.
- [11] C.D. Stevens, W.G. Renné, J. Vág, Translucency of chairside monolithic zirconias using different sintering ovens: An in vitro investigation, *J. Dent.* 142 (2024). <https://doi.org/10.1016/j.jdent.2024.104839>.
- [12] J. Hjerpe, M. Özcan, Zirconia: More and More Translucent, *Curr. Oral Heal. Reports*. 10 (2023) 203–211. <https://doi.org/10.1007/s40496-023-00344-1>.
- [13] Y.-K. Lee, Translucency of human teeth and dental restorative materials and its clinical relevance, *J. Biomed. Opt.* 20 (2015) 045002. <https://doi.org/10.1117/1.jbo.20.4.045002>.
- [14] R. Shahmiri, O.C. Standard, J.N. Hart, C.C. Sorrell, Optical properties of zirconia ceramics for esthetic dental restorations: A systematic review, *J. Prosthet. Dent.* 119 (2018) 36–46. <https://doi.org/10.1016/j.prosdent.2017.07.009>.
- [15] S. Mekled, S. Elwazeer, C.A. Jurado, J. White, F. Faddoul, K.I. Afrashtehfar, N.G. Fischer, Ultra-Translucent Zirconia Laminate Veneers: The Influence of Restoration Thickness and Stump Tooth-Shade, *Materials (Basel)*. 16 (2023) 1–12. <https://doi.org/10.3390/ma16083030>.
- [16] A.D. Malallah, N.H. Hasan, Thickness and yttria percentage influences the fracture resistance of laminate veneer zirconia restorations, *Clin. Exp. Dent. Res.* 8 (2022) 1413–1420. <https://doi.org/10.1002/cre2.658>.
- [17] R. Souza, F. Barbosa, G. Araújo, E. Miyashita, M.A. Bottino, R. Melo, Y. Zhang, Ultrathin monolithic zirconia veneers: Reality or future? report of a clinical case and one-year follow-up, *Oper. Dent.* 43 (2018) 3–11. <https://doi.org/10.2341/16-350-T>.
- [18] I.A. Alraheem, T. Donovan, L. Boushell, R. Cook, A. V. Ritter, T.A. Sulaiman, Fracture load of two thicknesses of different zirconia types after fatiguing and thermocycling, *J. Prosthet. Dent.* 123 (2020) 635–640. <https://doi.org/10.1016/j.prosdent.2019.05.012>.
- [19] T.A. Sulaiman, A.A. Suliman, A.A. Abdulmajeed, Y. Zhang, Zirconia restoration types, properties, tooth preparation design, and bonding. A narrative review, *J. Esthet. Restor. Dent.* 36 (2024) 78–84. <https://doi.org/10.1111/jerd.13151>.

- [20] A. Abdulmajeed, T. Sulaiman, A. Abdulmajeed, S. Bencharit, T. Närhi, Fracture Load of Different Zirconia Types: A Mastication Simulation Study, *J. Prosthodont.* 29 (2020) 787–791. <https://doi.org/10.1111/jopr.13242>.
- [21] F. Vafaei, A. Shahbazi, A. Hooshyarfard, A. Najafi, M. Ebrahimi, M. Farhadian, Effect of sintering temperature on translucency parameter of zirconia blocks, *Dent. Res. J. (Isfahan)*. 19 (2022) 82. <https://doi.org/10.4103/1735-3327.359319>.
- [22] C.M. Kang, Y.W. Huang, S.H. Wu, Y. Mine, I.T. Lee, T.Y. Peng, Evaluation of shade correspondence between high-translucency pre-colored zirconia and shade tab by considering the influence of cement shade and substrate materials, *Heliyon*. 9 (2023) e23046. <https://doi.org/10.1016/j.heliyon.2023.e23046>.
- [23] P.H. Chen, E. Elamin, A. Sayed Ahmed, D.A. Givan, C.C. Fu, N.C. Lawson, The Effect of Restoration Thickness on the Fracture Resistance of 5 mol% Yttria-Containing Zirconia Crowns, *Materials (Basel)*. 17 (2024) 1–10. <https://doi.org/10.3390/ma17020365>.
- [24] D. Awad, B. Stawarczyk, A. Liebermann, N. Ilie, Translucency of esthetic dental restorative CAD/CAM materials and composite resins with respect to thickness and surface roughness, *J. Prosthet. Dent.* 113 (2015) 534–540. <https://doi.org/10.1016/j.prosdent.2014.12.003>.
- [25] Y. Zhang, B.R. Lawn, Novel Zirconia Materials in Dentistry, *J. Dent. Res.* 97 (2018) 140–147. <https://doi.org/10.1177/0022034517737483>.
- [26] G. Alrabeah, N. Alamro, A. Alghamdi, A. Almslam, M. Azaaqi, Influences of luting cement shade on the color of various translucent monolithic zirconia and lithium disilicate ceramics for veneer restorations, *J. Adv. Prosthodont.* 15 (2023) 238–247. <https://doi.org/10.4047/jap.2023.15.5.238>.
- [27] J.H. Cho, J.S. Han, H.I. Yoon, I.S.L. Yeo, Effect of phase fraction and grain size on translucency of 3 mol% yttria-stabilized tetragonal zirconia polycrystal, *J. Mater. Res. Technol.* 25 (2023) 1222–1230. <https://doi.org/10.1016/j.jmrt.2023.06.049>.
- [28] L.M. Schabbach, B.C. dos Santos, L.S. De Bortoli, D. Fabris, M.C. Fredel, B. Henriques, Translucent multi-layered zirconia: Sandblasting effect on optical and mechanical properties, *Dent. Mater.* 39 (2023) 807–819. <https://doi.org/10.1016/j.dental.2023.07.005>.
- [29] M. Michailova, A. Elsayed, G. Fabel, D. Edelhoff, I.M. Zylla, B. Stawarczyk, Comparison between novel strength-gradient and color-gradient multilayered zirconia using conventional and high-speed sintering, *J. Mech. Behav. Biomed. Mater.* 111 (2020) 103977. <https://doi.org/10.1016/j.jmbbm.2020.103977>.
- [30] B. Stawarczyk, M. Özcan, L. Hallmann, A. Ender, A. Mehl, C.H.F. Hammerlet, The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio, *Clin. Oral Investig.* 17 (2013) 269–274. <https://doi.org/10.1007/s00784-012-0692-6>.
- [31] P. Pinto, A. Carvalho, F.S. Silva, J.R. Gomes, O. Carvalho, S. Madeira, Comparative toothbrush abrasion resistance and surface analysis of different dental restorative materials, *Tribol. Int.* 175 (2022) 107799. <https://doi.org/10.1016/j.triboint.2022.107799>.
- [32] R. Ghinea, M.M. Pérez, L.J. Herrera, M.J. Rivas, A. Yebra, R.D. Paravina, Color difference thresholds in dental ceramics, *J. Dent.* 38 Suppl 2 (2010). <https://doi.org/10.1016/J.JDENT.2010.07.008>.
- [33] Y.K. Lee, B. Yu, S.H. Lee, M.S. Cho, C.Y. Lee, H.N. Lim, Variation in instrument-based color coordinates of esthetic restorative materials by measurement method—A review, *Dent. Mater.* 26 (2010) 1098–1105. <https://doi.org/10.1016/J.DENTAL.2010.07.010>.

