



YEDİTEPE UNIVERSITY

INSTITUTE OF HEALTH SCIENCES

DEPARTMENT OF PROSTHODONTICS

**COMPARISON OF MECHANICAL AND OPTICAL
PROPERTIES OF DIFFERENT MONOLITHIC
CAD/CAM RESTORATIVE MATERIALS**

DOCTOR OF PHILOSOPHY THESIS

SALIMA MUFTAH AOUN, MDS, PHD

Istanbul-2021



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APPROVAL

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DECLARATION

I declare that this thesis has been written solely by myself and has not been submitted in whole or in part for any previous degree. The experimental work is entirely my own work; except where due references have been provided on all supporting literatures and resources.

28/07/2021

Salima Aoun

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DEDICATION

I wish to dedicate this PhD thesis to everyone looking for knowledge and striving to obtain it.

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LIST OF SYMBOLS AND ABBREVIATIONS

CAD/CAM	Computer Aided Design/Computer Aided Manufacturing
Y-TZP	Yttrium-stabilized tetragonal zirconia
SCs	Single crowns
FDPs	Fixed dental prostheses
MPa	Mega pascal
SiO ₂	Silicon oxide
K ₂ O	Potassium oxide
Na ₂ O	Sodium oxide
Al ₂ O ₃	Aluminum oxide
Li ₂ SiO ₃	Lithium disilicate crystal nuclei
Min	Minute
C°	Centigrade
CTE	Coefficient of thermal expansion
HT	High translucency
LT	Low translucency
USA	United States of America
ZLS	Zirconium dioxide
m-ZrO ₂	monoclinic phase of zirconium dioxide
t-ZrO ₂	tetragonal phase of zirconium dioxide

c-ZrO ₂	cubic phase of zirconium dioxide
Y-TZP	Yttrium oxide zirconia
O ₃	Trioxxygen
Y ₂ O ₃	Yttrium oxide
ZTA	Zirconia toughened alumina
PSZ	Partially stabilized zirconia
CSZ	Cubic stabilized zirconia
Wt%	Weight percent
La ₂ O ₃	Lanthanum oxide
Al ³⁺	Aluminum cation monoatomic trication
La ³⁺	Lanthanum ion
CR	Contrast ratio
TP	Translucency parameter
Nm	Nanometer
MO	Medium opaque
MT	Medium translucency
MT multi	Polychromatic multidisc
BL	Bleach shade
C17	Block size
B45	Block size
In fire HTC	High temperature furnace

In fire HTC speed	High temperature high speed furnace
ISO	International organization for standardization
Knoop test	Microhardness test
Vickers test	Microhardness test
Kg	Kilogram
N	Newton
CIE	Commission International de l'Eclairage
ΔE	Equation of color difference
EVRSAM	Extended visual rating scale
OP	Opalescence parameter
CIE a^*	Red green color coordinate
CIE b^*	Yellow blue color coordinate
W	White background
B	Black background
Sec	Second
SEM	Scanning electron microscope
ANOVA	Analysis of variance
SPSS	Software for data analysis
mm	Millimeter
μm	Micrometer

ABSTRACT

Aoun, S. (2021). Comparison of Mechanical and Optical Properties of Different Monolithic CAD/CAM Restorative Materials. Yeditepe University, Institute of Health Sciences, Department of Prosthodontics. PhD Thesis, Istanbul.

This study was conducted to compare the mechanical, optical properties and effect of thermocycling on four types of CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) monolithic restorative materials. A total of 120 specimens from IPS Empress CAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein), Celtra DUO (Dentsply, Sirona, Hana-Wolfgang, Germany), InCoris TZI C (Dentsply, Sirona, Hana-Wolfgang, Germany) and IPS e.max ZirCAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein) were used. 80 specimens with 14 mm x 1.2 mm x 4 mm dimensions for 2 subgroups with and without thermocycling were prepared for mechanical tests and sizes of 12 mm x 12 mm square shaped 10 samples from each material were prepared for optical tests before and after thermocycling. Each sample was polished and cleaned using ultrasonic cleaner. The flexural strength was determined by three-point flexural tests using a universal testing machine according to ISO 6872:2015. The surface microhardness was calculated using a Vickers hardness test device. Color change (ΔE), translucency (TP) and opalescence parameters (OP) of all tested materials were examined using spectrophotometer. Three samples from each group of material were randomly selected and evaluated under a scanning electron microscope (SEM). The statistical analysis was accomplished by SPSS (statistical package for social sciences) V.25. For flexural strength test, the effect of thermal aging was not statistically significant ($p > 0.05$), while the effect of material type was statistically significant ($p < 0.05$). The highest flexural strength value was for IPS e.max ZirCAD (1447.6 ± 286.3 MPa), while IPS Empress CAD the lowest value (155.8 ± 27.2 MPa) of flexural strength. There were statistically significant differences in surface microhardness values between material groups and subgroups ($p < 0.05$). Thermal aging Celtra DUO subgroup had a statistically significantly lower surface microhardness (VHN) value than non-thermal aging subgroup ($p = 0.001$), and IPS Empress CAD thermal aging subgroup had a statistically significantly higher VHN value than non-thermal aging subgroup ($p = 0.000$). There was a statistically significant difference in ΔE between material groups with thermal aging ($p = 0.000$). After thermal aging, ΔE was the highest in IPS Empress CAD (2.78 ± 1.21). Thermocycling led to statistically significant color change on all materials except for InCoris TZI C ($p < 0.05$). Regarding the translucency parameter (TP) and opalescence parameter (OP), the effect of thermal aging was not statistically significant for any groups [TP ($p = 0.664$), OP ($p = 0.502$)]. The highest TP value was for IPS Empress CAD (20.73 ± 0.79) and highest OP value was for Celtra DUO (12.97 ± 0.34). Monolithic zirconia ceramics have higher mechanical properties than glass ceramic materials. The reinforced glass ceramic material has superior optical properties than monolithic zirconia ceramics. The OP value for all tested groups showed lower value than criteria for natural tooth OP values.

Key Words: Mechanical properties, flexural strength, surface microhardness, optical properties, translucency, opalescence, color change, leucite reinforced glass ceramic, zirconia reinforced lithium silicate, monolithic zirconia, monolithic CAD/CAM materials.

ÖZET

Aoun, S. (2021). Farklı CAD/CAM Restoratif Materyallerinin Mekanik ve Optik Özelliklerinin Karşılaştırılması, Yeditepe Üniversitesi Sağlık Bilimleri Enstitüsü, Protetik Diş Tedavisi AD., Doktora Tezi, İstanbul.

Bu çalışma dört farklı CAD/CAM (Bilgisayar Destekli Tasarım/Bilgisayar Destekli Üretim) monolitik restoratif materyalin mekanik ve optik özelliklerini karşılaştırmak üzere yürütülmüştür. IPS Empress CAD (Ivoclar Vivadent, AG, Schaan, Lichtenstein), Celtra DUO (Dentsply, Sirona, Hana-Wolfgang, Germany), InCoris TZI C (Dentsply, Sirona, Hana-Wolfgang, Germany) ve IPS e.max ZirCAD (Ivoclar Vivadent, AG, Schaan, Lichtenstein) monolitik restoratif materyallerinden toplam 120 numune hazırlanmıştır (n=10). Mekanik testler için termal yaşlandırmaya maruz bırakılan ve bırakılmayan 2 alt grup için 14 mm x 1.2 mm x 4 mm boyutlarında toplam 80 numune, optik testler için ise her bir materyalden 12 x 12 mm boyutlarında kare şeklinde 10'ar numune hazırlanmıştır, termal yaşlandırma öncesi ve sonrası olarak kullanılmıştır. Her bir numune cilalandıktan sonra ultrasonik olarak temizlenmiştir. ISO 6872:2015 doğrultusunda üç noktalı eğme testleri ile eğilme dayanımı değerleri hesaplanmıştır. Vickers sertlik test cihazı ile yüzey mikrosertlik değerleri ölçülmüştür. Test edilen tüm monolitik restoratif materyallerin renk değişimleri (ΔE), translüsensi (TP) ve opaklık parametreleri (OP) spektrofotometre kullanılarak incelenmiştir. Her materyal grubundan rastgele üç numune seçilmiş ve yüzeyleri taramalı elektron mikroskobu (SEM) altında değerlendirilmiştir. Verilerin istatistiksel analizi SPSS (statistical package for the social sciences) V.25 programı kullanılarak gerçekleştirilmiştir. Eğilme dayanımı testi için termal yaşlandırmanın etkisi istatistiksel olarak anlamlı değilken ($p>0.05$), materyal türünün etkisi istatistiksel olarak anlamlı bulunmuştur ($p<0.05$). IPS e.max ZirCAD materyali en yüksek eğilme dayanımı değerini gösterirken (1447.6 ± 286.3 MPa), IPS Empress CAD en düşük değeri göstermiştir (155.8 ± 27.2 MPa). Materyal grupları ve alt gruplar arasında yüzey mikrosertlik değerlerinde istatistiksel olarak anlamlı fark olduğu görülmüştür ($p<0.05$). Celtra DUO materyalinin termal yaşlandırmaya maruz bırakılan alt grubunun yüzey mikrosertliği, termal yaşlandırmaya maruz bırakılmayan alt grubuna göre daha düşük bulunmuşken ($p=0.001$); IPS Empress CAD materyalinin yüzey mikrosertliği ise termal yaşlandırılmaya maruz bırakılan alt grubunda termal yaşlandırmaya maruz bırakılmayan alt grubuna göre daha yüksek bulunmuştur ($p=0.000$). Her iki zirkonyum materyalinin yüzey mikrosertliklerinde termal yaşlandırma öncesi ve sonrasında istatistiksel anlamlı fark olmamıştır ($p>0.05$). Termal yaşlandırma uygulanan malzeme grupları arasında ΔE açısından istatistiksel olarak anlamlı fark bulunmuştur ($p=0.000$). Termal yaşlandırma sonrasında IPS Empress CAD en yüksek ΔE değerini göstermişken (2.78 ± 1.21). InCoris TZI C haricinde tüm materyallerde termal yaşlandırma istatistiksel anlamlı renk değişimi oluşturmuştur ($p<0.05$). Termal yaşlandırmanın materyallerin translüsensi parametresi (TP) ve opalesans parametresi (OP) üzerine etkisi istatistiksel olarak anlamlı bulunmamıştır [TP ($p=0.664$), OP ($p=0.502$)]. IPS Empress CAD en yüksek TP (20.73 ± 0.79), Celtra DUO ise en yüksek OP değeri (12.97 ± 0.34) gösteren materyallerdir. Monolitik zirkonya materyaller monolitik cam seramik materyallerden daha üstün mekanik özelliklere sahiptir. Güçlendirilmiş cam seramik materyaller, monolitik zirkonya seramik materyallerden daha üstün optik özelliklere sahiptir. Test edilen tüm gruplar için OP değeri, doğal diş için belirlenmiş OP değeri kriterinden daha düşüktür.

Anahtar Kelimeler: Mekanik özellikler, eğilme dayanımı, yüzey mikrosertliği, optik özellikler, translüsensi, opalesans, renk değişimi, lösitle güçlendirilmiş cam seramik, zirkonya ile güçlendirilmiş lityum silikat, monolitik zirkonya, monolitik CAD/CAM materyalleri.

1. INTRODUCTION AND PURPOSE

The main goal of restorative dentistry is producing a natural-looking appearance together with appropriate mechanical properties for the patients.¹ The loss of teeth associated with many complications related to the function and aesthetic of the oral cavity especially the missing of anterior teeth.² To overcome these complications much treatment like single crowns, multiunit fixed prostheses, and implant-supported crowns options has been indicated to obtain good functional and aesthetic results and improve the life quality of the patient.³

The durability of restorations is critical for clinical dentistry, in as much mechanical failures have financial and biological concerns for the patient and dentist. Therefore, choosing the best restorative materials with mechanical durability is depending on the results of previous in-vitro studies.⁴

There was a big revolution in dentistry started by the end of the 20th century due to the introduction of new technology such as the Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) systems.⁵

The concept of using this latest dental technology, particularly in field of restorative dentistry, supported by varied research approaches, many systems respectively introduced to the market using optical impressions, from which milling and designing of a restored tooth and teeth were possible.⁶

This technology cause a huge changes in the clinical dentistry by allowing dentists to deal with ceramic restorations such as full crowns, inlays, onlays, and veneers in a clinic and laboratory faster with highly performed restorative materials.⁷

Ceramic restorative materials are being advanced as highly strength dental materials for prostheses, and due to the progress of digital chairside milling with new rapid-sintering

technology, the outputs of the dental prosthesis are becoming more mechanized, time conserving and achieve superior esthetics with long term durability.⁸

Further, the dental materials scientists have made superb efforts to produce dental restorative materials imitating both the strength and color of natural teeth.⁹ All that effort results in the production of various types of ceramics for example; feldspathic ceramics, leucite reinforced, lithium disilicate, fluoroapatite glass ceramics, zirconia, monolithic zirconia based materials and hybrid ceramics.^{10,11}

All ceramic restorative materials including different types of zirconia and zirconia reinforced ceramic are become the main alternative to metal-ceramic restoration and this is attributed to their biocompatibility and esthetic properties which are improving recently.¹⁰

Among all these types of ceramic dental materials, yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) have been used as high strength ceramic material for many restorative options.¹²

The fast improvement in Y-TZP restorations has been seen during the last thirty years, Y-TZP restorations have become recently used on a routine work for tooth/implant-supported single crowns and multi-unit restorations.¹³ Due to their noteworthy biocompatibility, strength, toughness, and optical properties also the latest advances in (CAD/CAM) computer-aided design and computer-aided machine technology have supported its rapid spread in the dental field.¹² In spite of high rates of clinical success associated with Y-TZP restorations, clinical failure, still noted due to chipping of porcelain veneer in the case of framework veneered zirconia ceramics.^{14,15}

As a result of this tremendous development in the field of dentistry, many new types of ceramic materials have been introduced in the market with competitive properties especially monolithic zirconia which combines superior mechanical and optical properties at the same time.³

Most of the zirconia restorations have low translucency especially when there is a natural tooth adjacent to the anterior zirconia crown which affects the light scattering.¹⁶

In bilayer restoration the porcelain veneer thicker to cover the opacity of the zirconia core as an effort to simulate the color of natural teeth. Furthermore, the clinical research has disclosed that the zirconia bilayer restorations showed low fracture resistance, chipping and delamination of porcelain veneers that cover it.¹⁶

