VICTOR BABEȘ UNIVERSITY OF MEDICINE AND PHARMACY FACULTY OF DENTAL MEDICINE
DEPARTMENT 1 MD
VASILIU ROXANA-DIANA

UNIVERSITATEA DE MEDICINĂ ȘI FARMACIE VICTOR BABEȘ | TIMIȘOARA

Ph.D. THESIS

SURFACE CHARACTERISATION AND OPTICAL PROPERTIES OF DENTAL GLASS-CERAMICS RELATED TO ARTIFICIAL AGING

ABSTRACT

Scientific coordinator

PROF. UNIV. DR. POROJAN LILIANA

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KEYWORDS: glass-ceramics, optical behavior, mechanical behavior, surface characterization, atomic force microscopy, scanning electron microscopy.

INTRODUCTION

This research topic was selected due to the rapid development of ceramic materials that can be processed using conventional and subtractive computerized technologies.

All-ceramic restorations have always been a choice in the esthetical treatments, but because of their lack of mechanical strength were often replaced with metal-ceramic restorations. The intense development of these materials in restorative dentistry has led to new studies. This explains the vast research about these materials with numerous publications in prestigious journals (1).

The purpose of this research was to evaluate the benefits of these new materials brought to practitioners and patients. Glass-ceramic materials are inert and biocompatible materials in the oral environment.

Glass-ceramics and reinforced glass-ceramics materials are essential materials used in restorative dentistry due to their color stability, mechanical strength, and high biocompatibility. Their enamel-like appearance is based on their translucency and texture, which makes them the first choice in esthetics treatments (2).

Ceramic dental materials and their processing technologies were important topics for our research team at the Discipline of Dental Prostheses Technologies, and numerous articles and projects stand as proof. The extensive development of the materials motivated and challenged the team to start this research.

The novelty and rapid development of materials and technologies required further studies regarding their optical, mechanical, and adaptability of all-ceramic restorations obtained by heat-pressing and milling.

The **scientific objectives** of this research were:

1.Comparative assessment of optical properties on different types of glass-ceramics obtained by heat-pressing and subtractive CAD/CAM technologies.

2. A microstructural, topographic, and microhardness assessment of glass-ceramics processed using heat-pressing and subtractive technologies.

3. Evaluation of adaptability after thermal aging on all-ceramic crowns processed using heat-pressing and milling technologies.

This research was based on a multidisciplinary approach to evaluate these ceramic materials from the perspective of various scientific fields such as mechanics, optics, digital technologies, and surface characterization technologies.

GENERAL PART

1. CERAMIC MATERIAL

Dental glass-ceramics are nowadays widely used in restorative dentistry, due to their excellent esthetic appearance as a result of their optical properties (translucency, opalescence, and fluorescence), chromatic stability (3).

Newly developed types of ceramics equally provide excellent aesthetics but also, good mechanical qualities and biocompatibility. These properties contribute to making all-ceramic restorations superior to porcelain-fused-to-metal restorations (4).

The technological development in the dental field and especially in the ceramic area has led to obtaining restorations that have no metal, all-ceramic restorations (5).

1.1. CLASSIFICATION OF ALL-CERAMIC SYSTEMS ACCORDING TO THEIR CHEMICAL COMPOSITION AND MICROSTRUCTURE:

Microstructural, dental ceramics are composed of two phases: a glassy phase that contributes to the aesthetics, and the crystalline phase, which is responsible for the mechanical properties of ceramics. Grain size is also essential for functional ceramics because it has a direct impact on surface friction and wears behavior.

1.2. CLASSIFICATION OF GLASS-CERAMICS ACCORDING TO THEIR PROCESSING TECHNOLOGIES AND FINISHING PROCESSES:

Ceramic materials can be processed using conventional or digital CAD/CAM technologies.