- [34] H.H. Harianawala, M.G. Kheur, S.K. Apte, B.B. Kale, T.S. Sethi, S.M. Kheur, Comparative analysis of transmittance for different types of commercially available zirconia and lithium disilicate materials, *J. Adv. Prosthodont.* 6 (2014) 456–461. <https://doi.org/10.4047/jap.2014.6.6.456>.
- [35] L. Mao, M.R. Kaizer, M. Zhao, B. Guo, Y.F. Song, Y. Zhang, Graded Ultra-Translucent Zirconia (5Y-PSZ) for Strength and Functionalities, *J. Dent. Res.* 97 (2018) 1222–1228. <https://doi.org/10.1177/0022034518771287>.
- [36] R. Shahmiri, O.C. Standard, J.N. Hart, G. Bahmanrokh, Y. Yin, A. Samiee, N. Gharagozlu, C.C. Sorrell, Critical effects of thermal processing conditions on grain size and microstructure of dental Y-TZP during layering and glazing, *J. Mater. Sci.* 58 (2023) 3854–3878. <https://doi.org/10.1007/s10853-023-08227-7>.
- [37] Y. Zhang, Making yttria-stabilized tetragonal zirconia translucent, *Dent. Mater.* 30 (2014) 1195–1203. <https://doi.org/10.1016/j.dental.2014.08.375>.
- [38] Y.E. Cho, Y.J. Lim, J.S. Han, I.S.L. Yeo, H.I. Yoon, Effect of yttria content on the translucency and masking ability of yttria-stabilized tetragonal zirconia polycrystal, *Materials (Basel)*. 13 (2020) 1–10. <https://doi.org/10.3390/ma13214726>.
- [39] A. Alameldin, T. Shokry, A. El-Kouedi, Effect of Thickness Variation on the Translucency of Two Recent High Translucent Monolithic Zirconia Ceramics, *Al-Azhar J. Dent. Sci.* 23 (2020) 321–326. <https://doi.org/10.21608/ajdsm.2020.25066.1012>.
- [40] E. Kontonasaki, A.E. Rigos, C. Ilia, T. Istantos, Monolithic Zirconia : An Update to Current, *Dent. J.* 90 (2019) 23.
- [41] C.M. Kang, T.Y. Peng, H.H. Huang, Effects of thickness of different types of high-translucency monolithic multilayer precolored zirconia on color accuracy: An in vitro study, *J. Prosthet. Dent.* 126 (2021) 587.e1-587.e8. <https://doi.org/10.1016/j.prosdent.2021.07.011>.
- [42] A.M. de O. Dal Piva, J.P.M. Tribst, E.B. Benalcázar Jalkh, L.C. Anami, E.A. Bonfante, M.A. Bottino, Minimal tooth preparation for posterior monolithic ceramic crowns: Effect on the mechanical behavior, reliability and translucency, *Dent. Mater.* 37 (2021) e140–e150. <https://doi.org/10.1016/j.dental.2020.11.001>.
- [43] S.R. Khosravani, M.A. Kahnamoui, S. Kimyai, E.J. Navimipour, F.S. Mahounak, F.P. Azar, Final Colour of Ultratranslucent Multilayered Zirconia Veneers, Effect of Thickness, and Resin Cement Shade, *Biomed Res. Int.* 2022 (2022). <https://doi.org/10.1155/2022/2555797>.
- [44] C. Santos, J. Baltazar, M. F.R.P. Alves, S.M. Olhero, Development of translucent zirconia by robocasting, *Mater. Lett.* 325 (2022) 132785. <https://doi.org/10.1016/j.matlet.2022.132785>.
- [45] F.C.S. Chu, T.W. Chow, J. Chai, Contrast-ratios-and-masking-ability-of-three-types-of-ceramic-veneers_2007_The-Journal-of-Prosthetic-Dentistry, (2007).
- [46] T.A. Sulaiman, A.A. Abdulmajeed, T.E. Donovan, A. V. Ritter, P.K. Vallittu, T.O. Närhi, L. V. Lassila, Optical properties and light irradiance of monolithic zirconia at variable thicknesses, *Dent. Mater.* 31 (2015) 1180–1187. <https://doi.org/10.1016/j.dental.2015.06.016>.
- [47] V. Kulyk, Z. Duriagina, A. Kostyryhev, B. Vasylyv, V. Vavrukh, O. Marenych, The Effect of Yttria Content on Microstructure, Strength, and Fracture Behavior of Yttria-Stabilized Zirconia, *Materials (Basel)*. 15 (2022). <https://doi.org/10.3390/ma15155212>.
- [48] M. Trunec, Effect of grain size on mechanical properties of 3Y-TZP ceramics, *Ceram. - Silikaty.* 52 (2008) 165–171.
- [49] N. Ilie, B. Stawarczyk, Quantification of the amount of blue light passing through monolithic zirconia with respect to thickness and polymerization conditions, *J. Prosthet. Dent.* 113 (2015) 114–121. <https://doi.org/10.1016/j.prosdent.2014.08.013>.

- [50] H. Chai, M. Kaizer, A. Chughtai, H. Tong, C. Tanaka, Y. Zhang, On the interfacial fracture resistance of resin-bonded zirconia and glass-infiltrated graded zirconia, *Dent. Mater.* 31 (2015) 1304–1311. <https://doi.org/10.1016/j.dental.2015.08.161>.
- [51] K. Harada, A.J. Raigrodski, K.H. Chung, B.D. Flinn, S. Dogan, L.A. Mancl, A comparative evaluation of the translucency of zirconias and lithium disilicate for monolithic restorations, *J. Prosthet. Dent.* 116 (2016) 257–263. <https://doi.org/10.1016/j.prosdent.2015.11.019>.
- [52] T. Strasser, M. Wertz, A. Koenig, T. Koetzsch, M. Rosentritt, Microstructure, composition, and flexural strength of different layers within zirconia materials with strength gradient, *Dent. Mater.* 39 (2023) 463–468. <https://doi.org/10.1016/j.dental.2023.03.012>.
- [53] G. Carpena DDS, MS, PhD, A. Balaron DDS, J. Aguiar, A new ceramics approach for contact lens, *Odovtos - Int. J. Dent. Sci.* 17 (2015) 14. <https://doi.org/10.15517/ijds.v0i0.22040>.
- [54] M. Zahran, S. Abo El-Farag, H. Soltan, A. Attia, Fracture load of ultrathin occlusal veneers: Effect of thickness and surface conditioning, *J. Mech. Behav. Biomed. Mater.* 145 (2023) 106030. <https://doi.org/10.1016/j.jmbbm.2023.106030>.
- [55] Z. Begum, P. Chheda, C.S. Shruthi, R. Sonika, Effect of Ceramic Thickness and Luting Agent Shade on the Color Masking Ability of Laminate Veneers, *J. Indian Prosthodont. Soc.* 14 (2014) 46–50. <https://doi.org/10.1007/s13191-014-0362-2>.
- [56] Ali Allen Nasseh, Endodontic Complications of Esthetic Restorative Procedures, *Insid. Dent.* (2006) Volume 2, Issue 4. <https://www.aegisdentalnetwork.com/id/2006/05/endodontics-endodontic-complications-of-esthetic-restorative-procedures> (accessed October 4, 2024).
- [57] Timberlands Dental Clinic, The Connection Between Dental Veneers and Tooth Sensitivity, (n.d.). <https://timberlandsdental.com.au/the-connection-between-dental-veneers-and-tooth-sensitivity/> (accessed October 4, 2024).
- [58] J. Ajayan, S. Sreejith, M. Manikandan, V.B. Sreenivasulu, N. Aruna, A. Ravindran, Micro and Nanostructures An intensive study on organic thin film transistors (OTFTs) for future flexible / wearable electronics applications, *Micro and Nanostructures.* 187 (2024) 207766. <https://doi.org/10.1016/j.micrna.2024.207766>.