To avoid all these serious problems like veneer chipping and delamination and to preserve tooth structure the monolithic zirconia is utilized in most tooth restorations as full crowns and FDPs, although the monolithic zirconia still has a degree of opacity not suitable for anterior teeth.¹⁶

The main reasons for favoring the monolithic zirconia over the framework veneered were the higher reliability, fewer expenses, no chipping or cracking of veneering, less thickness lead to less invasive tooth preparation. Thanking for all these reasons, monolithic fixed restorations made from new translucent zirconia materials have been recommended as a suitable alternative that might offer a solution to those complications.³

From another hand, the main shortage of most dental restorative ceramics was their brittleness which restricted it is indication only for anterior teeth restorations.¹⁷ While 3Y-TZP ceramics are strong, and aging-resistant due to the effect of transformation toughening of sub-micrometer-sized tetragonal zirconia.¹⁸ It can be used in the molar area with high-load.¹⁹

Taking into consideration, that the selection of restorative material according to the mechanical properties also need more interest while, so many different types of zirconia restorative materials with improved strength ranging from 600 MPa to 1400 MPa.²⁰

Recently, with the huge development of different types of monolithic restorative materials with different chemical compositions, optical, and mechanical properties; the proper selection of restorative materials is a big dilemma due to the wide difference in their properties depending on their compositions and manufacturing procedures.²¹

To enhance the mechanical behavior of feldspathic porcelain, lithium-disilicate glass-ceramic was introduced and recommended as an alternative veneering material for Y-TZP.²²

Although, in cases with a highly aesthetic requirement the new translucent zirconia ceramic can be used, while in the case of FDPs with long spans where more strength is needed the translucent zirconia not sufficient.²¹

The main aim of all comparative studies is to provide assistance for the dentist to correctly utilize the glass-ceramic and zirconia ceramic material for proper indications.²³

The aims and purpose of this study are:

1. To compare the mechanical properties (flexural strength and surface microhardness) of four different CAD/CAM monolithic restorative materials (two types of monolithic zirconia, a zirconia reinforced lithium silicate ceramic and a leucite reinforced glass ceramic) with and without thermocycling.
2. To compare the optical properties (color difference, translucency and opalescence) of these four different CAD/CAM restorative materials before and after thermocycling.
3. To evaluate the effect of thermocycling on the mechanical and optical properties for the different tested monolithic CAD/CAM restorative materials.

The null hypotheses stated that:

1. There will be no difference among four different monolithic CAD/CAM materials' mechanical properties (flexural strength and surface microhardness) with and without thermocycling.
2. There will be no difference among four different monolithic CAD/CAM materials' optical properties (color difference, translucency and opalescence) before and after thermocycling.
3. There is no effect of thermocycling on the mechanical properties (flexural strength and surface microhardness) and optical properties (translucency and opalescence) for tested monolithic CAD/CAM restorative materials.



2. LITERATURE REVIEW

2.1. History of Dental Ceramics

About more than three decades ago the metal-ceramic restorations were introduced and have continued up today.²⁴ It considered a gold standard for the replacement of teeth due to its durability and reasonable esthetics. While the need for the best esthetics results was the driving force behind the emerging of all-ceramic restorations. Also, the concern about the metal biocompatibility was a mean factor for ceramic outperformed.²⁵

McLean in 1965 introduced the aluminous porcelain all-ceramic, it was the first material and the key for the more advanced ceramic restorative materials with improved esthetics and mechanical properties. But, the most common complications for traditional all-ceramic restorations were the brittleness, low fracture toughness and low tensile strength causing potential cracks under masticatory stress.²⁶

Several studies investigate the optical and mechanical properties in order to improve clinical indications and overcome the main clinical complications for all-ceramic material. Those studies include the physical properties, method of fabrications, and sintering temperature.²⁷

In the last decade, all-ceramic restorations gained more popularity due to their notable developments in the esthetic, biocompatibility, and mechanical properties.²⁸ These improvements lead to a great extension of their clinical applications from single-unit restorations to multiple-unit restorations and implant abutments, for enhance the material resistance to the force of mastication many improvements related to the material composition as the reductions of the glassy matrix for more crystalline content have been made. But unfortunately, this procedure improved the flexural strength and associated with low translucency of the materials.²⁹ However, other physical properties play an important role in dental ceramics' success, like cementation procedure, the thickness of the restorations, and surface roughness which participated to determine the mechanical behavior of the dental ceramics.³⁰

2.2 Classification of Dental Ceramics

Depending on the introduction of many different ceramic materials, many classifications have been emerged for these materials according to their microstructures or method of fabrication. Also, classification of dental ceramics materials may be made depending on the fusion temperature, fabrication method, crystalline phase, and hydrofluoric acid sensitivity or etching procedure.³⁰

Generally, all-ceramic restorative materials can be manufactured by four main methods or techniques including slip-casting technique, gradually powder liquid condensation, and (CAD-CAM) computer-aided design /computer-aided manufacturing technique.¹⁹

The classification according to the microstructural phases of the materials where all-ceramic restorative materials can be classified as: predominantly glass-based, glassy-crystalline, crystalline-based with glass fillers and polycrystalline.³¹

Various classifications have been introduced for dental ceramic depended on many factors. Chemical composition, microstructure, mechanism of fabrication, toughness, strength and clinical indications.^{16,26}

Kelly and Bennett²⁴ classified ceramic material depending on the content of glass as following:

- Predominantly glassy material
- Particle filled glasses
- Polycrystalline ceramics without glass.

Gracis et al.³² mentioned a new method ceramic restorative materials classification by dividing them into three categories according to the presence of particular elements in their components as following.

- **Glass-matrix ceramics:** nonmetallic, inorganic ceramics including glass materials.
- **Polycrystalline ceramics:** nonmetallic inorganic ceramic materials without any glass phase.
- **Resin-matrix ceramics:** polymer-matrices containing mainly inorganic, porcelain, glass, ceramic and glass- ceramic pottery refractory compounds.

The glass-matrix ceramics category is subdivided again into three subgroups: Naturally feldspathic ceramics, synthetic ceramics, and glass infiltrated ceramics.³² Polycrystalline ceramics are also subdivided into four subgroups: alumina stabilized zirconia, zirconia-toughened alumina, and alumina toughened zirconia. The third group is resin-matrix ceramics, which subdivided into multiple subgroups related to their composition.³² (Figure 2.1)

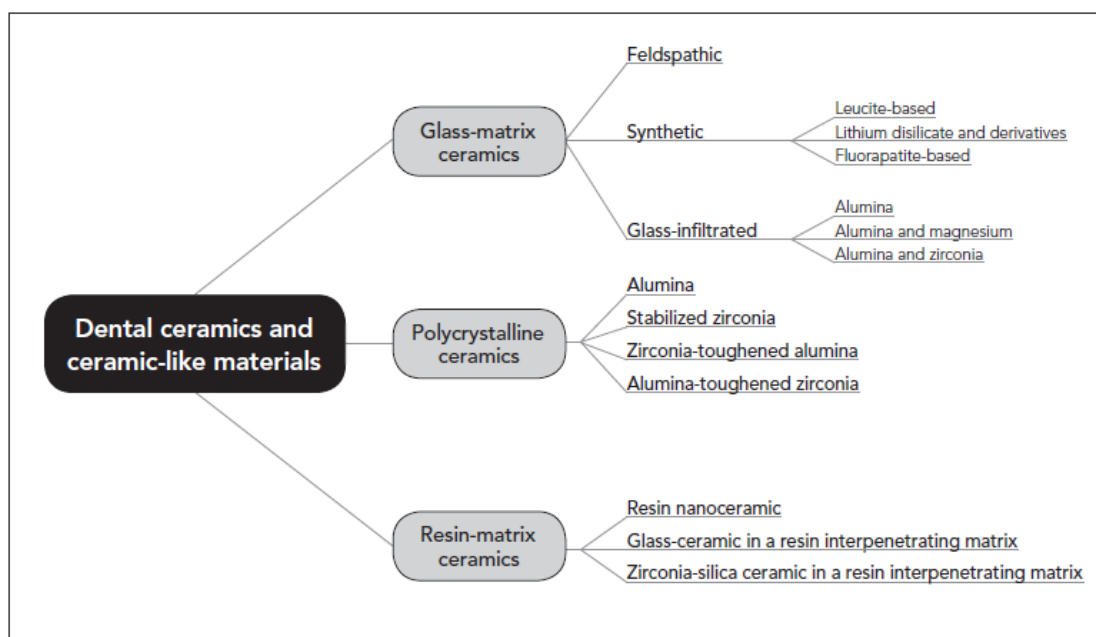


Figure 2.1: Classification system of all ceramic and ceramic like materials.³²

2.2.1 Glass matrix ceramics

2.2.1.1 Feldspathic ceramics

Feldspathic porcelains belong to the glass matrix ceramic family, it is an amorphous (a non-crystalline traditional type of ceramics composed of feldspar which occurs in nature) (a mixture of sodium and potassium alumina silicate), clay (hydrated alumina

silicate), and quartz (silica). the potassium feldspar produces a crystalline phase by forming leucite crystals which increase the strength of the restorations and make it suitable for veneering metal substructure, these materials have been used as veneering for ceramic metal restorations or directly bonded onto tooth surface for better esthetics.^{33,34} The aesthetic of feldspathic ceramic is high and its fracture strength ranges from 50 to 120 MPa.³⁵

Glass-ceramic materials for dental restorations are manufactured by the reinforcement of the glass matrix of the alumina silicate with various crystals such as leucite, lithium disilicate or fluoroapatite. Reinforced ceramic can be attained by heat-induced crystallization of glass or by dispersion strengthening inducing growth of crystals and this procedure called creaming leads to an increasing the strength of glass material without prejudice to the optical properties.³⁶

Feldspathic porcelain and leucite reinforced glass-ceramics are largely used in clinical cases with high esthetic demand as a veneering material in bilayer restoration and as a monolithic restorations for anterior laminate veneers, crowns and also as bridges for three unit from anterior to the second premolar region .^{37,38,39}

2.2.1.2. Synthetic glass matrix ceramics

This group includes lithium disilicate and derivatives (e.g., IPS e.max CAD and IPS e.max Press, Ivoclar Vivadent; Celtra DUO, Dentsply Sirona) which are not dependent on the natural raw materials, and their composition mainly include silicon oxide (SiO_2), potassium oxide (K_2O), sodium oxide (Na_2O_3) and aluminum oxide (Al_2O_3).³²

The ceramic's glass phases contain apatite crystals and leucite to improve strength and thermal expansion compatibility with metal, feldspathic porcelains reinforced by crystalline which also has role for enhancing mechanical properties and used as a substructure material .³²

Leucite-reinforced (IPS Empress, OPC) and lithium disilicate-reinforced (IPS Empress II, IPS e.max CAD) and press glass ceramics are most common materials in this group. The flexural strength of leucite-reinforced glass-ceramic varying from 120 MPa to 180 MPa and not more than $1.5 \text{ MPa.m}^{1/2}$ for fracture toughness.^{38,39} The flexural strength reinforced by refining crystal size and increasing number of crystal in lithium disilicate glass matrix, increasingly from 360 to 500 MPa for flexural strength with about $3 \text{ MPa.m}^{1/2}$ for fracture toughness.⁴⁰

2.2.1.2. A. Lithium disilicate

Lithium disilicate (LS₂) is particle-filled glass material within the glass-ceramic family that has been introduced to the market with the commercial name (IPS Empress II) Ivoclar Vivadent, which is available as ingots for heat pressing technique for fabrication of core materials veneered with translucent fluoroapatite embedded in glassy matrix ceramic to simulate natural teeth color.⁴¹

In 2005 IPS e.max (Ivoclar Vivadent) was marketed with the new formula of LS₂ showing improvement in the mechanical and optical properties higher than that of an old version, this improvement explained by the tightly interlocking of disilicate crystals obstructing the spread of crack and at the same time, the difference in thermal expansion coefficient of a glassy matrix enhance the compressive strength around the crystal.⁴²

Recently, the widespread of CAD/CAM technologies lead to the displaying of many restorations by milling devices for chairside procedures (eg, IPS e.max CAD).⁴³

Partially, IPS e.max CAD pre-crystallized blocks, are fabricated as a blue block containing lithium disilicate crystal nuclei (Li_2SiO_3) with 40% metasilicates (Li_2SiO_3); this composition gives moderate flexural strength lead to higher cutting efficiency with the lower wearing of milling tools.⁴⁴ After milling in the pre-crystallized phase at (840-850°C for 10 min) the metasilicate crystals changed to lithium disilicate (70%) which enhance the mechanical characteristics, and for optical properties the blocks available in

different shades with different translucency by diffusing staining ions in the glass matrix with varied size and distribution of the crystal.⁴²

IPS Empress CAD (Ivoclar Vivadent; Schaan, Liechtenstein) this material composed of leucite-reinforced ceramic and available as monochromatic blocks as well as polychromatic blocks with the progression of translucency to match the transmission of natural teeth color from cervical to incisal.^{45, 46}

IPS Empress CAD is a leucite-reinforced high-performance blocks created for the CAD/CAM system, it is suitable for esthetically, monolithic single-tooth restorations using CAD/CAM system. It has a balanced chameleon effect with good homogeneity and light scattering behavior, on top of splendid optical properties. For its average biaxial flexural strength, 185 MPa ceramic blocks are more distinct.⁴⁷

IPS Empress CAD blocks are manufactured from a glassy-ceramic matrix which consists of both glass and crystal phases reinforced by leucite. Leucite crystals with a few microns equally grow from the amorphous glass phase into multi-stage mode. The manufacture of the blocks accomplished by pressed the powder-shaped semi-finished product in a fully automated manner, a high difference in the thermal expansion coefficient (CTE) between the crystal phase and the glass phase produced by this method, this procedure affect the compressive strength of material after sintering cause increase of the strength of IPS Empress CAD to fulfill a medium flexural strength about 185 MPa.⁴⁷ This fabrication method made that type of material occupied a prominent place in the market with distinction for a decade. The milled restorations from IPS Empress CAD displayed featured reliability of fit and an identical surface structure.⁴⁷ (Figure 2.2)

<u>Standard composition:</u>	(in weight %)
SiO ₂	60.0 - 65.0
Al ₂ O ₃	16.0 - 20.0
K ₂ O	10.0 - 14.0
Na ₂ O	3.5 - 6.5
Other oxides	0.5 - 7.0
Pigments	0.2 - 1.0

Figure 2.2: Standard composition of IPS Empress CAD⁴⁸

2.2.1.2. B. Celtra DUO (Zirconia-reinforced lithium silicate)

Most of non-zirconia based dental ceramics restorative materials, take into consideration feldspathic porcelain and glass ceramics, provide high esthetic results when the light transmitted during substructure of non-discolored tooth bonded to restoration ceramics.^{49,24}

Also, these types of materials are considered more color stable and less worn than composite resin materials.⁵⁰ Yet, ceramics are still considered brittle materials and susceptible to fatigue fracture after cyclic loading, that is why the materials scientists have invented new ceramics reinforced by zirconia particles.⁵¹

Zirconia-reinforced lithium silicates Celtra DUO (Sirona Dentsply) is an example of high strength CAD/CAM ceramic was introduced in 2012 as a zirconia-reinforced lithium silicate (ZLS) CAD/CAM material which can be optional heat treated. The microstructure of this material contains a unique composition of ultra-fine glass-ceramic crystals (I.e., <1.0µm), reinforced with Zirconium dioxide (10%) in an amorphous glass matrix, this fine-grained microstructure increase the material strength, allow the material to be easily machined.⁵²

Celtra DUO provided in a fully crystallized state that may be either hand-polished or glazed in a ceramic furnace before delivery with higher flexural strength for glazed one, even though, heat treatment is not mandatory for the crystallization of the material, the flexural strength of glazed Celtra DUO has been declared to be significantly greater than the milled material about 370 MPa.⁵²

This type of high strength ceramic material with good translucency recently becomes the most popular for the fabrication of natural-looking chairside monolithic restorations. It is used for inlays, onlays, veneers, and crowns. Also, available in specifically designed blocks for short-span fixed partial dentures and the restoration of dental implants.⁵⁰

The implying of 10% zirconium oxide sponsors its especially high strength. The formed crystallites are 4 to 8-fold smaller than conventional lithium disilicate crystals. This structure provides a very fine microstructure, which combines a high flexural resistance with high content of glass and at the same time gives the material better optical and mechanical properties^{52,53} (Figure 2.3).