Conventional technologies include powder condensation, slip casting, and heat-pressing (injection Moulding or the lost wax-technique). Heat-pressing was at first used only for leucite glass-ceramics. Lithium disilicate and zirconia reinforced lithium silicate glass-ceramics were the second generations to be processed using this process (6).

Modern technologies used to process ceramic materials consist of digital technologies, subtractive, and additive. Two methods can be used to subtractive process prefabricated CAD/CAM blocks. The first one, also known as hard milling, processes a fully sintered block to obtain the restorations. This can lead to excessive tool wear and multiple defects in the final restoration (7). The second developed method, known as green milling, utilizes only partially sintered materials. Novel processing technologies have been developed to obtain dental ceramics. Some examples of technologies used in this field: Selective Laser Sintering, Direct 3D Printing, and Stereolithography (8).

Along with the processing technologies, another important aspect is the finishing procedures of ceramic materials. Glazing and polishing influence the surface roughness of dental ceramics differently. Kawai at al., (9) discovered that glazed samples are susceptible to attract more plaque when compared to the polished surfaces. Glazing can induce rougher surfaces and irregularities, and this can lead to adhesion for the plaque. Another study (10) concluded that glazed ceramic surfaces display a rougher surface compared to the polished ones.

1.3.CLINICAL IMPLICATIONS OF ALL-CERAMIC SYSTEMS:

Glass-ceramics have different indications according to their properties and microstructure.

Glass matrix ceramics such as feldspathic and leucite-reinforced ceramics can only be used for single-unit restorations, such as veneers, inlays onlays, anterior or posterior crowns because of their low strength on flexural forces (154 MPa and 160 MPa). Zirconia reinforced lithium silicate glass-ceramic is suitable for implant-supported crowns because of its high flexural strength, 420 MPa. Restorations such as veneers, onlays, inlays, crowns, and partial crowns are the best indication for these ceramics. Lithium disilicate glass-ceramics are indicated for all the above and as well for FPD with a maximum of 3 units, due to their high crystalline phase and flexural strength (360-400 MPa).

1.3.1.ORAL ENVIRONMENT INFLUENCE ON GLASS-CERAMICS:

In the oral cavity, the ceramic restorations suffer stresses such as high and low temperatures, which can lead, in time, to crack evolution and strength loss of the material. Thermocycling could alter the failure load of the material to some degree (11,12).

The oral environment can be reproduced in vitro using standardized parameters. Usually, cycles can be between 600-50000, and a year is represented by 10.000 cycles (13).

Acidic beverages influence the optical behavior of ceramics. After a while, color changes occurred after immersing the samples in orange juice and cola, and increased opacity appeared after immersion in coffee (14). Recent studies are investigating the role of artificial gastric acid in combination with toothbrushing on surface roughness and optical properties (15).

1.3.2.CEMENTATION THE DENTAL ALL-CERAMIC RESTORATIONS:

Ceramic materials have shown a low tensile resistance, meaning that the selected cementation method has a direct impact on the fracture load of ceramic restorations.

Initially, posterior ceramic restorations were cemented using conventional zinc phosphate types of cement or glass ionomers.

Studies have proven that posterior ceramic restorations cemented with the adhesive technique have significantly higher fracture strength compared to the conventional techniques (16). Light cured resin-modified glass ionomers were designed to combine advantages of the glass ionomers and composite resins.

2. EVALUATION OF ALL- CERAMIC DENTAL RESTORATIONS

2.1. OPTICAL EVALUATION

The optical properties of ceramics are determined by composition and crystals sizes (17). Three distinct phenomena determine the optical behavior for ceramics, refraction, deflection, and total transmission of the light (18). These interactions are dependent on the wavelength of the light. The standard clinal procedure of visually taking the color can be affected by factors such as the experience, color perception defects of the evaluator, and the quality of light (19-21). To improve the color registration, shade taking devices were invented. There are three distinct categories: colorimeters, spectroradiometers, and spectrophotometers (22).