CHAPTER 6.

Digital workflow for ultra-thin 3Y-TZP dental veneers: CAD/CAM
manufacturing approach

6.1 Introduction

As previously mentioned, the integration of digital technologies in dentistry has significantly transformed the processes of diagnosis, treatment planning, and fabrication of restorations, enabling a more precise, efficient, and personalized approach [1–3]. Although manual layering techniques can achieve good aesthetic outcomes and clinical success rates, they are associated with high technical effort and limited by the low flexural strength of the layered structures [3].

In the context of ultra-thin dental veneers made from 3Y-TZP zirconia, the implementation of a fully digital workflow is essential to ensure the quality and longevity of the restorations, particularly when aiming to minimize the invasiveness of clinical procedures.

The digital workflow for producing 3Y-TZP zirconia veneers comprises several stages, ranging from the acquisition of three-dimensional data of the dental arches to the final finishing and surface conditioning for adhesive bonding.

This chapter presents the digital strategy employed to fabricate an ultra-thin 3Y-TZP zirconia veneer with a thickness of 0.15 mm for tooth 11 (selected as example), using a realistic anatomical training model identical to the one shown in Figure 6.1 [Model SP-DPM-32, NISSIN Dental Products, Japan].



Figure 6.1 - Realistic anatomical dental model used (NISSIN Dental Products, Japan).

6.2 Acquisition of the digital model

The acquisition of an accurate three-dimensional (3D) model of the patient's dentition is the first essential step in the digital workflow for the production of ultra-thin dental veneers. This stage aims to capture the intraoral geometry with high fidelity, enabling precise digital design (CAD) and subsequent fabrication (CAM) of the restoration.

For this purpose, the Medit i500 intraoral scanner (Figure 6.2A) [Medit Corp., South Korea] was used. Thanks to its real-time video capture at 35 fps, the device automatically converts the images into a three-dimensional mesh (Figure 6.2B).



Figure 6.2 - Medit i500 intraoral scanner (A) and scanning process (B).

The reference values for the dimensional accuracy of the images acquired by this scanner are presented in Table 6.1 and fall within the recommended tolerances for high-precision indirect restorations [4].

Table 6.1 - Accuracy of IOS Medit i500 usado neste estudo. * Accuracy test in vitro was conducted by Yonsei University College of Dentistry according to the methods in "Accuracy comparison analysis on scan data of single tooth, quadrant, and full arch between two types of intraoral scanner, i500 and Trios 3" Ji-Man Park, 19 December 2018. ** Accuracy test in vivo was conducted by Medit

	In Vitro* (μm)		In vivo** (μm)
	Trueness	Precision	
Single	5.3 ± 0.34	3.2 ± 0.49	10
Quadrant	17.3 ± 0.43	6.3 ± 0.63	25
Full arch	21.0 ± 1.48	22.6 ± 7.55	50

The scanning was performed directly on the anatomical model, starting with the maxilla and then proceeding to the mandible, with real-time visual feedback using the MEDIT Link software [Medit, South Korea].

The following quality criteria were adopted during the scanning procedure:

- (i) complete coverage of all dental arch surfaces;
- (ii) elimination of mesh noise using the “Smart Scan” filter in the software;
- (iii) use of neutral lighting to avoid shadows or reflections.

Figure 6.3 shows the 3Dmodel obtained from both arches, with tooth 11 (upper arch) identified for veneer application. After acquisition and review of the digital model, the file was exported in STL format for subsequent CAD/CAM processing.

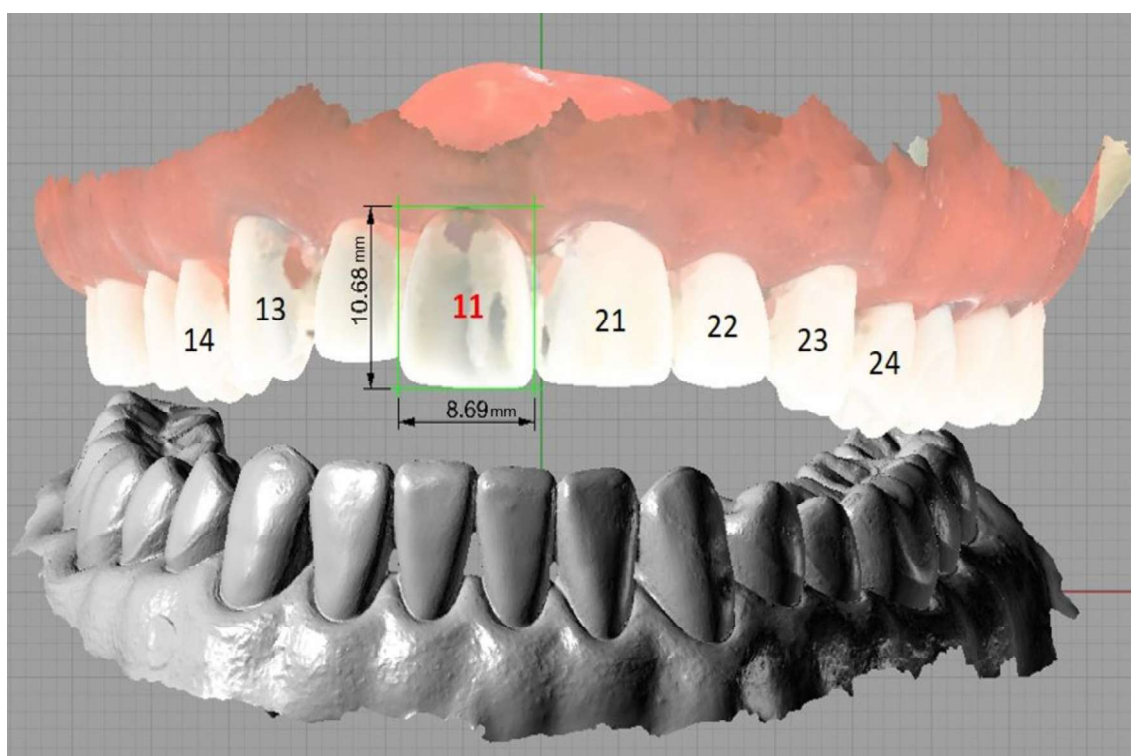


Figure 6.3 - Three-dimensional model of the maxillary and mandibular arches of the anatomical training model used in this study.

6.3 CAD design of the veneer

The next stage starts by importing the previously obtained STL file into the ExoCAD software (version V3.1-8349/64), where the type of restoration and the material to be used are defined (Figure 6.4). After selecting tooth “11” as the element to be restored, the key parameters - type of

restoration (veneer), material (zirconia), restoration thickness, cement gap, and shade - guiding the entire digital design process are configured (Table 6.2).