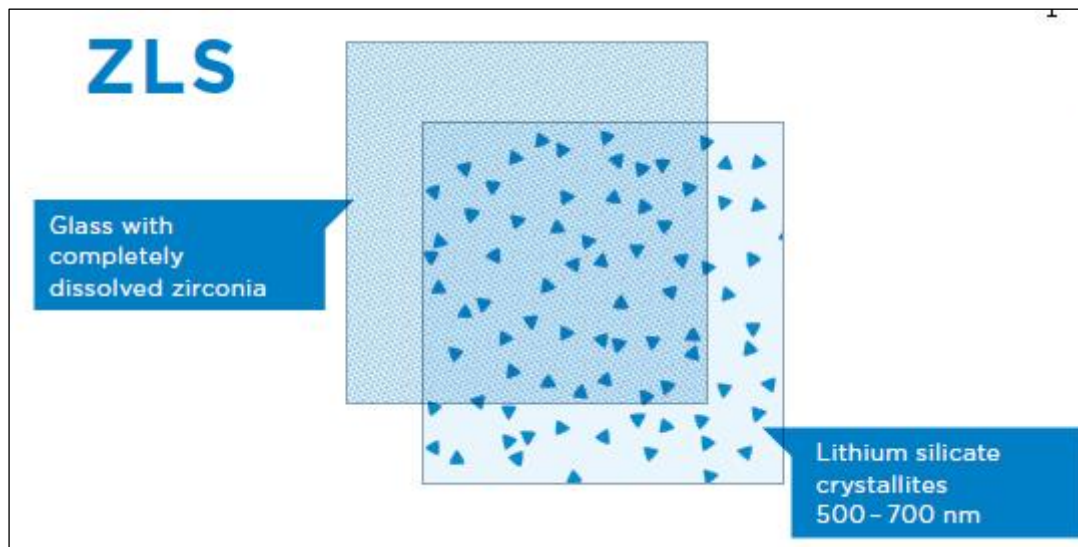


Figure 2.3: Microstructure of Celtra DUO

Celtra DUO (ZLS) has many advantages regarding its high level of strength with strength stability in long-term in chewing simulator. Even after 6,000 (41° F to 131°F) thermal cycles and 1.2 million (70 Newton) chewing cycles, the edge is excellent and restored with robust and lasting margins that can withstand chipping. At the same time, it got excellent optical properties due to its unique lithium silicate crystals composition with 500-700 particle size participate in an opalescence that like natural tooth enamel.⁵⁴

This effect act with high light conductivity and color adaptation of Celtra DUO to the rest of tooth structure create a chameleon effect. For example, full contour crowns are practically not recognizable among adjacent natural teeth.⁵⁴

2.3. Zirconia

As a crude material, in proportional manner zirconium is a soft, ductile, shiny, and silvery metal. In dentistry, they used the zirconium dioxide (ZrO_2), known as zirconia, which is achieved after complex and comprehensive procedures.³² After processing of refinement the zirconium available as high fusing white crystalline powder.⁵⁵

Mainly, zirconia is found in several forms of metal oxide and present in three crystal phases as monolithic, tetragonal, and cubic, showing continuous phase transformations with changing of their temperature.^{12, 56}

Starting from room temperature to approaching temperature of 1170°C, zirconia exhibits a monoclinic phase (m-ZrO₂). From this degree of temperature up to (2370°C) it transforms into a tetragonal crystal design (t-ZrO₂); and it keeps it forms until achieving the temperature of melting then transforms to the configuration of cubic crystal (c-ZrO₂); the cubic phase crystallizes out at 2680°C temperature.^{57,58}

The transition into the monoclinic phase, where zirconia is obtainable at room temperature, eventually takes place at 1170°C. This transformation is called martensitic transformation which is from tetragonal to the monolithic phase and is characterized by an increase in the volume of about 4%. This increase in the volume leads to exceedingly high tension and producing unwanted cracks in the ceramic structure⁵⁵ (Figure 2.4).

This dimensional change takes the energy away from cracks and their growth stops, this phenomenon is called transformation toughening. From another hand, the compressive stress developed among the particle can provide extra inhibition to the crack's growth. The same thing is found in human teeth which contain numerous cracks in the enamel structure that are blocked by the matchless interface at dentine enamel junction and crystal microstructure of enamel.⁵⁹

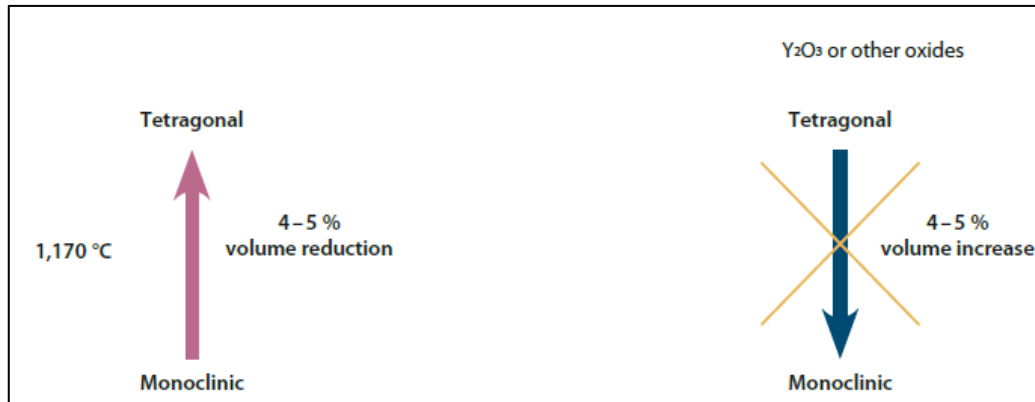


Figure 2.4: Zirconia phase transformation⁵⁵

Two different mechanisms cause toughening transformation. One is, a spontaneous tetragonal to the monoclinic phase local transformation can induce the formation of very fine micro-cracks, caused by the more voluminous monoclinic crystal form. The spreading crack either gradually disappears into these micro cracks or is deviate to the zirconia particles.^{55,60}

Otherwise, this transformation can also bring out by the high tensile stresses which are developed at the tip of a growing crack. The tensile stresses cause reduction in the pressure of the matrix on the zirconia particles which surrounding the crack leads to the transformation of the tetragonal form to the monoclinic form. This growth in the volume of the monoclinic crystal causes local compressive stress at the tip of the crack so this inhibits the further growth of the crack. This is called transformation behavior⁵⁵ (Figure2.5).

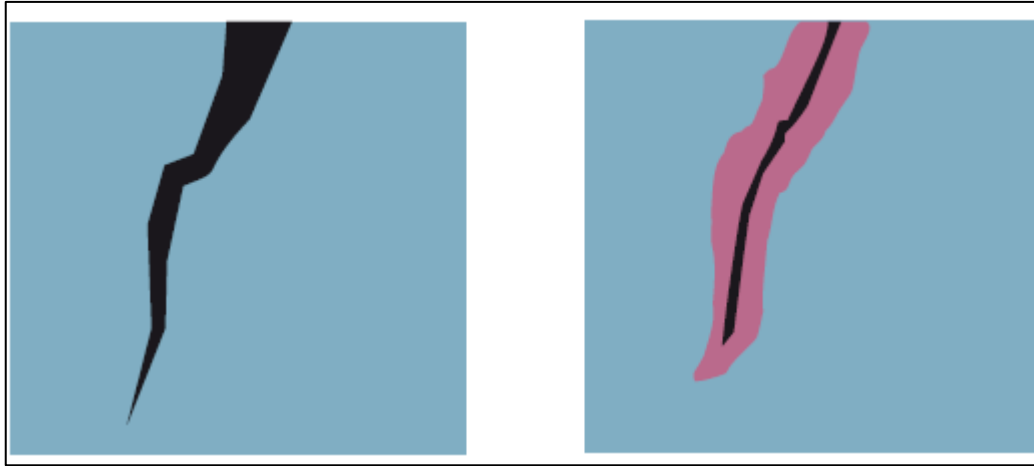


Figure 2.5: Behavior of zirconia cracks

To prevent the martensitic transformation during the cool phase of zirconia, stabilizing oxides adding to the zirconia which are integrated into the crystal lattice of the zirconia components.²¹

The most commonly used zirconia on the market is stabilized by adding yttrium oxide (Y-TZP). In the new-generation of zirconia there is increase in the proportion of yttrium oxide leads to the formation of the stable tetragonal phase and the cubic parts of the zirconia simultaneously. This heterogeneous form is recognized as fully stabilized zirconia and embody the third generation, which is, as opposed to the first and second generation partially stabilized zirconia where there is not structuring phase's transformation takes place under induced stresses, according to the manufacturer information.^{21,61}

2.3.1. Modifications of dental zirconia

The main goal of extra developing zirconia materials was to produce monolithic restorations that achieve greater translucency in addition to its high mechanical properties, and for that purpose different modifications of the material composition could be used:

- Addition of glass phase and sintering additive to enhance the zirconia structure.

- Refinement of zirconia microstructure to prevent interference of grain boundaries with passing light.⁶²
- Increase the grain size to decrease the boundaries number.
- Inventing material with a cubic phase that has isotropic optical properties.^{63,15}

Currently, zirconia can be categorized into four generations depending on their mechanical and optical properties.⁶⁴

2.3.1.1. First generation (3Y-TZP)

This material is partially stabilized tetragonal phase, introduced to the market about 20 years ago with high mechanical properties and distinguished biocompatibility, and used generally for a framework material and hybrid implant abutments. Also it can be recommended for three units anterior and posterior restorations with very good survival rate. But, due to its less translucency and high opacity the first generation contraindicated as monolithic restorations especially in the anterior teeth region.²¹

To enhance the optical properties of the first generation the material scientists increased the sintering temperature up to 1,600 C° which lead to a decrease in the flexural strength resulted in a spontaneous fracture and reduced reliability.^{21,65} For that reason, it is no longer widespread in the market.⁶⁶

2.3.1.2. Second generation (3Y-TZP)

A new version from zirconia with reduced alumina content where the size and number of the alumina grains (Al_2O_3) modified. High level of translucency was achieved by decrease O_3 and placed the alumina grains at the zirconia grain boundaries causing a higher transmission of the light, simultaneously the flexural strength remained high.^{67,68} With better optical properties and long-lasting strength this version of zirconia mostly used as framework materials for partially veneered restorations and full veneered one unit and multi-unit restorations.⁶⁹

2.3.1.3. Third generation (5Y-TZP)

In the year 2015 a new type of zirconia material introduced with cubic–tetragonal microstructure informally called cubic zirconia, this type evolved by an increase of the yttrium (Y_2O_3) content 5 mol %. The material appears more translucent because it contains 50% cubic microstructure with cubic crystals bigger than tetragonal crystals, so the light transmission through the material passes small porosities and boundaries causing refractions, as a result, the material becomes more translucent. By increasing the amount of the cubic crystals up to 53% zirconia obtains more translucency, however, the higher content of cubic crystal has a negative effect on fracture toughness and flexural strength for that reason, the most third-generation zirconia recommended for the production of single-unit restoration and FDPs up to three units in the premolar teeth regions.^{21,70}

2.3.1.4. Fourth generation (4Y-TZP)

In 2017 scientists have made more modifications to the third generation of zirconia, for the purpose of improving the properties of zirconia restorations and increase the range of indications, they reduced the yttrium content to 4 mol%, which improved the mechanical properties causing a decrease optical properties of the material. Therefore, fourth-generation zirconia is limited to the use in multi-unit FDPS of short spans only.¹⁵

2.3.2. Recent modifications of zirconia

Multilayered or gradient color blocks are available for all generations of zirconia restorations to simulate the natural color of human dentation.⁷¹

Moreover, they produced blanks that merge different zirconia generations in one product (multilayer) which combine the best mechanical and optical properties, and consist of a body formed from fourth-generation zirconia (4Y-TZP) for more stability, and incisal portion made from the third generation (5Y-TZP) for more translucency.⁷⁰

More combination from a different manufacturer, incorporating fluorescent particles in some zirconia blanks, and speed or super speed sintering is another trend of modification has been introduced recently.⁷⁰

Recently, the speeding up of the sintering process (speed or high speed sintering) is done according to the manufacturing instructions, and it depends on the restoration shape and size.¹⁵