2.2. MICROSTRUCTURAL, TOPOGRAPHIC, AND MICROHARDNESS CHARACTERISATION:

There are nondestructive evaluations (NDE) that permit and encourage the applications of ceramics in all fields. Nondestructive testing is useful in detecting, locating, and measuring the defects in the ceramic structure. Defects and porosity must be measured and assessed because they can change the strength and longevity of the restorations. In general, monolithic ceramics display some defects and porosity, and the hybrid ceramic that also contains resins can display interlaminar porosity and processes induced gaps (23).

Examples of non-destructive structure analyses are computer tomography (CT) evaluation, profilometry, atomic force microscopy (AFM), scanning electron microscopy (SEM), and environmental scanning electron microscopy (ESEM).

2.3 MARGINAL ADAPTABILITY EVALUATION:

Different measuring methods were developed, and they can have an influence on the final values for the marginal adaptability of all-ceramic crowns.

Impression techniques and visual examination rely on the researcher's experience, and this is often influenced by subjectivism (24). Another aspect that influences these techniques is the demarcation line location. It is harder to objectively evaluate the marginal gap when the demarcation line is subgingivally (25). The most used method is the replica technique. It consists of using a low viscosity silicone, but it can induce inaccurate results.

The silicon impression can be damaged once removed from the die (26). Other methods consist of profilometry, scanning electron microscopy SEM), optical stereomicroscope, and micro-CT (27-30).

EXPERIMENTAL PART

3. OPTICAL BEHAVIOR OF DENTAL GLASS-CERAMIC MATERIALS RELATED TO INTRINSIC AND EXTRINSIC FACTORS

3.1.AIM OF THE STUDY

The purpose of the first in vitro study was to evaluate the effect of intrinsic factors (the type of ceramic, processing technologies) and extrinsic factors (beverages, different pH, temperature, and thermocycling) on the optical properties of dental glass-ceramics.

3.2.MATERIALS AND METHODS

To assess, the objects of the first study two types (n=16) of glass-ceramic were included in this study, a feldspathic glass-ceramic, and a leucite glass-ceramic. The samples were divided into two groups- half with 1 mm thickness and another half with 2 mm thickness.

The second study included two types of glass-ceramics (n=24) heat-pressed lithium silicate samples and feldspathic glass-ceramic samples. The ceramic specimens of each material (n=12) were randomly divided into three groups (n=4) according to the immersion solution: Tea (Black Tea, Vallery)(pH=6,5), carbonated acidic beverages (Coca- Cola)(pH = 2,5), Distilled Water (pH=7) as the testing group. The temperature was chosen to simulate the oral environment with its temperature variations.

The third study included (n= 48) heat-pressed and milled feldspathic glass-ceramics, lithium disilicate glass-ceramic, and zirconia reinforced lithium silicate glass-ceramic. A thermocycler (Thermocycler, SD Mechatronik, Germany) with distilled water basins of 5°Celsius and 55° Celsius was used. After the determination of the optical properties, the ceramic specimens were subjected to aging for 10.000 thermocycles in distilled water. Samples were aged 10.000 cycles, estimating one year in the oral environment.

3.3. RESULTS AND DISCUSSIONS

The surface treatments (glazing and polishing) and different thicknesses influenced the optical properties of glass-ceramics. Spectrophotometric reflectance showed little differences between the two materials (Figure 1).

The pH and temperature did not produce significant changes in the tested samples. Immersion in different beverages provided a significant effect on the optical properties of the ceramics; the black tea revealed greater changes in the tested samples.

The best values for the translucency parameter before thermocycling were obtained for the milled feldspathic glass-ceramic, followed by milled and heat-pressed lithium glass-ceramic.

The results for the opalescence parameter revealed that the milled zirconia reinforced lithium silicate proved higher OP values, followed by heat-pressed zirconia reinforced lithium silicate glass-ceramics.