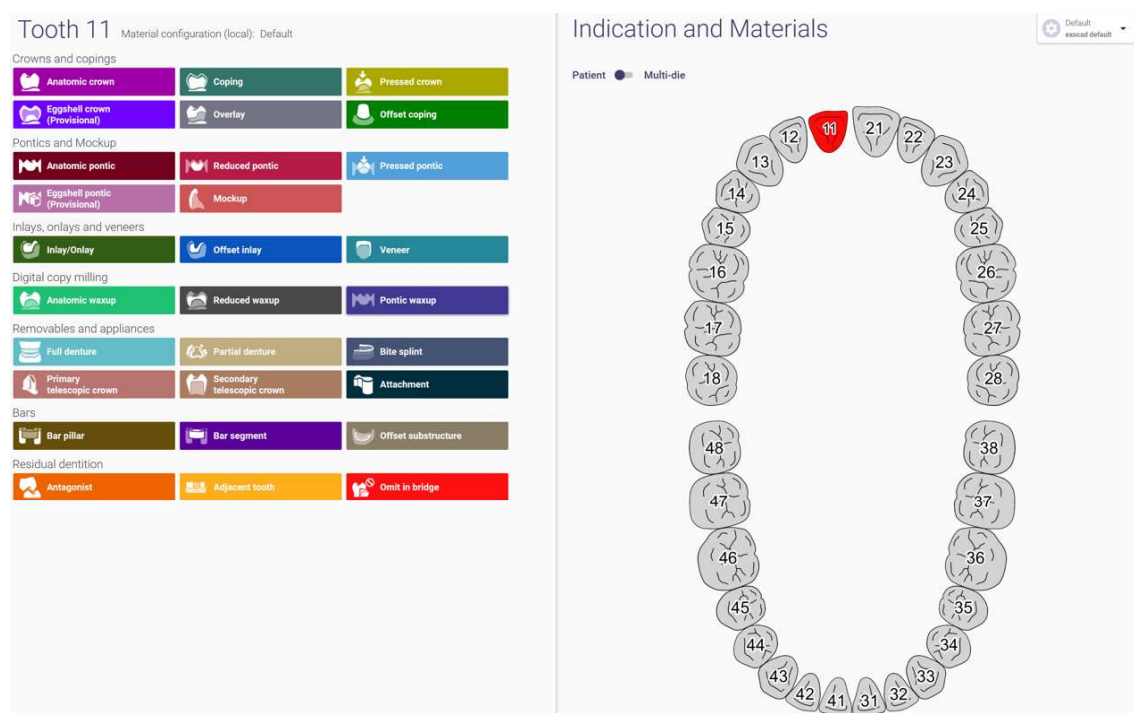


Figure 6.4 - ExoCAD software interface for restoration type selection.

Table 6.2 - Key parameters defined in the ExoCAD interface for the digital design of the zirconia veneer on tooth 11.

Parameter	Value	Interface ExoCAD image
Tooth	11	
Restoration	Veneer	
Material	Zirconia	
Thickness	0.15 mm	
Gap for resin bonding layer	0.03 mm	
Shade	A1	

Once the initial parameters are set, the next step involves the definition of the margin line, which is especially critical in no-prep restorations. This step ensures a smooth transition between enamel and ceramic, preserving the natural morphology and avoiding visible discontinuities. The margin line is marked in green in Figure 6.5.

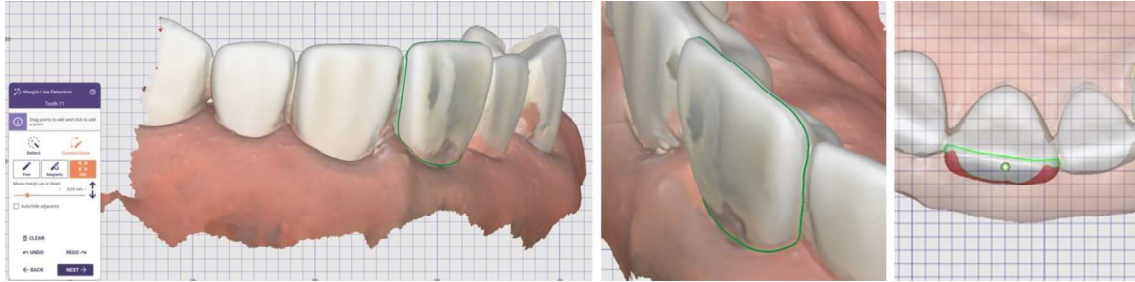


Figure 6.5 – Margin line creation step (green).

With the margin established, the vestibular anatomy (visible outer surface) of the veneer is constructed using anatomical libraries as a morphological reference. The goal is to accurately replicate the natural form of adjacent teeth, promoting ideal aesthetic integration.

The nominal thickness of the veneer was set at 0.15 mm. To ensure uniformity, ExoCAD automatically generates a thickness map, which is then manually adjusted to slightly reduce thickness near tooth 21 and reinforce the occlusal surface. The superimposed veneer (in white) over the digital model is shown in Figure 6.6.

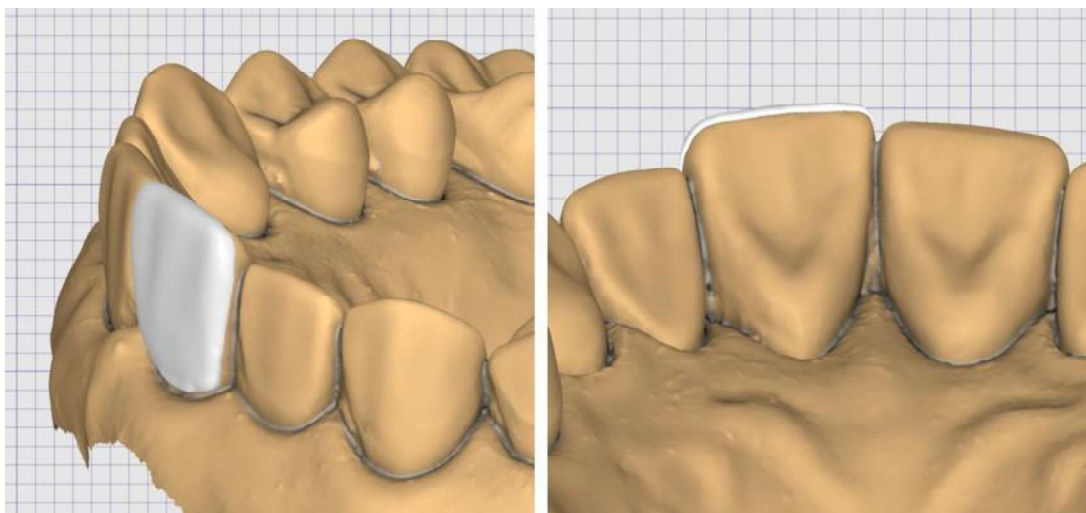


Figure 6.6 – Superimposition of the veneer (white) over the CAD model.

Once the design is completed, the model is exported in STL format, compatible with major CAM software for milling.

Before the final STL export, a rapid dimensional inspection is carried out using sectional measurements to confirm that the average thickness of the designed model falls within the expected tolerance — in this case, 0.15347 mm, which is very close to the target value of 0.15 mm (Figure 6.7). After this validation, the CAD model is ready for the production phase.



Figure 6.7 - Thickness verification of the veneer.

6.4 CAM and milling parameters

Once the CAD model has been validated, the production phase of the ultra-thin veneer begins, using computer-aided manufacturing (CAM). This process comprises three main stages:

- (1) CAM software programming;
- (2) Milling of the pre-sintered zirconia block;
- (3) Final sintering, which provides the material with the required densification to achieve optimal strength and translucency.

The STL file was imported into the HyperDent Compact V9.1 software [FOLLOW-ME! TECHNOLOGY, Munich, Germany], where the initial parameters were defined, including: machine type, restoration type, primary milling direction, margin line, blank type, material, and scaling factor. The used parameters are listed in Table 6.3.