2.3.3. Conventional and veneered zirconia

There are different brands of zirconium-dioxide materials according to the difference in their chemical compositions.⁷² These materials available for multiple dental applications and they are produced by several reinforcement methods, and varying crystal configurations for enhancing the optical and mechanical properties.⁷³

Also, many following zirconia generations of restorative materials, beginning with utilizing zirconia toughened alumina (ZTA), going to partially stabilized zirconia (PSZ) and then traditional (3YTZP) then recently, production of many highly translucent zirconia materials including cubic stabilized zirconia (CSZ).^{74,3}

The first generation from zirconia is known as conventional zirconia which is tetragonal partially stabilized zirconia. Conventional zirconia has a very small crystal structure, lead to a high number of interfaces and high light refraction index this creates a material with an opaque character.³

Two different types of zirconia blanks were introduced to the marked dyed and non-dyed. The dyed one simulates the teeth color but, the non-dyed have white monochrome color which can be stained manually after milling and sintered by coloring oxides to avoid the hard white color of these restorations.¹³

To overcome the disadvantage of the conventional type of zirconia restorations with compromised esthetics the zirconia framework is veneered with glass-ceramic to mimic the natural tooth color.⁵⁵

Subsequently, the veneered restorations developed fracture attributed to inner tensions, which result from coefficient of thermal expansion (CTE) differences between the veneering ceramic and substructure zirconia, and Geometric crown preparation structure. These inner tensions endue by external tensions from the masticatory force, when both external and inner tensions exceed the strength of restoration material fracture happened.⁷⁵ The combination between zirconia framework and veneering ceramic produce brittle materials, Since ceramic veneering has less strength than zirconia so, it is more susceptible to fracture, and can be fracture rapidly causing the chipping of the veneered restorations.⁷⁶

Nowadays, many all-ceramic restorations materials are manufactured using high crystalline content ceramic coping to improve strength, and veneered with porcelain to provide enhanced esthetics.^{77,78} Demonstrated in many studies that, all-ceramic restorations face cracking, chipping, or delamination of the delivered veneering porcelain.⁷⁹

In case of zirconia-based materials the main reason of failure of the restoration is chipping.^{80,81} Nevertheless, heat-pressed zirconia based restorations have shown less chipping of the veneering material than other restorations gained by conventional layering method.⁸²

2.3.4. Monolithic processing of zirconia

To overcome the chipping of zirconia restorations and increase it is durability, the use of veneered zirconia has recently desisted and monolithic zirconia takes place, monolithic term comes from the Greek word (monolith) which meaning single stone or stone from one casting, one material, and one unit.⁸³

In dentistry milled or pressed glass-ceramic crowns without veneering consider as typical monolithic restorations and these materials should have a certain requirement like long term stability and more translucency.⁸³

Monolithic zirconia restoration materials, fabricated solely by the technology of the CAD/CAM, have great benefits:⁸⁴

- They offer higher flexural strength.
- More tooth structure conservation.
- Less wear on the antagonist teeth.
- Offers good aesthetics performance.
- Laboratory time saving and less dental visits.
- Get rid of the unwanted complication of chipping.^{85,25}

From another hand, until a few years ago zirconia was with unsatisfactory translucency, this concept changed with the introduction of highly translucent cubic/tetragonal zirconia based restorations.⁸⁶ At the same time zirconia restorations got a lot of attention because of its excellent biocompatibility, esthetic properties, and enhanced mechanical properties.^{87,88}

In a typical manner, monolithic lithium disilicate has been used for the anterior region while highly stable monolithic has been preferred for posterior teeth.^{70,89}

Depending on its unique mechanical properties zirconia considered as strongest dental restorative material. And in the last few years, at the expense of strength, the translucency of ceramic zirconia has increased, and as a result of improvements in CAD/CAM techniques the translucent monolithic zirconia restorations have become more common clinically.^{90,42}

2.3.4.1. Mechanical properties of monolithic zirconia

Dental restorations based on zirconia have the greatest flexural strength, toughness strength, resistance to fracture and hardness among all other ceramic products, their flexural strength exceed 900 MPa, surface microhardness about 1200–1350 VHN, coefficient of thermal expansion more than $10 \cdot 10^{-6} \text{ K}^{-1}$, and thermal conductivity of less

than 2 W/mK.⁹¹ These mechanical properties of zirconia can be influenced by different production and milling procedures of the material blanks.⁹²

The excellent mechanical properties of zirconia attributed to the transformation toughening process, in the presence of external stress zirconia expands in volume, and this process obstructs the crack propagation leading to improvement of the zirconia toughness. The zirconia based restorative materials own the highest flexural strength about (900-1400 MPa) and have fracture toughness (5-9 MPa.m^{1/2}) among all dental restorative materials.⁹¹

Both types of zirconia based materials, framework zirconia and full-contour zirconia, are commonly 3 mol% yttria-stabilized zirconia and demonstrate similar transformation toughening and also have comparable flexural strength and toughness.⁹²

Monolithic zirconia with polished and finished surface showed less wear to the antagonist enamel than glazed zirconia, and feldspathic porcelain or glass ceramics.⁹³

2.3.4.2. Optical properties of monolithic zirconia

Currently, monolithic zirconia with improved optical properties is more translucent and has an aesthetic appearance, achieved by eliminating the light dispersion from the material bulk. This dispersion of internal light is due to many reasons, for example the presence of pores, different crystalline phases, incomplete sintering, contamination, deformity and grain borders.⁸⁵

The main factors affecting light scattering in zirconia are as following:

2.3.4.2.A. Composition

For manufacturing of translucent monolithic zirconia, the quantity and size of alumina sintering aids grains (Al₂O₃) has been reduced to improve the density of Y-TZP ceramics creating a light homogeneous micro grain.^{94,95}

However, because of the different alumina and zirconia alumina refraction indices which were regarded as remarkable dispersal centers.⁹⁶ It has been reported that, reduction of alumina content from 0.25 to 0.1 or 0.05 wt% can worthy increase the translucency. Although the reduction of alumina enhances color, it needs a higher sintering temperature >1500°C, with a longer dwell time of about 6 hours which leads to an increase in the grain size causing low translucency.^{42,97}

Recently Zhang et al.⁹⁸ introduced a highly translucent and highly strong zirconia by doping it with 0.2 mol% La_2O_3 in established 3Y-TZP contain 0.1-0.25wt% Al_2O_3 result in a material with high translucency, excellent hydrothermal stability and improved strength. Alumina (Al^{3+}) and lanthanum (La^{3+}), eliminate separation of grains at grain borders, accordingly porosity and birefringence decreased.^{99,98}

The increased yttrium percent about 4 to 5 mol% in the partially stabilized zirconia ceramics materials (second-generation), produced materials with high amounts of isotopic cubic phase >25–50% with large grains and no birefringent.^{100,64} Thereby, increasing translucency and decrease light scattering at the grain boundaries.¹⁰¹

2.3.4.2. B. Grain size

The effect of grain size explained in two concepts, the first propose that large grain size gives fewer grain boundaries lead to increase light transmission.¹⁰² And to produce large grains, the material subjected to higher temperature sintering, lead to more compact material with high density and no porosity, accordingly the translucency increased, the other suggest, in the polycrystals of tetragonal zirconia, the large size of grain has been related to increased light scattering and reduction of translucency.^{103,104} And this is imputed to the intrinsic birefringent qualities of the tetragonal crystal of zirconia. The grain size cannot therefore define translucency on its own, but other parameters, like cubic phase presence, porosity and final density, play a significant role.^{23,104}

2.3.4.2. C. Sintering

Most of the studies reported that, the sintering parameter such as sintering temperature and holding time effect directly the monolithic zirconia color properties.¹⁰⁵ The increase of sintering temperature, leads to improved translucency by increasing translucent parameter TP and decreasing in contrast ratio (CR).^{106,107}

Generally, the dental monolithic zirconia must be sintered in a degree of temperature between 1400-1550°C but, with a higher degree of about 1600 or 1700 or by prolonged sintering, there will be generating of cracks at grain boundaries increasing light scattering consequently, lead to enhanced translucency of the material.¹⁰⁸

2.3.4.2. D. Porosity

Pores play important role in the esthetic properties of monolithic zirconia, especially in translucency. The pores consider as the main reason of internal light scattering when their size equal to the size of the visible light wavelength (400 to 600 nm).¹⁰⁹

For excellent translucent material the pores should be extremely limited (<0.01 vol%), and the pores among grains (intergranular pores) should be eliminated during sintering which can be achieved by high sintering temperature and increased times of holding. When pores size larger than 50 nm can increase scattering and decrease transmission of light. The recent studies reported that the powder of zirconia needs to be in the nonmetric scale of cubic and tetragonal phase in order to eliminate the residual porosity and minimize the size of pore <20 nm which too little to produce prominent light dispersal.^{110,111}

However, there are other defects like impurities that may be noted in polycrystalline zirconia which working as a scattering center and affect negatively the light properties of the material.¹¹²

Monolithic zirconia based material with improved optical properties was attained by increasing the holding time and temperature rate of sintering temperature , causing microstructural alteration lead to high translucency.¹¹³ However, if the sintering temperatures exceed 1600°C will led to a decrease in flexural strength.¹¹⁴

Another improvements were made by decrease the amount and size of alumina grains together with dislodging them on the grain boundaries of zirconia.¹¹⁵ This resulted in high light transmission, long-term stability and improved strength.¹¹⁶

For more improvement of zirconia optical properties, recently, fully stabilized cubic/tetragonal zirconia materials, with about 50% of cubic phase have been introduced as highly translucent material has color modifications achieved by an increase of stabilizing oxides and increase in isotropic cubic crystal maintaining the strength of the material.^{97,42}

2.4. IPS e.max ZirCAD

IPS e.max ZirCAD is an advanced and inventive zirconium oxide products, which can be used in many types of dental restorations. It is appropriate for both, frameworks fabrication and monolithic crowns and bridges. IPS e.max ZirCAD is the choice material for the fabrication of restorations that need high strength, and natural appearance at the same time. This material is obtainable in form of a disc or block for CAD/CAM system. The discs are produced in three levels of translucency for a wide range of indications: medium opaque (MO), low translucency (LT) and medium translucency (MT), and multi discs polychromatic with transitional translucency (MT Multi).¹¹⁷

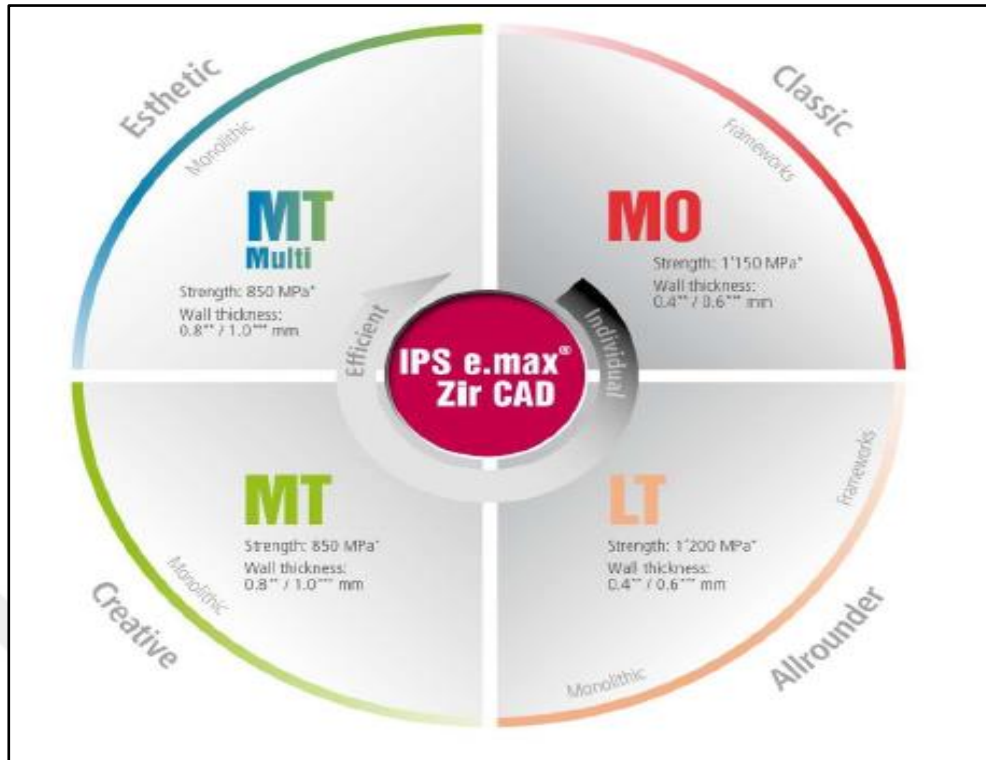


Figure 2.6: IPS e.max ZirCAD range by translucency (MO, LT, MT, and MT Multi) and indication. ¹¹⁷

2.4.1. IPS e.max ZirCAD – CHAIRSIDE

IPS e.max ZirCAD LT blocks are produced for the construction of "one-visit" restorations as a monolithic restoration by the dentist. The (LT) low translucency blocks are monochromatic, pre-shaded provide the fabrication of esthetic restorations without veneering techniques. They are supplied in 7 A–D shades and 1 Bleach color (BL), and are provided in block with size of C17 and B45. They can be indicated as monolithic restorations for monolithic full-contour crowns in the anterior and posterior teeth regions and monolithic full-contour three-unit bridges for anterior and posterior teeth.¹¹⁸ They contraindicated only in case of patients with severely reduced residual dentition.¹¹⁸

The fully crystalline 3Y-TZP ceramics (IPS e.max ZirCAD LT and MO) have the following composition (Table 2.1).