The aging processes affected the milled ceramic significantly compared to the other ones. From both groups, ZLS glass-ceramic experienced a significant change in the values of TP and OP. The aging process altered the glazed samples in all of the samples; the glaze layer structure and roughness changed. The only exception was for the F heat-pressed glass-ceramic.

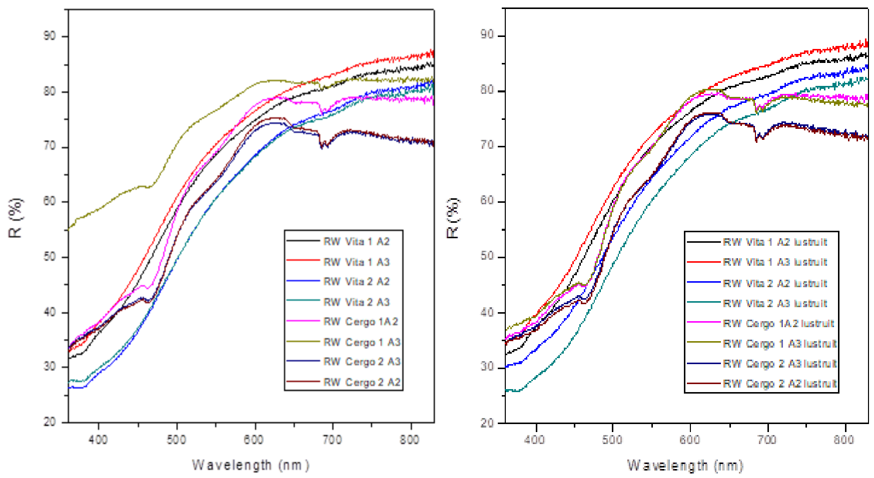

Figure 1. Spectrophotometric reflectance for the tested samples.

4. MICROSTRUCTURAL, TOPOGRAPHY AND MICROHARDNESS CHARACTERISATION OF DENTAL GLASS-CERAMIC MATERIALS RELATED TO IN VITRO THERMAL AGING

4.1.THE OBJECTIVE OF THE STUDY

The objective of these studies was to evaluate the microstructural, surface texture, and mechanical behavior of glass-ceramics obtained by heat-pressing and milling the ceramics, after immersing them in different pH beverages and after thermal aging.

4.2. MATERIALS AND METHOD

Heat– pressed glass-ceramics included in this study were: a leucite glass-ceramic, a feldspathic glass-ceramic, a lithium disilicate glass-ceramic, and a zirconia reinforced lithium silicate glass-ceramic.

The milled ceramics included in this study were: a feldspathic glass-ceramic, a lithium disilicate, and zirconia reinforced lithium silicate glass.

After obtaining the samples, these were glazed on one side and polished on the other side. All the samples were thermocycled for 10, 000 cycles, estimating one year of clinical wear. Surface roughness measurements were made on each side of the samples with a contact profilometer before and after the thermal aging of the ceramic samples.

Atomic force microscopy and scanning electron microscopy were used to characterize the surface of each sample before and after thermocycling. A microhardness tester Vicker’s was used to assess before and after thermal aging the microhardness of each ceramic samples.

4.3. RESULTS AND DISCUSSIONS

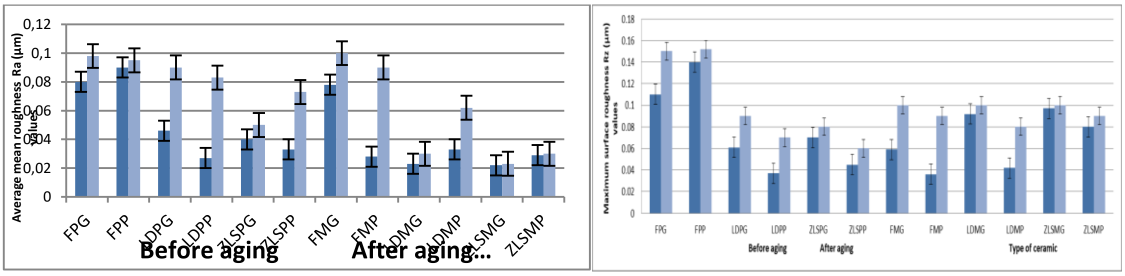

Figure 2. Obtained results– before and after thermocycling for the tested samples.