Table 6.3 - Initial parameters defined in HyperDent Compact software.

Parameter	Value	Image
Machine	Coritec 250i (4 axis)	
Restoration	Veneer	
Diretion	Z+	N/A
Blank	Ø 98 x 14 mm	
Material	3Y-TZP DD Bio ZWiso zirconia	
Shade	A1	
Scale factor	1.25	

Based on this data, the software automatically generates the virtual representation of the material blank holder and positions the CAD model at the center of the workspace (Figure 6.8).

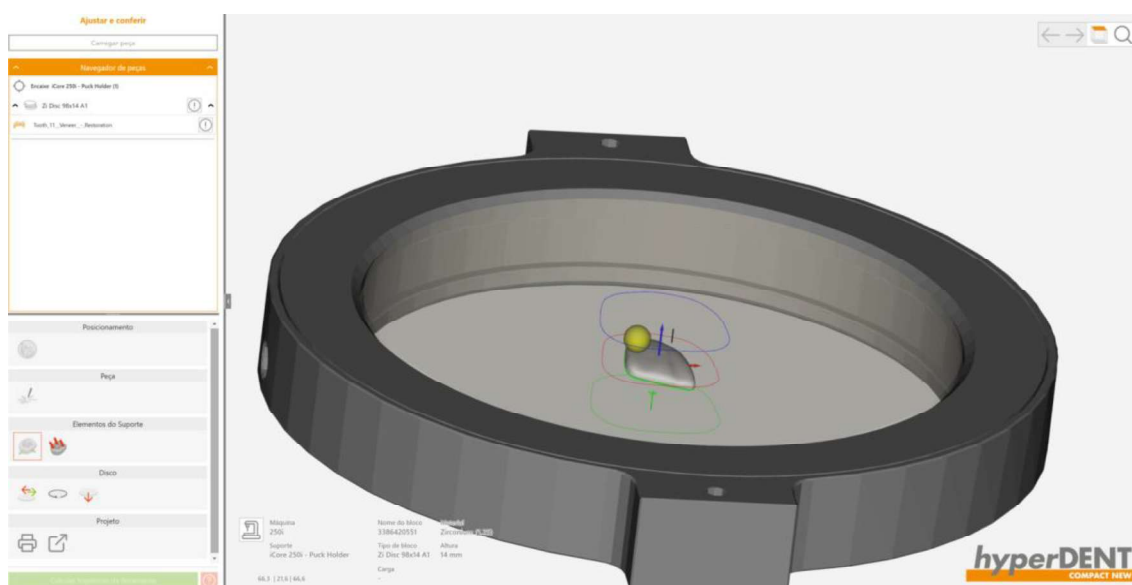


Figure 6.8 - Virtual representation of the zirconia blank holder and milling position of the veneer (centered).

The next step involves the definition of the milling operations, which in this case included one roughing operation and four finishing operations — two for each side (L1 – initial side; L2 – final side) — using tungsten carbide burs with CVD coating and spherical tips of Ø2 mm, Ø1 mm, and Ø0.5 mm. The roughing operation followed a standard protocol, employing a Ø2 mm bur at 25,000 rpm in a circular strategy. The stepover was set at one-third of the tool diameter, with a

feed rate of 25 mm/s and a Z-step of 0.6 mm per layer. A machining allowance of 0.1 mm was left for the finishing phase.

The finishing operations used Ø1 mm and Ø0.5 mm burs, with spindle speeds of 25,000 and 30,000 rpm, respectively. **The detailed procedure of the finishing strategy is restricted and subject to confidentiality due to patent protection.**

Once the tools and paths are defined, HyperDent calculates the toolpaths and simulates the material removal process until the final shape of the veneer is achieved. Figure 6.9 illustrates the different stages of the virtual milling simulation:

- A – Start of the roughing operation on side L1;
- B – End of the roughing operation on side L2;
- C – Finishing of side L1;
- D – Finishing of side L2.

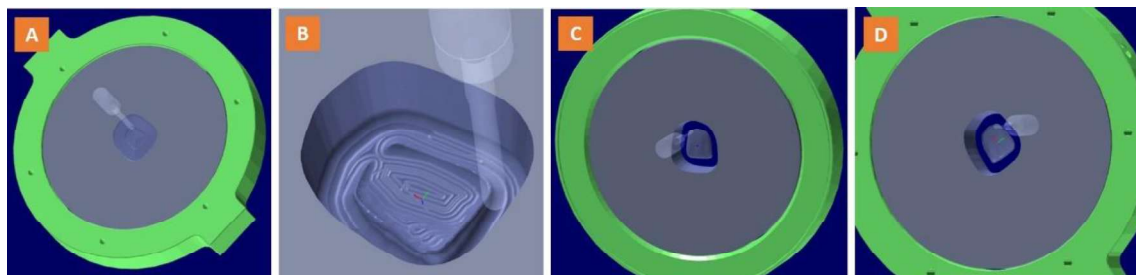


Figure 6.9 - Different stages of the virtual milling simulation. A – Start of roughing on side L1; B – End of roughing on side L2; C – Finishing on side L1; D – Finishing on side L2.

Finally, the CAM program (the milling "code") is exported, incorporating all the defined paths and parameters. Once loaded into the milling machine, this file ensures that the veneer is manufactured precisely, faithfully reproducing the contours and geometry of the pre-sintered zirconia structure.

6.5 Sintering process and inspection

Sintering is a critical step that transforms the pre-sintered zirconia piece — still porous, opaque, and mechanically fragile — into a dense, translucent, and mechanically resistant material. This process is essential for achieving the final properties of the dental veneer, particularly

mechanical strength, dimensional stability, and translucency. During sintering, zirconia particles undergo solid-state diffusion, leading to microstructural consolidation.

After milling, the veneer was placed at the center of a refractory base and introduced into the Zirkonofen 700 Ultra-Vakuum furnace (Zirkonzahn) (Figure 6.10).



Figure 6.10 - Veneer centered on the refractory base inside the Zirkonofen 700 furnace.

The control panel was used to select a sintering program with the following thermal cycle (Figure 6.11):

- Heating at 20 °C/min up to 900 °C, with a holding time of 30 minutes;
- Heating at 3 °C/min up to 1450 °C, with a holding time of 120 minutes;
- Cooling at 10 °C/min down to 200 °C.

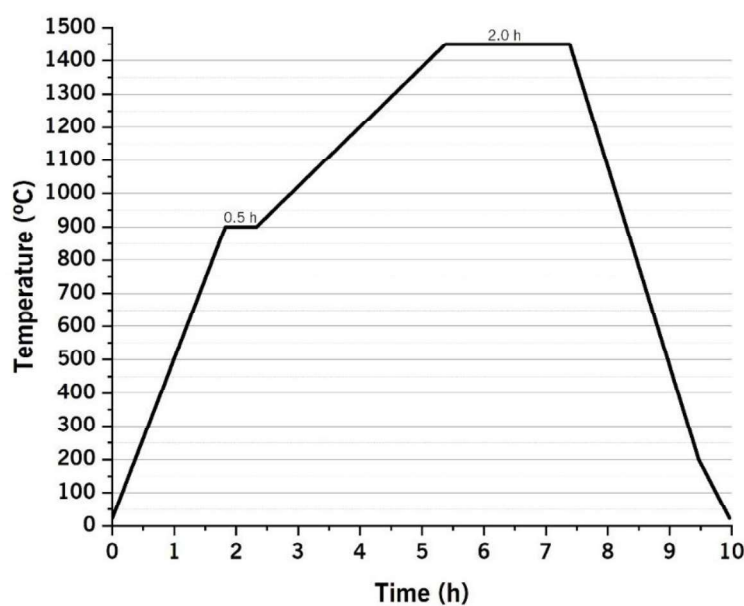


Figure 6.11 - Thermal cycle used for sintering the 3Y-TZP zirconia veneer.