Table2.1: IPS e.max ZirCAD LT and MO, typical composition of 3Y-TZP¹¹⁸

Component	Content
Zirconium oxide (ZrO ₂)	88.0 - 95.5 wt%
Yttrium oxide (Y ₂ O ₃)	> 4.5 - ≤ 6.0 wt%
Hafnium oxide (HfO ₂)	≤ 5.0 wt%
Aluminium oxide (Al ₂ O ₃)	≤ 1.0 wt%
Other oxides for colouring	≤ 1.0 wt%

The improvement of zirconium oxide restoration materials was to word the production of highly translucent pure 5Y-TZP raw material in IPS e.max ZirCAD MT to provide a full contour crown without veneering. However, the translucency of the material was limited in less wall thickness preparation. As a result, combination of 5Y-TZP with 3Y-TZP was used to give an optimum translucency for monolithic crown restoration in the anterior region prepared with about 1.5 mm wall thickness.¹¹⁹

IPS e.max ZirCAD LT is indicated for esthetically full-contour zirconia restoration at a wall-thickness from 0.5 to 0.8 mm, but >0.9 mm wall thickness makes the restoration more bright and opaque. For that, in occlusal or incisal sections minimum layering is recommended when using IPS e.max ZirCAD LT.¹²⁰

2.5. The inCoris TZI C zirconia

The inCoris TZI C is zirconia included ceramics materials designate blocks which are manufactured in a partially sintered state; then enlarged by the in Lab CAD/CAM system, they are separately treated to identification and densely sintered in the end. It can be used as fully anatomical crowns and bridges restorations and have many advantages including:

- High flexural strength.
- High corrosion resistance.
- Excellent biological compatibility.
- High translucency.^{122,121}

Table 2.2: Chemical composition of inCoris TZI C¹²¹

Component	inCoris TZI C
ZrO ₂ +HfO ₂ +Y ₂ O ₃	≥ 99.0%
Y ₂ O ₃	> 4.5 - ≤ 6.0%
HfO ₂	≤ 5%
Al ₂ O ₃	≤ 0.07%
Other oxides	≤ 1.1%

The inCoris TZI C indicated for fully anatomical restorations (monolithic) crowns and bridges of two pontics, but they should be avoided for the uses in some situations like insufficient oral hygiene, underprepared teeth and insufficient space or tooth structure to support the restorations.¹²²

The inCoris restorations should be sintered in dry conditions and only by using the pre-programmed Sirona in Fire HTC/in Fire HTC speed with inCoris ZI program or can be fulfilled in VITA Zyrcomat or Ivoclar Vivadent Sintramat high-temperature apparatus as an alternative.¹²³

The inCoris TZI C are translucent pre-shaded zirconium based ceramic blocks they save time because needed no coloring procedure they can be used in case of limited space between teeth and antagonist for restoration, no chipping occurred, can be used with speed sintering or conventional sintering and then glazed.¹²⁴

2.6. Restorative Material Properties

Due to the rapid way of production of new restorative dental material, and more variety of products available depending on the advanced technology, in the field of dentistry when the clinician chooses the restorative ceramic material for a particular indicator, he faces a big dilemma. The choice based on the properties of restorative materials that should be known by dentists, in order to establish the material's suitability

for particular indications. These properties can be classified into two types; mechanical properties and optical properties.³²

2.6.1. Mechanical Properties

The zirconia ceramics materials (ZrO_2) are polycrystalline, heterogeneous and highly resistant ceramics, also zirconia based restorative materials have excellent biocompatibility, good radio opacity and no water solubility, so it is not susceptible to corrosion in the oral environment.¹²⁵

The highly improved mechanical features of monolithic zirconia restorations demonstrated by many in vitro studies, which proved that crowns with 0.5 mm thickness showed sufficient fracture resistance for clinical uses in posterior restorations.¹²⁶

The successful dental treatments depends on sufficient knowledge of the mechanical and physical properties of used materials, and the evaluation of the mechanical properties of restorative materials used in dentistry, was Performed by various types of measuring tests of dental materials such as the compressive strength, tensile strength, shear bond strength, flexural strength, bending, scratch, hardness and micro indentation tests.¹²⁷

2.6.1.1 Flexural Strength

The flexural property is an important issue in clinical applications of ceramic materials. Therefore, the laboratory study can help to evaluate the clinical usability of new dental restorative material.¹²⁸ Nevertheless, the dental restorations have complex geometry and no standard method for measuring the strength of these designs. Therefore, the will defined shapes of materials specimens like disks or bars can be used for evaluation of strength value of structural performance for the materials.^{128,129}

There are many types of test methods that have been approved for measuring the flexural strength of ceramic materials; biaxial bending test, 3-point testing and 4-point which developed to be the commonest tests.¹²⁴

The flexural strength is the material potentiality to bend prior to breaking. It is gained if a material is eventually flexible before its relative limit.¹³⁰ Bending forces mean that forces occur in clinical circumstances and dental materials must resist repeated flexing, bending and twisting during chewing. High flexural strength is required because the dental restorative materials which are under the influence of mastication may cause permanent deformation.¹³⁰

Contamination of the test specimens by foreign substances, small unnoticed defects, or mini scratches occurring during the sample preparation may negatively affect the strengths of materials; therefore, it is very important to examine the samples thoroughly before starting laboratory tests.¹³¹

Flexural strength can be measured by using biaxial flexural test, three point or four point test flexural tests. But, still the three-point bending test is commonly used because it is uncomplicated and relatively simple.¹³²

In all types of the test, the static load is applied until failure. The four-point flexure test can be done by creating uniform field of stress centered in the half of the distance between two loading pistons, while in the three-point flexure test a non-uniform central stress field is created. In the biaxial flexure strength test, the chances of edge failures are reduced by loading the disk sample in the center.¹³³

The three-point bending test results are correlated with the four-point bending tests. However, in comparison with both other tests for all ceramics examined, the link between three-point flexure test and biaxial flexure test was not consistent and the values of the four-point bend test were lower.¹³³

To assess the bending strength of dental material, beam or bar-shaped samples the size according to Standard No ISO 6872 standard are generally used.¹³⁴

Specimens are tested by bending a bar-shaped sample which is supported by two rollers at the end, the load is centrally placed on the top of the bar, where the grips and fixtures selected depending on the standards for each specimen, the tests and calculations allow the determination of:

- The diagram of force-displacement.
- The displacements and breaking loads.
- The deformation and breaking strength.
- The stress-strain diagram.¹³⁵

This test is known as a three-point flexural test. (Figure 2.7)

When the sample undergoes fracture at that point the flexural strength is calculated by the following formula:

$$\sigma = 3 PI/2wb^2$$

Where: σ = the flexural strength of tested specimen.

P = the ultimate load at fracture.

I = the distance of the supports.

B = the width of the specimen.^{136,136}

The high force produced by mastication can produce permanent deformation of restorative materials unless these materials have the capability to withstand these forces by high flexural strength property.¹³⁶

The strength of the material and the expected distortion value are determined by flexural strength test which combine both tensile and compressive stresses and modulus of elasticity of the material. In dentistry, the strength of both ceramic and resin based restorations is frequently estimated with such tests.¹³⁷

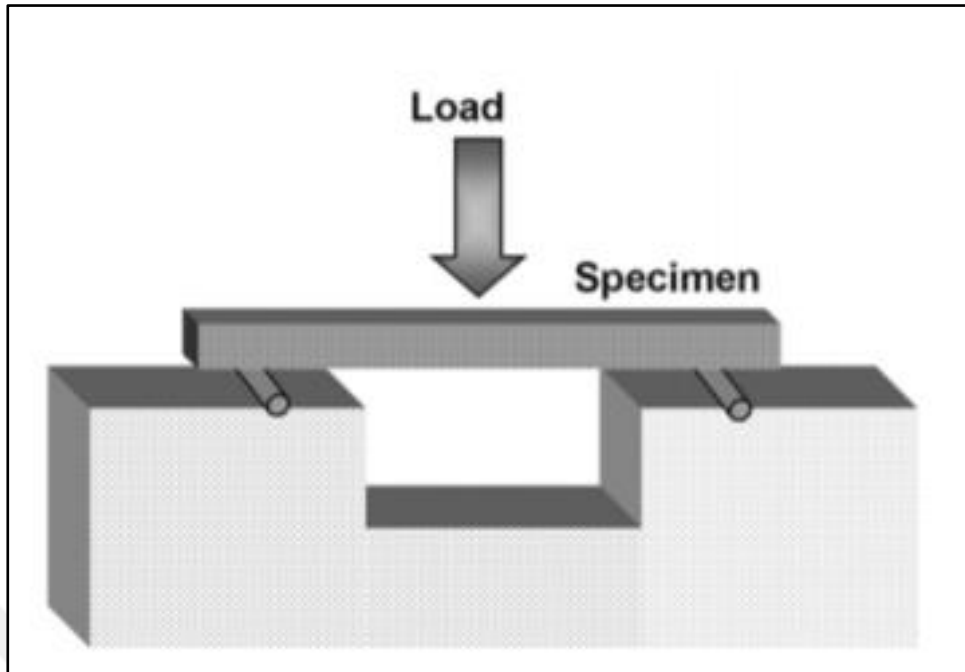


Figure 2.7: Schematic illustration of flexural strength test of a material¹³⁶

2.6.1.2 Surface Microhardness

Hardness is the material's resistance to continuous deformation and is one of the very important measured properties in ceramic material which helps to distinguish between deformation, density and fracture resistance.¹³⁸

The hardness characteristics can be measured by using conventional microhardness machines using Knoop or Vickers diamond indenters with an optical microscope, whereas hardness values can be calculated based on the dimension of indentation and the load applied. In the Vickers Hardness test, a pyramid-shaped 136-degree diamond indenter with standardized load is used to create a microscope diagonal shape indentation.¹³⁹

2.6.2. Thermal aging

The aging resistance of dental restorations, in particular monolithic zirconia, is a key property because the restoration entire surface is subject to various oral conditions.

Many tests used to examine the restorative materials include accelerated autoclave aging, boiling in warm water or artificial saliva, thermocycling, mechanical cycling loading, and simultaneous thermal mechanical fatigue.^{61,140}

Thermocycling device composed of two water baths at a different temperature within several cycles.¹⁴⁰ The thermal cycles represent repeated stress of expansion and contraction by changing the degree of the temperature of water baths resulting in cracks and fractures, loss of retention, loss of bounding, and degradation of the material specimen.¹⁴¹ Also, water absorption and hydrolytic degradation during the process of thermal cycling may cause a deleterious effect on materials.¹⁴² Nevertheless, these effects are all dependent on many factors controlling the aging protocols, such as the number of cycles, resting time in each bath, and time interval between baths, one or all of these factors variations can affect the results.¹⁴³ There are many studies indicating the number of cycles that represents a certain period of clinical service for example; 10,000 thermal cycles are equal to five years of clinical use.¹⁴⁴

2.6.3. Optical properties (Colorimetry)

Visible light is electromagnetic radiant energy that can be detected by the human eye within the wavelength range of about 380-780nm.¹ An object must either emit light, transmit or reflect the beam of light from external sources to make it visible.^{1,145} The colors we grasp is a combination of several wavelengths, thus, a graphic design called the spectral curve of the color can explain the color spectrum.¹⁴⁶

In colorimetry the color space is a distinct term that identifies the combination of a color model and a mapping. Munsell in 1905 produced a method for expressing color by using colored paper chips divided according to their lightness, Chroma and Hue, he arranged the color in a circle according to their hue which refers to the color by which we recognize red from green, yellow and so and he used value as an indicator for the lightness of the colors, value (0) indicate pure black and (10) value indicate pure white.¹⁴⁷

In the since research the color can be analyzed by colorimeters and spectrophotometers devices, which reflect the light from the surface of an object, and these devices have high precision and increased versatility for calculating the spectral reflectance at each wavelength and the light flow data into color parameters. It can be used to established shade and calculate color differences in numeric value.^{1,148}

The CIE (Commission International de l'Eclairage) color system identifies a color in a color space using three coordinates. The CIE system supports two color scales CIE ($L^*a^*b^*$) and CIE ($L^*C^*h^*$), where L^* indicates lightness, a^* indicates the red-green value, and b^* indicates the yellow-blue value. The CIE ($L^*C^*h^*$) uses polar coordinates L^* indicates lightness, C^* indicates Chroma, and h^* indicates Hue¹⁴⁹ (Figure 2.8).

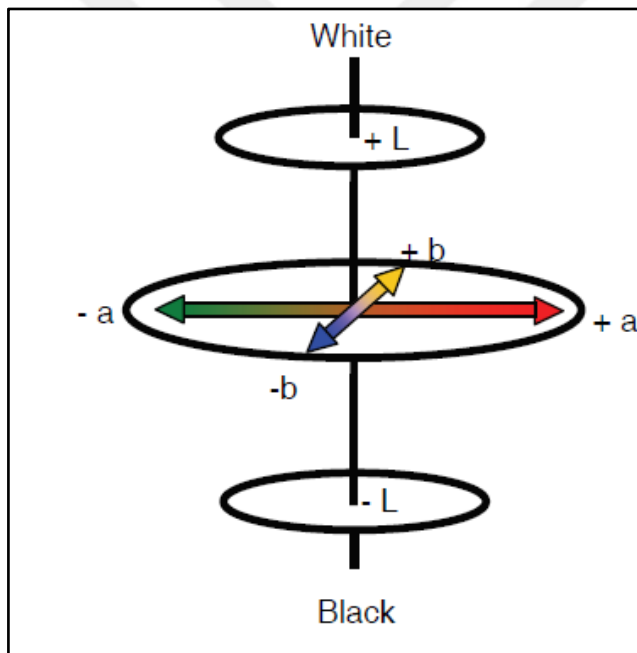


Figure 2.8: CIE $L^*a^*b^*$ system¹⁵⁰

While the color chromatic value is shown on the lightness axis (L^*) and on the chromatic axes (a^* and b^*) the color spatial projection of a data is presented to allow the color change to be determined. The values L^* a^* b^* from the tristimulus X, Y, and Z are obtained from the light reflections of observed object and calculated with special equations.¹⁴⁹

For color studies, the main measurement is a color difference (ΔE).¹⁴⁵ The ΔE_{ab}^* equation of color difference which gives a single value for number can indicate the color difference size depended on the difference between color coordinates of two objects target and changed color, for example, tooth and tooth restoration.¹⁵¹ ΔE_{ab}^* is defined by the following equation¹⁵²:

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

where:

$$\Delta L^* = L^*_1 - L^*_2$$

$$\Delta a^* = a^*_1 - a^*_2$$

$$\Delta b^* = b^*_1 - b^*_2$$

2.6.3.1. Clinical significance

The relationship between visual and color analysis is very important in clinical applications. Johnston and Kao in 1989 evaluated the color difference between samples under ideal conditions of illumination and due to the difficulty of controlling the light condition inside the oral cavity the average value of ΔE up to 307 was acceptable and recommended an Extended Visual Rating Scale called (EVR SAM) to explain the clinical importance of numerical value results from dental restorative material color analysis¹⁵² (Table 2.3).