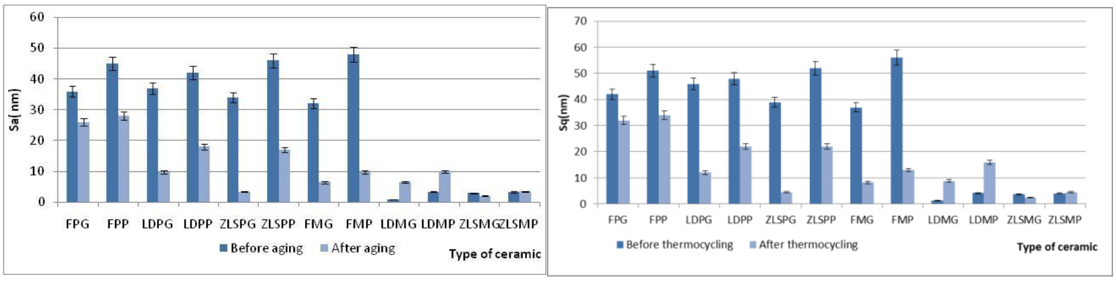

Figure 3. Obtained values before and after thermocycling for the tested samples.

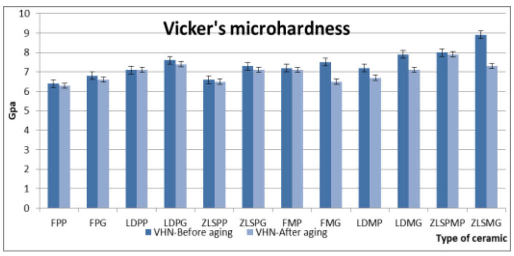

Figure 4. Obtained values for microhardness before and after thermocycling.

High values for microroughness were reported for the ceramic samples obtained by heat-pressing, especially for the glazed and polished felspathic glass-ceramic.

Thermocycling almost doubled the values for microroughness for both lithium disilicate glass-ceramics (Figure 2).

Values for nanoroughness were increased for all the tested heat-pressed glass-ceramics and decreased for the milled glass-ceramics. Thermocycling resulted in lower nanoroughness values for heat-pressed glass-ceramics (Figure 3).

High microhardness values were increased for the zirconia reinforced lithium silicate and as well for the lithium disilicate glass –ceramics, both heat-pressed and milled.

The present studies promote milled glass-ceramics with high Vickers microhardness and lower nanoroughness values. Glazing the samples is indicated for an increased microhardness (31-35).

5. FIT EVALUATION OF MONOLITHIC GLASS CERAMIC RESTORATIONS

5.1.THE PURPOSE OF THE STUDY:

The purpose of this study was to evaluate the adaptability of monolithic glass-ceramic restorations in correlation to the type of ceramic, processing technique, and in vitro thermocycling.

5.2.MATERIALS AND METHODS:

For this study, an upper first premolar was prepared, digitally scanned and 32 abutments were divided into four groups (n=8) according to the ceramic material (feldspathic glass ceramic and zirconia reinforced lithium silicate glass-ceramic) and to their technological obtaining production (milling and heat-pressing). The thirty-two abutments were obtained using additive technology (Stereolithography).

5.3. RESULTS AND DISCUSSIONS:

Before thermal aging, the best marginal and internal fit were seen in the FM crowns, followed by milled the zirconia reinforced lithium silicate crowns.

In vitro, thermocycling showed insignificant differences in the marginal and internal areas for all the tested crowns.

The present study promotes milled glass-ceramics and especially milled feldspathic ceramic for better marginal and internal fitness.