This sintering process resulted in a volumetric shrinkage of approximately 20%, which had been previously compensated in the CAM software using a scale factor of 1.25, as recommended by the zirconia blank manufacturer. After sintering, the veneer presented a homogeneous and translucent appearance as shown in Figure 6.12.



Figure 6.12 - Final appearance of the veneer after sintering

Subsequently, a basic inspection was carried out using optical microscopy at 6× magnification, along with measurements in the ImageJ software. Figure 6.13 displays the thickness values across different regions of the veneer, confirming a slight reduction near the cervical margin (adjacent to tooth 21) and a moderate increase on the occlusal surface — consistent with the original CAD design.

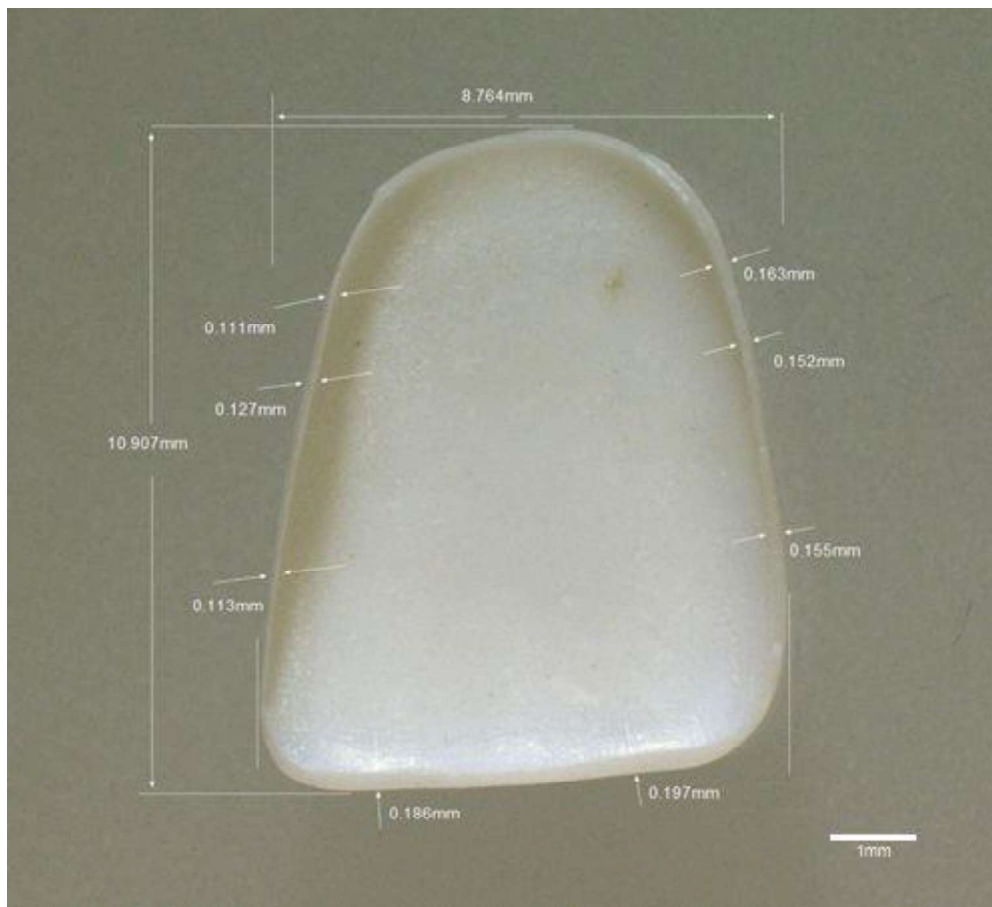


Figure 6.13 - Dimensional inspection using ImageJ software (6× magnification).

6.6 Surface finishing and surface treatment

After sintering, the 3Y-TZP zirconia veneer becomes dense, translucent, and mechanically stable. However, to ensure optimal clinical performance, both aesthetically and adhesively, it is essential to perform appropriate finishing and surface treatment procedures.

The process begins with polishing of the vestibular surface — a crucial step to enhance brightness, reduce surface roughness, and minimize biofilm retention [5,6]. For this purpose, a high-speed rotary tool (Dremel 3000, Bosch Power Tools) operating at up to 35,000 rpm was used (Figure 6.15A), along with dedicated zirconia finishing tools (Zirconia Adjustment & Polishing Kit, DIATECH, Switzerland). Figure 6.14 shows the kit used in this case, which includes tools for adjustment, pre-polishing, and high-gloss polishing.

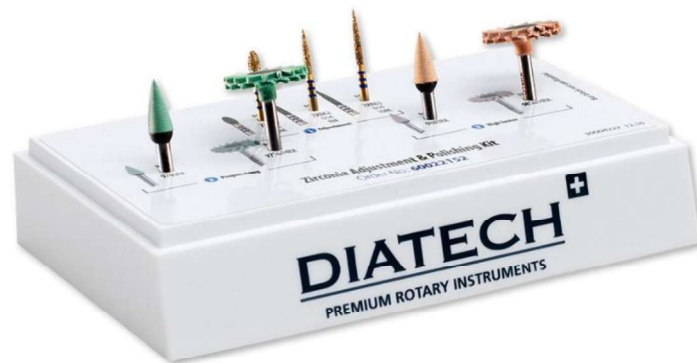


Figure 6.14 - Zirconia Adjustment & Polishing Kit (DIATECH, Switzerland) for zirconia finishing and polishing.

In this case, pre-polishing was performed using a green silicone rotary tip (97SG14RA) at 30,000 rpm (Figures 6.15B and 6.15C) to remove micro-irregularities, followed by high-gloss polishing with a beige tip (98SG14RA) at the maximum speed of 35,000 rpm (Figure 6.15D), resulting in a smooth, highly reflective surface.

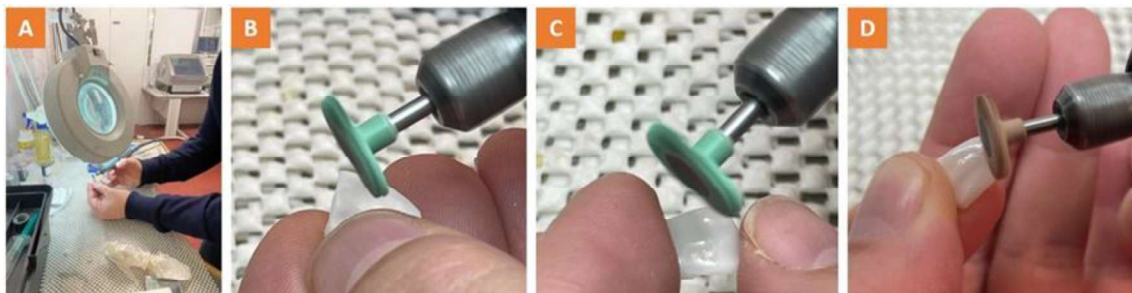


Figure 6.15 - Polishing sequence of the vestibular surface of the veneer. (A) Initial positioning and inspection of the veneer; (B) Pre-polishing using a green silicone disc (97SG14RA); (C) Continued pre-polishing; (D) High-gloss polishing

Maintaining a smooth occlusal surface is also crucial to avoid excessive wear of the opposing tooth; studies have shown [7] that a surface roughness (Ra) below $1.5\ \mu\text{m}$ helps preserve antagonist integrity.