Table 2.3: Extended visual rating scale (EVRSAM)¹⁵²

ΔE	Clinical significance.
0	Excellent esthetics with accurate color choice, not being clinically perceived, or only with great difficulty.
2	Very slight difference in color, with very good aesthetics.
4	Obvious difference, but with an average acceptable to most patients.
6	Poor aesthetics, but within the limits of acceptability.
8	Aesthetics are very poor and unacceptable to most patients.
10	Aesthetics are totally unacceptable.

However, the CIE (Commission Internationale de l'Eclairage) main color systems considered (ΔE) color difference concepts the standard for color evaluation, and it examines the CIELAB color space (L*:value coordinate; a*:red-green coordinate; b*:yellow–blue coordinate), while other factors of color perception, such as translucency, opalescence, contrast and fluorescence not included in this concept.¹⁵³

However, in the studies of the threshold value for the human eye, there is no unanimity in which the color difference between two materials begins to be detected and which one more noticeable to the observer, and how ΔE is clinically reasonable.¹⁵⁴

In a certain number of studies $\Delta E=3.0$ accept to be imperceptible to the human eye, other studies indicates acceptance of $\Delta E=3.3$ and $\Delta E=2.0$. Another study considered restricted criteria to limit ΔE , which revealed that small values less than 1 are unnoticeable to the eye of observer, ΔE from 1 to 2 are accepted clinically. ΔE higher than 2 clinically unacceptable.¹⁵³

O'Brien stated that the color difference is acceptable to clinic up to 3.5 ΔE .¹⁵⁵ (Table 2.4)

Table 2.4: Clinical color matching of O'Brien

ΔE	Clinical Color Matching
0-05	Excellent
0,5-1	Very good
1-2	Good
2-3,5	Clinically acceptable
> 3,5	Incompatible

2.6.3.2. Color measurements

There are two types of instruments used for the measurement of color difference called colorimeters and spectrophotometers. The colorimeters are portable, small in their size, and express colors numerically by using the tristimulus method according to international standards.¹ While colorimeters have sensitivities matching the human eye, they are not suitable for complex color analysis such as metamerism and stain strength.¹⁵⁶

The spectrophotometer is used for color measurement with the tristimulus method and the numerical data with spectral reflectance graph for the color of the samples can be obtained, the spectrophotometer provided more accuracy than a colorimeter in the research field because it has a high precision sensor and included data from various illuminant conditions, but spectrophotometers can be more expensive than colorimeters.¹⁵⁶

Spectrophotometers with their different types are used for color measurement especially in the field of dental researches for ceramic restorative materials.^{157,158}

2.6.3.3. Translucency and opacity

Antinomy, ceramic dental substances, should be both translucent and opaque to simulate natural structures of teeth. In the natural dentition, the opacity relates to dentine, as the light passes through clear enamel and reaches the opaque dentin body, which again

reflects the light. Furthermore, enamel acts like a translucent object allowing for light passage and visualization and providing for the dispersion of light by the hydroxyapatite crystals at the wavelength of the blue.¹⁵⁹

Translucency represents the amount of light passing through an object.¹ And this property gives a natural appearance to the restorative dental materials in order to achieve the most favorable aesthetic result.¹⁶⁰

Unfortunately, there is no dental restorative material that has the required opacity and translucency in a single product. Accordingly, factories produced opaque ceramic for infrastructure and covered by translucent glazed ceramic to be used in layering techniques.¹⁶¹

Nowadays, with rapid progression in the production of monolithic zirconia still exhibit some difficulty in term of esthetics.¹⁶² The original shade of material, color properties of ceramics and various laboratory processes may influence the color of the product. On the other side, the resulting color of zirconia may be affected by other practical factors such as dental background, cement type and restorations thickness. The final restoration color may be caused by the effects of elements, including manufacturing methods, laboratory processes and clinical factors.¹⁶³

TP and CR parameter are the most common optical characteristics which are evaluated in monolithic zirconia ceramics theoretically and physically.¹⁶⁴

The contrast ratio CR is the relationship between the reflections of a sample tested over a white background and a black background of a known reflections and the analysis of opacity.¹⁶⁴ CR ranges from 0 to 1, with 0 for total translucency and 1 for total opacity.⁸⁵

The translucency parameter TP is the color difference between a specimens of uniform material thickness measured over the white and black background. The parameter translucency is measured in the following formulation.⁸⁵

$$TP = [(L^*_w - L^*_b)^2 + (a^*_w - a^*_b)^2 + (b^*_w - b^*_b)^2]^{1/2}$$

The TP denotes the L*, a*, b* value for a ceramic placed on a white (W) and black (B) backgrounds.

The translucency of dental ceramics depends on how the incident light interacts with ceramic martial type. TP value is about zero in the case of absolute opaque material. Thus, the higher the TP value the more translucent material.^{165,166}

The CR and TP values of the ceramic systems have been assessed for strong correlation. Thus, the relative translucency of ceramic systems may be evaluated using either CR or TP.¹⁶⁷

2.6.3.4. Criteria for translucency evaluation

For clinical estimation of the translucency, there are two aspects: firstly, whether the values of the material's translucency are comparable to that of natural tooth enamel or, there is a difference which is noticeable by the human eyes.¹⁶⁸ The translucency parameter (TP) of 15 to 19 for 1mm thickness could be recommended as criteria for translucency evaluation for dental restorative materials.¹⁶⁰

2.6.3.5. Opalescence

The scattering of the short wavelength of the small visible light on the particles gives an object with bluish appearance in the reflected color, and an orange brown looks during color transmit this phenomenon is called opalescence.¹⁶⁹ For highly aesthetic dental

restorations should use the material with opalescent properties to simulate the natural tooth appearance.¹⁷⁰

This optical phenomenon is mainly linked to the highly mineralized structure of the enamel, and work as a filter for waves, it is able to reflect the short waves and forward the long-wave, for that reason the incisal margin having bluish-white appearance in color. The long waves when transmitted to dentin and reflected back gave the orange glaze to enamel ,and this effect called counter opalescence that can be produced in ceramic material by using opalescent glazes with bluish and orange color depending on added pigments, also at the time of lab work the enamel fabricated based on the optical appearance of the tooth as layers.¹⁶⁹

The property of the opalescence material can be assessed with opalescence parameters (OP) which gives the differences between the red-green color coordinate (CIE a^*); and the yellow-blue color coordinate (CIE b^*); in the reflected and transmitted colors, which are calculated out of ΔE value of a^* and b^* against white (W) and black (B) backgrounds consecutively using this formula:

$$OP = [(a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2}$$

where, the subscripts B and W refer to color coordinates over black and white backgrounds, respectively. and that high OP value represents the opalescence value.¹⁷¹

3. MATERIALS AND METHODS

3.1. Materials

Four types of CAD/ CAM restorative materials are used in this study , Celtra DUO (Dentsply, Sirona, Hana-Wolfgang, Germany), inCoris TZI C (Dentsply, Sirona, Hana-Wolfgang, Germany), IPS e.max ZirCAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein) and IPS Empress CAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein) (Figures 9-12).



Figure 3.1: Celtra DUO (Dentsply, Sirona, Hana-Wolfgang, Germany)



Figure 3.2: IPS Empress CAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein)



Figure 3.3: inCoris TZI C (Dentsply, Sirona, Hana-Wolfgang, Germany)



Figure 3.4: IPS e.max ZirCAD (Ivoclar Vivadent, AG, Schaan, Liechtenstein)

A total of 120 specimens were used in this study 30 samples were prepared for each of the four materials 20 specimens for mechanical tests and 10 specimens for optical tests. The tested groups are listed in (Figure 3.5).

The main compositions and manufactures of the materials are listed in (Table 3.1).

Table 3.1: The main compositions and manufacturer of the materials and their symbols in the study.

Material	Symbol	Manufacture	Compositions (in weight %)	Lot number
Celtra DUO	Cel	Dentsply, Sirona, Hana-Wolfgang, Germany	<u>Zirconia-reinforced lithium silicate</u> Celtra DUO (ZLS) is mainly composed of 58% silica, lithium-metasilicate, -disilicate, and -phosphate crystals, and 10% zirconia crystals in addition to other minor ingredients.	16004327
inCoris TZI C	Inc	Dentsply, Sirona, Hana-Wolfgang, Germany	<u>Translucent zirconium oxide sinter ceramic</u> $ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0\%$ $Y_2O_3 \ 5.6\%$ $Al_2O_3 \leq 0.35\%$ Other oxides (except Er_2O_3) $\leq 0.2\%$	2018175682
IPS e.max ZirCAD	Zir	Ivoclar Vivadent, AG, Schaan, Liechtenstein	<u>Presintered yttrium stabilized zirconium oxide block (Y-TZP)</u> Zirconium oxide (ZrO_2) 88.0 – 95.5% Yttrium oxide (Y_2O_3) 4.5% – $\leq 6.0\%$ Hafnium oxide (HfO_2) $\leq 5.0\%$ Aluminium oxide (Al_2O_3) $\leq 1.0\%$ Other oxides $\leq 1.0\%$	Y07929
IPS Empress CAD	Ips	Ivoclar Vivadent, AG, Schaan, Liechtenstein	<u>Leucite reinforced glass ceramic</u> SiO_2 60.0 - 65.0 Al_2O_3 16.0 - 20.0 K_2O 10.0 - 14.0 Na_2O 3.5 - 6.5 Other oxides 0.5 - 7.0 Pigments 0.2 - 1.0	W87996

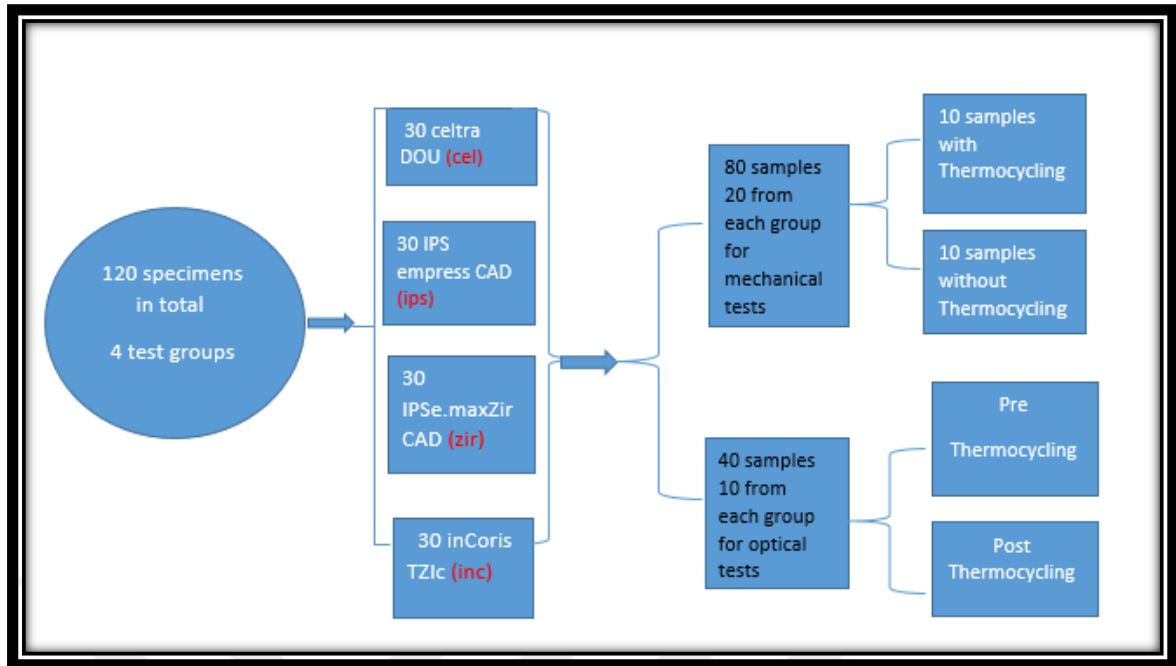


Figure 3.5: Diagram of the materials groups for mechanical and optical tests.

3.2. Methods

The test process was divided into two main parts, mechanical and optical. The descriptions of each test and the preparations of specimens are as follows:

3.2.1. Mechanical testing

The mechanical properties which tested for each material are flexural strength test and surface microhardness test (Vickers hardness test).

3.2.1.1. Specimens' preparations for mechanical tests

A total of 80 specimens for mechanical evaluation, 20 specimens from each type of materials with size (14 mm x 1.2mm x 4 mm) were prepared according to ISO6872 20015.¹⁷²

Each group divided into 2 subgroups, 10 specimens for each one subgroup are thermo-cycled and other none.