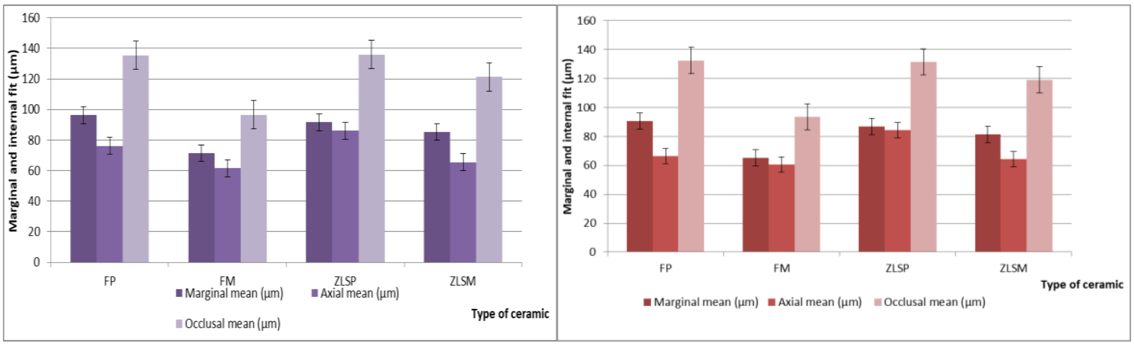

Figure 5. Obtained values-before and after thermocycling.

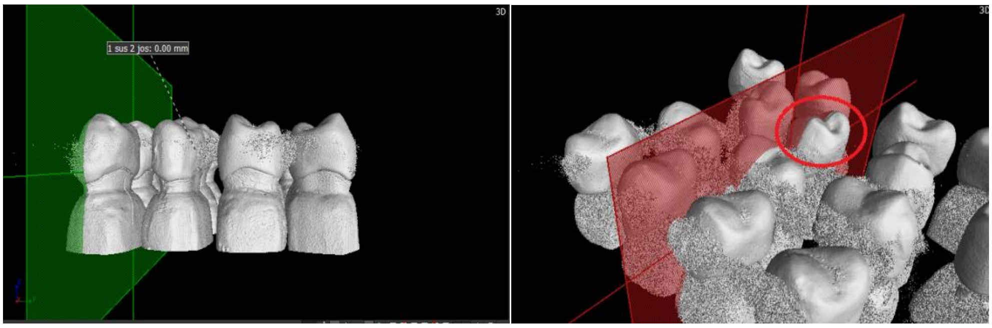

Figure 6. Micro-CT scanning of the ceramic crowns

CONCLUSIONS :

1. The first studies revealed that ceramic materials react differently to extrinsic factors based on their microstructure.

Different pH and temperatures did not influence the optical properties (translucency and opalescence) of dental ceramics after 72 hours, however, colorants found in the beverages did.

The thickness of the ceramic materials influences the optical properties of restorations, translucency, and opalescence.

Surface treatments play a significant role in the optical properties of ceramics. Glazing and polishing have a significant effect on the optical behavior of ceramics. Feldspathic ceramics display better optical properties when glazed; lithium disilicate glass-ceramic and zirconia reinforced glass-ceramics can be either polished or glazed. The thermal aging process has a significant impact on the optical behavior of milled, and mainly on the ZLSM.

2. These studies concluded that the microstructure of the ceramic materials has a significant impact on the topography and microhardness behavior of the glass-ceramic.

Thermal aging altered the milled ceramic materials significantly, increasing the surface roughness and decreasing the microhardness. The glazing layers were the first affected by the thermal aging, in comparison with the polished surface.

3. Using additive technologies for wax patterns and resin abutments represented a useful and controlled alternative to conventional manufacturing, being more precise and less time-consuming.

The technological processes influence the marginal and internal fitness of the crowns in favor of the CAD/CAM technology.

Another important aspect is that the crystalization processes after milling in partially crystallized crowns may negatively influence the marginal and internal adaptability.

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