Next, the internal surface of the veneer was treated following the procedure described in Subchapter 4.2.2 [8], which consisted of grit-blasting with $50\ \mu\text{m}$ Al_2O_3 particles at 2 bar pressure, applied from a distance of 15 mm for 20 seconds (Figure 6.16).

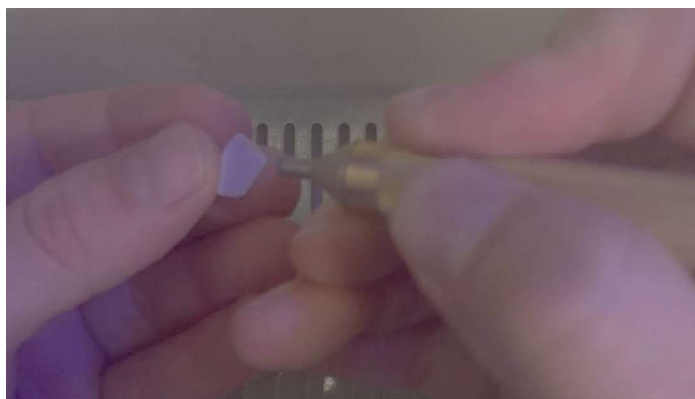


Figure 6.16 - Internal surface grit-blasting with 50 μm Al_2O_3 , 2 bar pressure, 15 mm distance, 20 seconds.

After completing the surface treatment, the veneer was cleaned in an ultrasonic bath for 3 minutes using isopropyl alcohol to remove residues and surface contaminants, leaving it ready for placement.

The veneer, prepared for try-in on tooth 11 of the anatomical model, is shown in Figure 6.17. It was temporarily positioned to assess fit, marginal adaptation, and anatomical and aesthetic integration.



Figure 6.17 - Final ultra-thin 3Y-TZP zirconia veneer ready for try-in.

Upon placement, excellent marginal adaptation and integration with surrounding tissues were observed. Although the veneer's shade did not exactly match that of the model tooth, this did not compromise the assessment, as the final color could be optimized by selecting a different ceramic block or using pigmented resin cements.

Figure 6.18 presents a detailed comparison of the model before (Figure 6.18A) and after (Figure 6.18B) veneer placement, as well as a color and brightness difference map generated through digital analysis (Figure 6.18C). The recorded color discrepancy was mainly confined to the interproximal area (evidenced in Figure 6.18C), likely associated with the margin definition in that

zone. This observation highlights the critical importance of precise control over the transition line between enamel and ceramic, especially in non-invasive restorations.

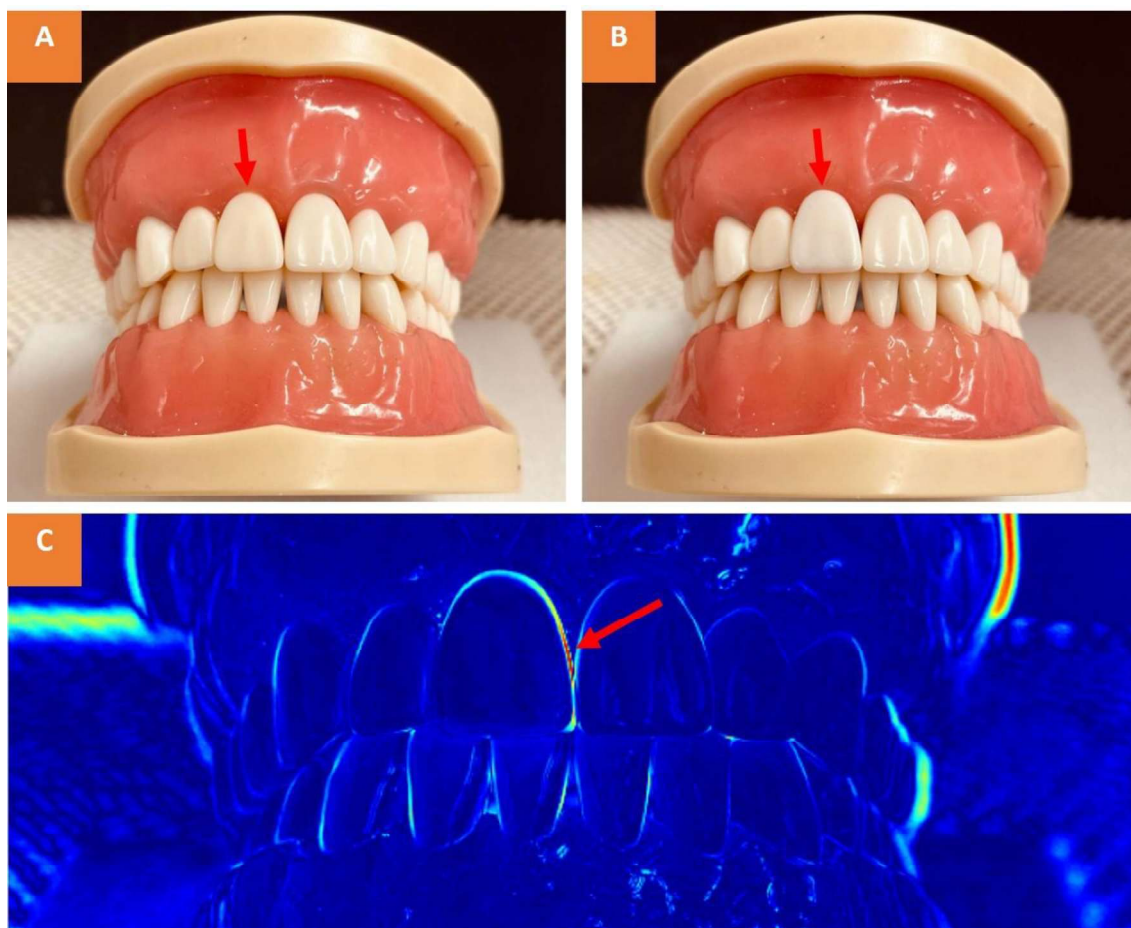


Figure 6.18 - Aesthetic integration assessment of the veneer in the anatomical model. (A) Model without veneer; (B) Model with veneer placed on tooth 11; (C) Color difference map indicating a small interproximal discontinuity (arrow indicates interproximal area).

6.7 Discussion and Critical Considerations

The fabrication of an ultra-thin dental veneer in 3Y-TZP zirconia proved to be technically feasible within the adopted digital workflow, although it presented significant challenges in terms of precision and strict control at each stage of the process. Milling was identified as the most demanding step, since fabrication of the structures of such reduced thickness is particularly difficult in fragile materials like pre-sintered zirconia. Nonetheless, the milling was successfully completed on the first attempt using advanced machining strategies developed under a currently active patent (details of which are confidential).

The adaptation of the veneer to tooth 11 of the anatomical model was generally satisfactory; however, digital comparative analysis before and after placement revealed a slight discrepancy in

the interproximal area. This result suggests that even with carefully controlled thickness mapping in CAD software, the fit may be affected by minor variations in the margin definition. Finishing was also a delicate step, requiring minimal pressure and the use of dedicated tools to preserve the structural integrity of the piece, particularly at the margins.