The commercially available CAD/CAM blocks for inCoris TZI C (Dentsply, Sirona, Hana-Wolfgang, Germany), IPS e.max ZirCAD (Ivoclar Vivadent, AG, Schaan,

Liechtenstein) were sintered in a furnace according to their manufacturer's instructions. (Tables 3.2 and 3.3)

Table 3.2: The classic program for sintering inCoris TZI C.¹⁷³

Heating rate °C/min	Holding temperature °C	Holding time min
25	800	0
15	1510	120
30	200	0

Table 3.3: Standard program for sintering IPS e.max ZirCAD

	Temperature 1 [°C]	Temperature 2 [°C]	Heating-up rate [°C/min]	Holding time [min]
Heating phase	20	900	10	–
Holding phase	900	900	–	30
Heating phase	900	1500	3.3	–
Holding phase	1500	1500	–	120
Cooling phase	1500	900	10	–
Cooling phase	900	300	8.3	–

After sintering zirconia blocks they were cut with the other two types, Celtra DUO (Dentsply), and IPS Empress CAD (Ivoclar Vivadent) used a blade of diamond wafer mounted on a saw (Isomet, 1000 Buehler, Precision Sectioning Saw) under constant irrigation of water. (Figure 3.6, 3.7, 3.8)



Figure 3.6: Precision Sectioning Saw (Isomet, 1000 Buehler, Lake Bluff, IL, USA)

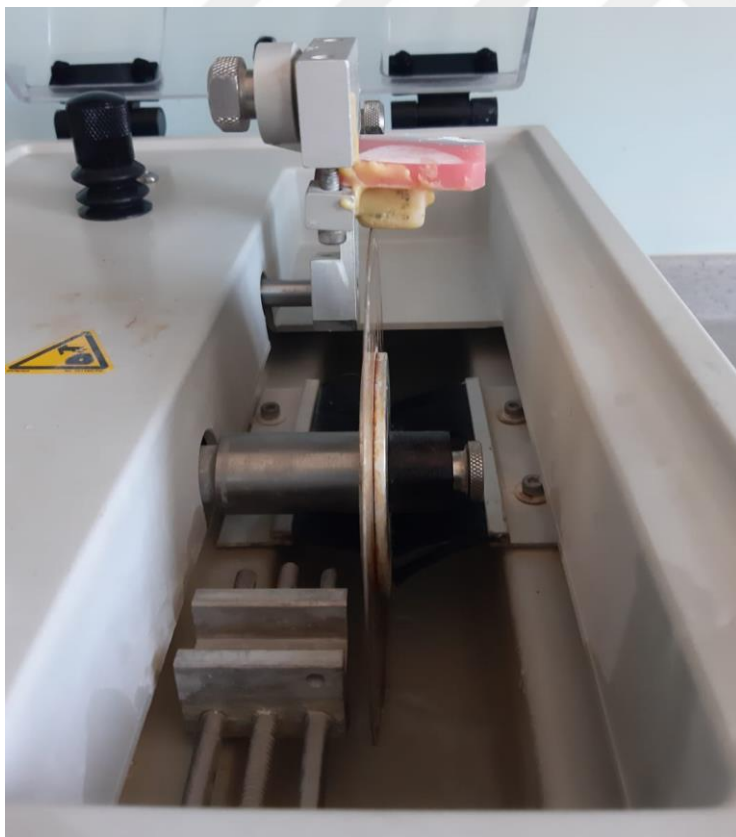


Figure 3.7: The saw with diamond blade

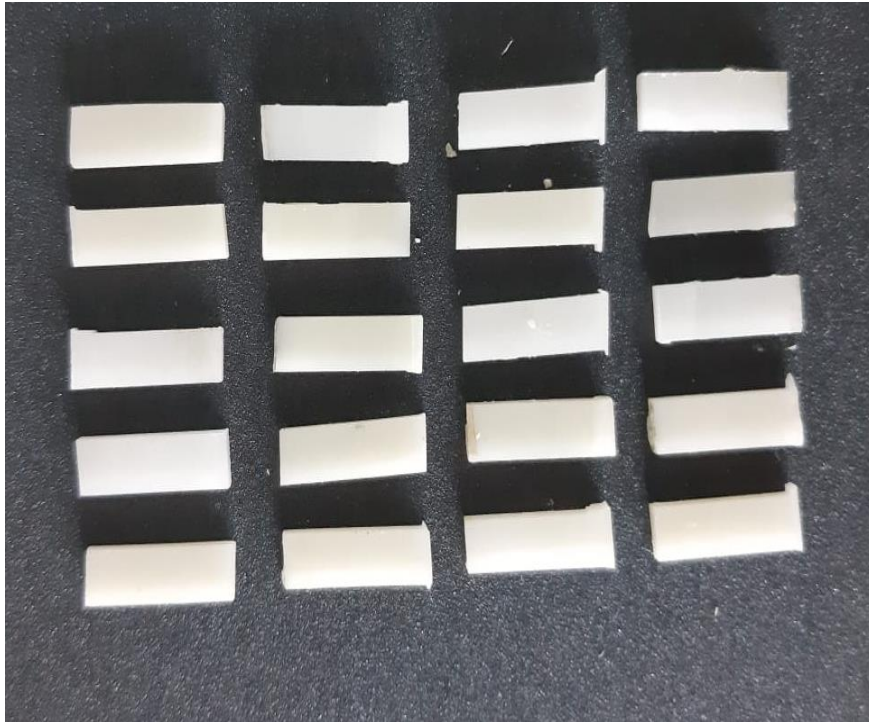


Figure 3.8: The specimens (14 mm x 1.2mm x 4 mm) before polishing

One face of each bar sample was polished and finished using 600, 800 and 1200-grit silicon carbide paper fixed on a grinder-polisher machine (Phoenix Beta Grinder/Polisher, Buehler, Dusseldorf, Germany) with water irrigation in order to get a uniform surface (Figure 3.9, 3.10).



Figure 3.9: Phoenix Beta Grinder/Polisher, Buehler, Dusseldorf, Germany



Figure 3.10: Polishing of the specimens

The definitive dimensions of the samples were determined after polishing by using a digital caliper (Absolute Coolant Caliper, Mitutoyo Corp ,Kawasaki, Japan) with an accuracy of $1.2 \text{ mm} \pm 0.02 \text{ mm}$ (Figure 3.11, 3.12, 3.13).

All specimens out the range ($\pm 0.02 \text{ mm}$) were discarded and rejected.

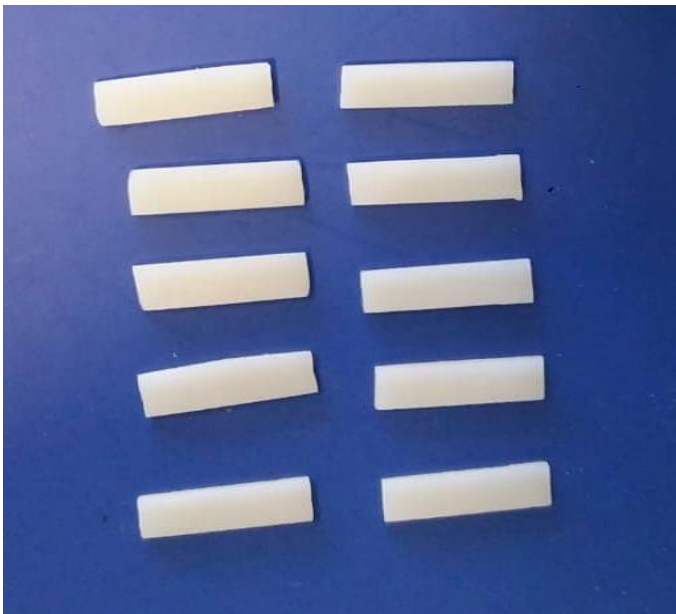


Figure 3.11: The specimens after cutting and polishing

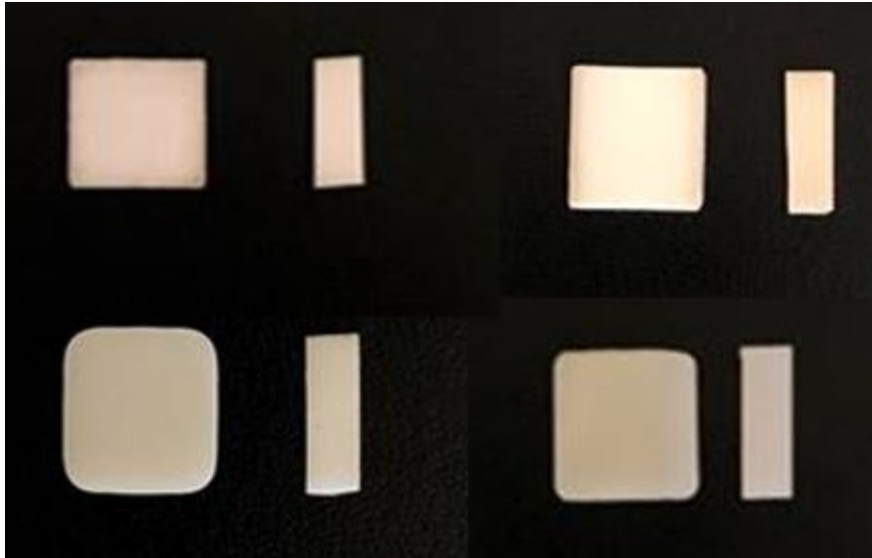


Figure 3.12: The specimens after polishing



Figure 3.13: Absolute Coolant Digital Caliper, Mitutoyo Corp, Kawasaki, Japan.

Then the specimens were cleaned in distilled water by using ultrasonic cleaner for 15 minutes (Figure 3.14).



Figure 3.14: iTronic IR8000EU ultrasonic cleaner
RMA19, UNIT B, 27/F, Billionplaza Ii, Hongkong, China

3.2.1.2. Specimens' aging (Thermocycling)

Each group for mechanical testing containing 20 samples were divided randomly into two subgroups ($n=10$).

One subgroup specimens ($n_1=10$) were stored in dry conditions, whereas the second subgroup ($n_2=10$) was aged with 10.000 thermal cycles (5°C to 55°C , dwelling time 30s).¹⁷⁴ In the thermocycling device (Salubris-technica, Dentester, Istanbul, Turkey) (Figure 3.15, 3.16).



Figure 3.15: Thermocycling device (Salubris-technica, Dentester, Istanbul, Turkey)



Figure 3.16: Specimens inside the bowl for thermal aging

3.2.1.3. Biaxial Flexural Strength Test

The flexural strength was determined for each material subgroups (thermo-cycled and non-thermocycled) three-point flexural tests using a universal testing machine (Instron, model 3345, Canton, Mass, USA) according to ISO 6872:2015¹³⁵ (Figure 3.17).

To measure the flexural strength for each specimen, the specimen was placed flat on the center of metal testing fixture with the polished surface facing down (away from the load direction), supported at each end by rounded rods for support 10 mm apart, with a load applied in the middle of each specimen (load cell 1 KN) with a rounded chisel (radius 3 mm) at a crosshead speed of 0.5 mm/min until fracture of specimen occurs the flexural strength was calculated in mega pascals (MPa) (Figure 3.18, 3.19).

The following equation was used for (σ) calculation:

$$\sigma = 3 PL / 2Wb^2$$

Where P is the fracture load (N),

L is the test span (10 mm),

W is the width of the specimen (mm),

b is the thickness of the specimen (mm).

The measurements of load and distance were calibrated to zero before every reading.



Figure 3.17: Universal testing machine (Instron, model 3345, Canton, Mass, USA).

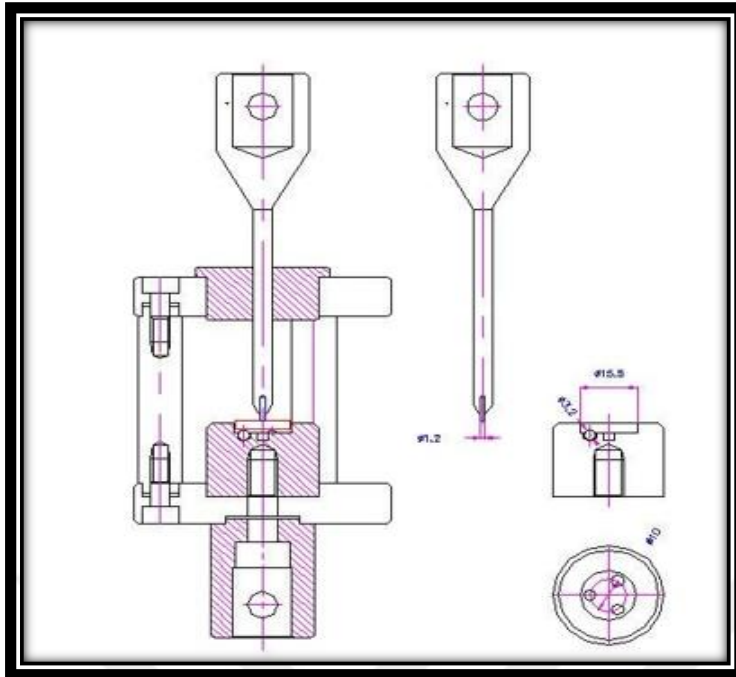


Figure 3.18: Diagram explaining the flexural strength measurements



Figure 3.19: The fractural specimen at constant crosshead speed of 0.5 mm/min

3.2.1.4. Surface Microhardness Test

The surface microhardness was calculated of each material using a Vickers Hardness Indenter (Model 1600-6125, Buehler, Lake Bluff, Illinois USA) (Figure 28).

Ten pieces of the bar-shaped specimens which used earlier for testing flexural strength for each subgroup of each material (with thermal aging and without thermal aging) were used for preform the hardness test.

On the high polished surface at the center of every specimen with a load of 1 kg/15 sec, a 136° pyramidal diamond indenter was placed to avoid the formation of radial cracks as required by ASTM C 1327-99.¹⁷⁵

For a specific 15-second dwell period the load was maintained to form a symmetrical diamond indent (Figure 3.20). The indent size was calculated using a fixed optical microscope by measuring the two diagonal forms of the diamond (Figure 3.21, 3.22).

In the following formula, the mean reading of the two diagonals was used to calculate the Vickers Hardness Number (VHN):

$$\text{VHN} = 1.8544p/d^2$$

where: **p** is the applied load (kg), **d** is the indent diagonal squared (mm). VHN is a constant which is a function of the indenter geometry and the units of force and the indent diagonal and known as the Vickers pyramid number. In each specimen at least 0.5 mm from each other in the center of the specimen were five indents made, and each specimen had an average of 3 reading.⁵²



Figure 3.20: The indentation tester (Model 1600-6125, Buehler, Lake Bluff, Illinois USA)

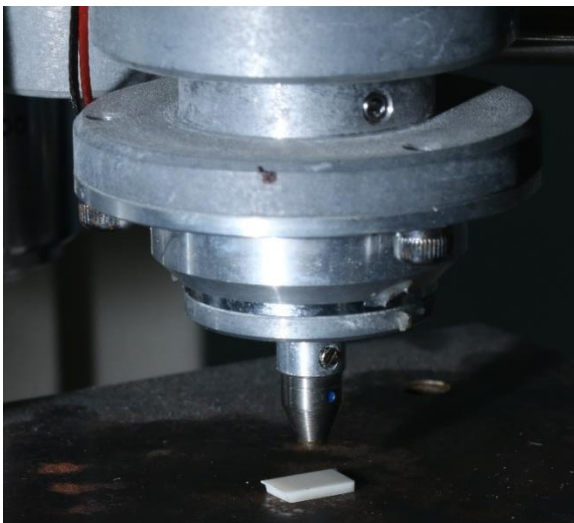


Figure 3.21: The specimen on the flat metal plate

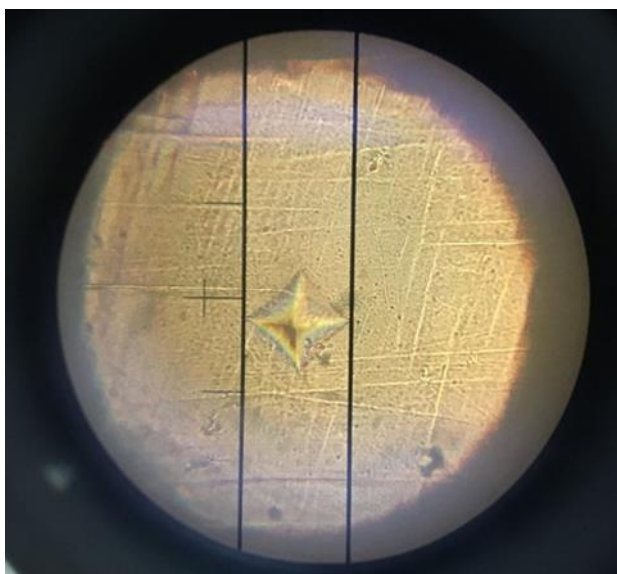


Figure 3.22: Vickers indentation mark

3.2.2. Optical tests

Total of 40 specimens for optical evaluations, 10 specimens from each type of materials were cut square shape by size of 12 mm by the same manner of cutting for the blocks which used for mechanical tests. The 12 mm × 12 mm × 1.2 mm monolithic blocks (inCoris TZI C and IPS e.max ZirCAD) were sintered before cutting according to their manufacturer instructions. For polychromatic IPS Empress CAD blocks, the specimens were cut to expose the parts of shade (Figure 3.23). One surface was polished and finished on a uniform surface for each sample using 600, 800, 1000 and 1200-grit silicon carbide paper retained on a grinder-polisher machine (Phoenix Beta Grinder/Polisher, Buehler, Dusseldorf, Germany) with constant water at 300 rpm to 1.2 mm thick and the specimen thickness was verified with a digital caliper (Absolute Coolant Digital Caliper, Mitutoyo Corp, Kawasaki, Japan.).