Surface treatment was completed without complications, as the pressure used (2 bar) did not compromise the structural stability of the veneer. It is important to note that the restoration was not cemented to the model, as this was a laboratory-based trial.

In summary, this work demonstrated that it is possible to produce 3Y-TZP zirconia veneers with thicknesses below 0.2 mm using a fully digital workflow. The clinical application of this type of restoration is feasible, but it demands a high level of laboratory expertise, and its success will always depend on the quality and consistency of the protocols implemented.

References

- [1] F.D. Salman, The Advancements in Non-Prep Veneers : A Review of Latest Techniques and Outcomes, 8 (2024) 1–2.
- [2] S. Marques, P. Ribeiro, C. Gama, M. Herrero-Climent, Digital guided veneer preparation: A dental technique, J. Prosthet. Dent. 131 (2024) 554–559. <https://doi.org/10.1016/j.prosdent.2022.04.035>.
- [3] O.S. J. Schweiger, D. Edelhoff, 3D printing of ultra-thin veneers made of lithium disilicate using the LCM method in a digital workflow: A feasibility study, (2023) 588–594. <https://doi.org/https://doi.org/10.1111/jerd.1315>.
- [4] X. Wang, F. Zhang, D. Ma, X. Ye, X. Zheng, R. Ren, N. Ren, S. Bai, Evaluation of the accuracy of seven intraoral scanners for the full dentate and partially edentulous complete-arch mandibular casts: An in vitro comparison, Heliyon. 10 (2024) e31063. <https://doi.org/10.1016/j.heliyon.2024.e31063>.
- [5] T. Dantas, J. Padrão, M.R. da Silva, P. Pinto, S. Madeira, P. Vaz, A. Zille, F. Silva, Bacteria co-culture adhesion on different texturized zirconia surfaces, J. Mech. Behav. Biomed. Mater. 123 (2021). <https://doi.org/10.1016/j.jmbbm.2021.104786>.
- [6] L.C.D.M. Dantas, J.P. Da Silva-Neto, T.S. Dantas, L.Z. Naves, F.D. Das Neves, A.S. Da Mota, Bacterial adhesion and surface roughness for different clinical techniques for acrylic polymethyl methacrylate, Int. J. Dent. 2016 (2016). <https://doi.org/10.1155/2016/8685796>.
- [7] Okkar Kyaw, M. Inokoshi, M. Kanazawa, Tribological aspects of enamel wear caused by zirconia and lithium disilicate: A meta-narrative review, Jpn. Dent. Sci. Rev. 60 (2024) 258–270. <https://doi.org/10.1016/j.jdsr.2024.11.001>.

- [8] P. Pinto, Influence of Applied Pressure and Thickness Variation on the Bond Strength Between 3Y- - TZP Zirconia and Adhesive Resin Cement, (2025) 1–10. <https://doi.org/10.1002/jbm.b.35563>.

CHAPTER 7.

General discussion and conclusions

7.1 General discussion and conclusions

The growing demand for aesthetic minimally invasive restorative treatments has driven the development of solutions that combine durability, adhesive stability, and optical mimicry with minimal or no removal of dental tissue. In this context, ultra-thin veneers made of translucent 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) emerge as a promising alternative, capable of meeting current clinical, laboratory, and aesthetic criteria.

This work developed, through a structured and multidisciplinary approach, a set of experimental studies assessing the functional viability of the clinical application of ultra-thin 3Y-TZP zirconia veneers. It integrates three main axes: wear behavior, adhesive strength, and the influence of cementation pressure and optical properties. The technical feasibility of producing these restorations via digital methods was also evaluated through a detailed analysis of the CAD/CAM workflow.

Chapter 3 assessed the simulated toothbrush abrasion resistance of four restorative materials, including 3Y-TZP zirconia. The results revealed an excellent tribological performance of zirconia, with significantly lower volumetric loss and surface roughness compared to conventional materials. After 16 years of simulated brushing, zirconia maintained a final roughness below the clinical threshold for bacterial retention ($R_a < 0.2 \mu\text{m}$), ensuring long-term aesthetic and functional stability. These results strongly support the use of 3Y-TZP for minimally invasive rehabilitations.

Chapter 4 focused on the adhesive interface between 3Y-TZP zirconia and self-adhesive resin cement. The study investigated the influence of cementation pressure and cement layer thickness on bond strength using alumina air-abrasion. A strong inverse correlation ($R^2 = 0.82$) was found between cement thickness and shear bond strength, with the best results (58.8 MPa) obtained at pressures close to 195 kPa and thin cement layers ($\sim 36 \mu\text{m}$). These findings underscore the critical importance of surface treatment and pressure control to reduce the risk of early adhesive failure.

The study in Chapter 5 demonstrated that while 3Y-TZP zirconia exhibits lower translucency at conventional thicknesses ($>0.3 \text{ mm}$), it achieves values comparable to or greater than 5Y-PSZ zirconia at thicknesses below 0.15 mm. This finding is highly relevant for ultra-thin veneers, as it

enables the combination of the superior mechanical properties of 3Y-TZP with an optical behavior suitable for anterior aesthetic applications. By demonstrating that yttria content becomes less relevant for translucency at thicknesses below 0.15 mm, the study validates the use of 3Y-TZP as a viable aesthetic alternative without compromising structural strength.

Chapter 6 aims to verify the feasibility of fabricating an ultra-thin (0.15 mm) 3Y-TZP zirconia veneer using the latest CAD/CAM technologies. A realistic anatomical dental arch model was used to fabricate a veneer for tooth 11 without any preparation. The digital workflow involved model acquisition with a Medit i500 intraoral scanner, CAD design with ExoCAD, machining parameter definition with HyperDent, and controlled sintering in a dedicated furnace. The final veneer exhibited good marginal adaptation, a final thickness close to the design (0.153 mm), and post-sintering dimensional stability. The internal surface finishing and treatment confirmed the practical applicability of the studied parameters. Although this case was performed on a model without clinical cementation, the laboratory test demonstrated that producing veneers thinner than 0.2 mm is feasible, provided that specialized CAM strategies and tools are used.

The findings validate the thesis proposal: it is possible to fabricate and apply ultra-thin 3Y-TZP zirconia veneers with thicknesses between 0.15–0.30 mm, while respecting the principles of minimally invasive dentistry and maintaining mechanical, adhesive, and aesthetic properties. However, the success of this approach depends on the strict adherence to protocols and the technical expertise of the dental team involved.

7.2 Suggestions for Future Works

Based on the results obtained throughout this doctoral work, as well as the development and validation of a complete digital workflow for the production of ultra-thin 3Y-TZP zirconia veneers, several research directions can be identified to further explore and expand the knowledge generated:

- **Investigate the thermomechanical fatigue** behavior of ultra-thin zirconia veneers by simulating real clinical conditions of mastication and long-term thermal variations (e.g., thermocycling and controlled cyclic loading);

- **Assess the aging process** (Low Temperature Degradation) of 3Y-TZP zirconia at thicknesses ≤ 0.15 mm to understand its long-term stability in the oral environment;
- **Develop clinical protocols with pressure control during cementation**, using calibrated devices to ensure uniformity in the cement layer thickness;
- **Conduct numerical modeling studies** (Finite Element Analysis - FEA) to simulate internal stress distribution in veneers with different thicknesses and geometries, enabling the optimization of design based on biomechanical criteria;
- **Evaluate the clinical performance of ultra-thin veneers in real patients** through longitudinal clinical assessments, comparing 3Y-TZP zirconia with traditional materials in terms of marginal adaptation, color stability, and survival rate.