Accepted thickness value was 1.2 ± 0.02 mm. Any specimens out of this measured dimensions were discarded and rejected. The samples were also examined for chipping or distortion at the edges, any sample with visible chipping were rejected and discarded. The color selected was A2 for all types of materials and all materials are low translucent brand (LT) (Figure 3.23, 3.24).

Then the specimens were ultrasonically cleaned with distilled water for 15 minutes after the polishing by using ultrasonic cleaner. The same groups were used for optical analysis before and after thermal aging (thermocycling).



Figure 3.23: Polychromatic sample for IPS Empress CAD with shade degradation



Figure 3.24: Square shaped specimens for optical tests (12 mm x 12 mm x 1.2 mm)

A dental spectrophotometer (Spectrophotometer MINOLTA CM-2600D Konica Minolta Sensing,inc, Tokyo, Japan) was used to determine the CIELAB coordinates (L^* , a^* and b^*) of the ceramic and zirconia samples (Figure 3.25).

3.3.1. Color difference (ΔE)

Firstly, the ΔE values were calculated using spectrophotometer after calibration according to the manufacturer's instructions, then the device was directed to the middle point of each sample. According to the following formula:

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

ΔE : general difference in color changes.

Δa : difference color change in the red–green-axis.

Δb : difference in color change in the yellow–blue-axis.

ΔL : difference in brightness.

3.3.2. Translucency parameter (TP)

Translucency parameter (TP) values were determined by calculating the color difference between readings against black (L^* , a^* and b^*) and white (L^* , a^* and b^*) backgrounds for the same specimen, according to the following equation:

$$TP = [(L^*_B - L^*_W)^2 + (a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2}$$

Where the subscripts B and W are the color coordinates over black and white backgrounds, respectively (Figure 3.26, 3.27). Measurements on each background have been repeated 3 times, with a mean CIE $L^*a^*b^*$ for both backgrounds recorded. The TP value is zero, if the material is completely opaque; if it is completely transparent, it is 100 TP value. The greater the TP, the higher the ceramic specimen's translucency.¹¹³

3.3.3. Opalescence parameter (OP)

The opalescence parameter (OP) was evaluated by using the previous measurement of the specimens against black (B) and white (W) backgrounds for calculation of (OP) according to the following formula:

$$OP = [(a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2}$$

Where B and W for the coordinate **a** and **b** on the black and white backgrounds. The high OP value means high opalescence of the material.¹⁷⁶

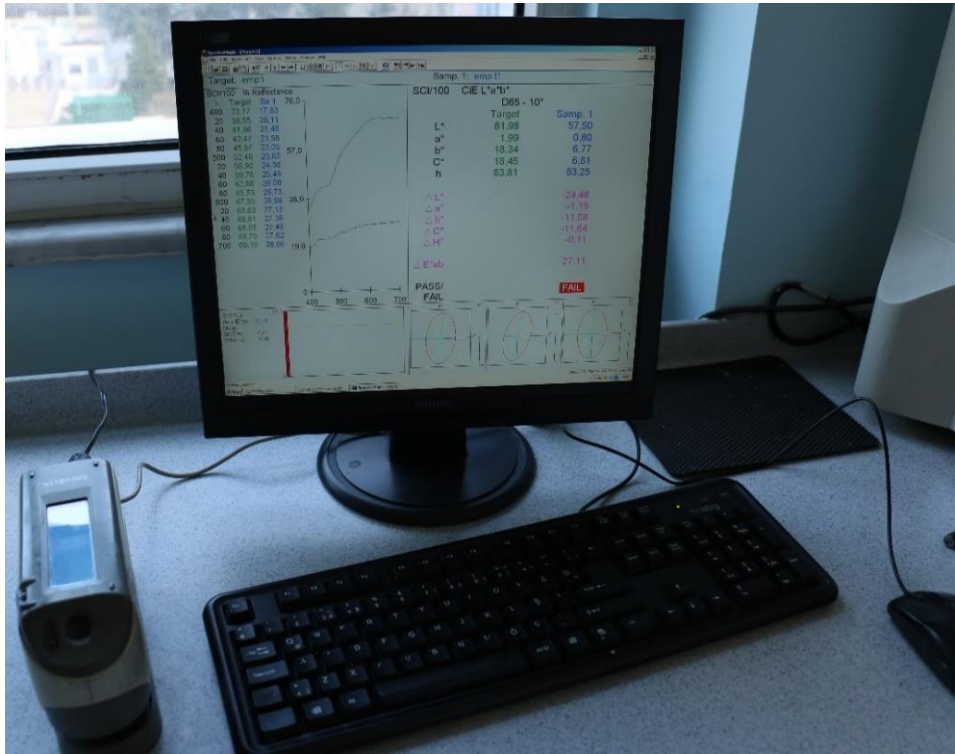


Figure 3.25: Spectrophotometer MINOL TA CM-2600 Konica Minolta Sensing.inc, Tokyo, Japan

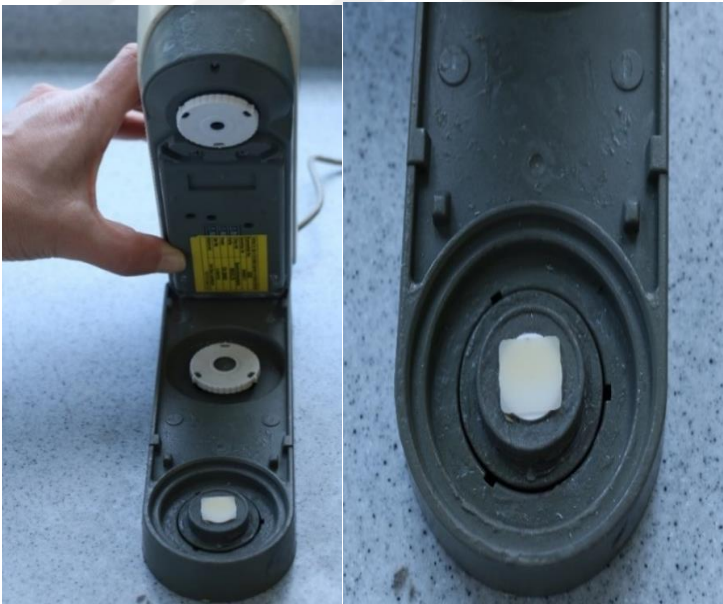


Figure 3.26: The specimen on a white background

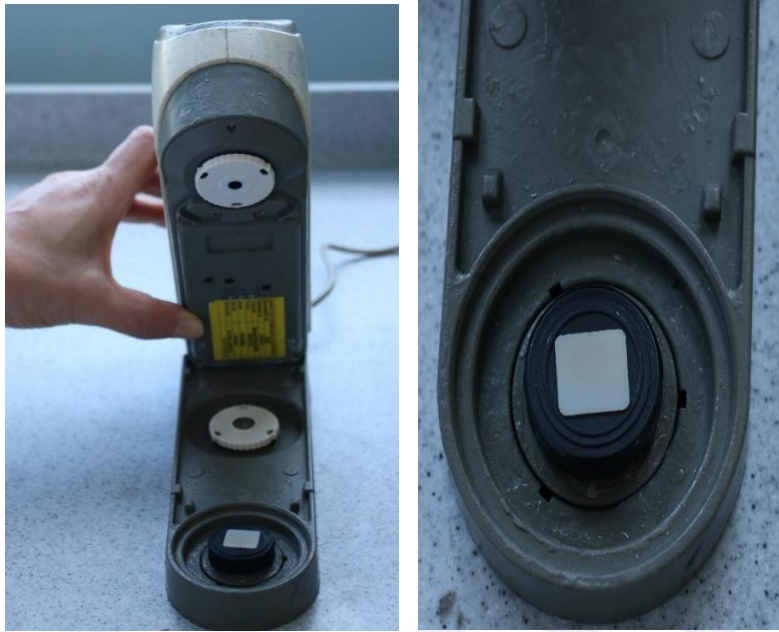


Figure 3.27: The specimen on a black background

3.4. Scanning Electron Microscope (SEM) Evaluation

Two specimens were randomly selected and assessed under an scanning electron microscope (SEM) from each tested subgroup of material.

The specimens were selected after flexural test from both groups of thermocycling and without thermocycling.

Samples were then sputtered with gold and surface topography was assessed under a scanning electron microscope. (Carl Zeiss Supra 50 VP FESEM, Carl Zeiss, Oberkochen, Germany) at low and high magnification (Figure 3.28).

SEM analysis was accomplished on the polished surface of every specimens focusing on changes of surface morphology for each materials after mechanical tests and thermal aging.



Figure 3.28: Gold-sputtered specimens

3.5. Statistical analysis

Statistical analysis was performed with SPSS (statistical package for the social sciences) v.25 (IBM, New York, NY). Statistical significance level was established at $p < 0.05$.

Two-way ANOVA test was conducted to examine the effects of material type and thermal aging on flexural strength, surface microhardness, ΔE , Translucency Parameter (TP), and Opalescence Parameter (OP).

One-way ANOVA test were used to study the main effects and simple main effects of material type on the studied variables.

All pairwise comparisons were done reported 95% confidence intervals and p-values are Bonferroni-adjusted.

Independent samples t-tests were used to study the simple main effect of thermal aging on the studied variables.

4. RESULTS

4.1. Flexural Strength

Below table and graph show the descriptive statistical analysis for flexural strength values in material groups with and without thermal aging (Table 4.1).

Table 4.1: Flexural Strength (MPa) in material groups with and without thermal aging

Groups	Aging	Mean	Standard Deviation	Minimum	Maximum	95% CL for Mean	
						Lower	Upper
Inc	Thermal	1407.8	321.8	1863.3	905.0	1177.6	1638.0
	Non	1452.3	160.9	1693.0	1225.7	1337.1	1567.4
Zir	Thermal	1499.9	316.0	1951.5	1049.4	1273.8	1725.9
	Non	1395.3	259.0	1754.4	977.9	1210.0	1580.6
Ips	Thermal	149.5	27.0	206.6	107.7	130.2	168.7
	Non	162.2	27.3	191.6	106.2	142.7	181.7
Cel	Thermal	328.3	43.9	417.2	279.3	296.9	359.7
	Non	363.7	21.8	395.1	320.0	348.1	379.3

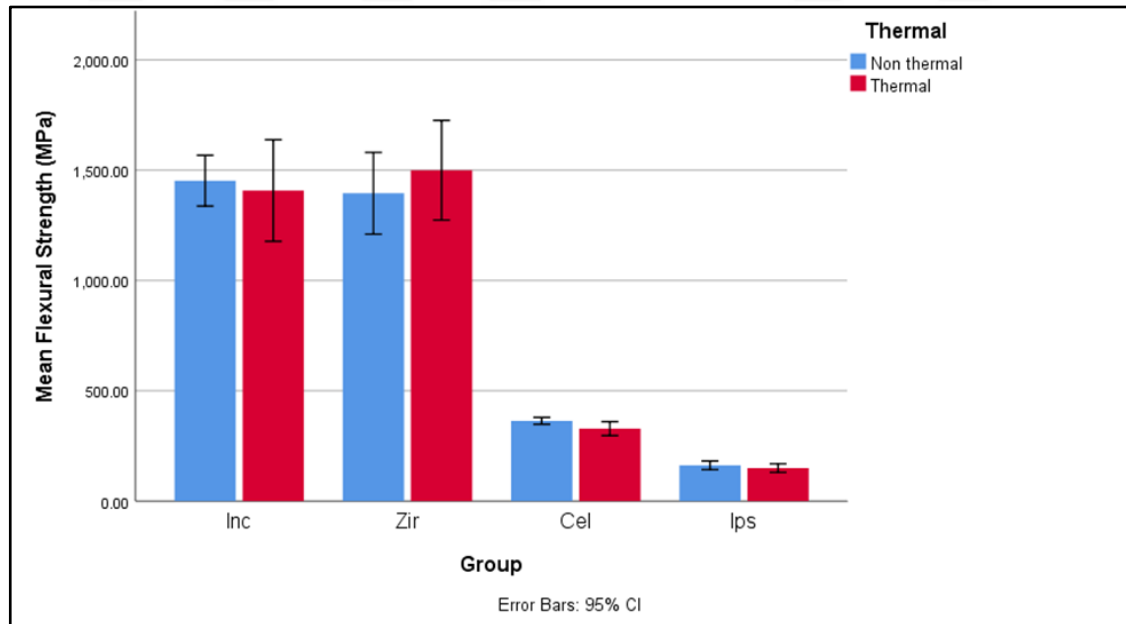


Figure 4.1: Graph of Clustered Bar Illustrates the mean Flexural Strength (MPa) in material groups with and without thermal aging

The two-way ANOVA test was conducted to examine the effects of material type and thermal aging on flexural strength. The interaction effect between material type and thermal aging on flexural strength was not statistically significant ($p=0.596$). Therefore, analyses of main effects for material type and thermal aging were performed, which indicated that the main effect of thermal aging was not statistically significant ($p=0.945$), while the main effect of material type was statistically significant ($p<0.05$) (Table 4.2) (Figure 4.1).

Table 4.2: Two-way ANOVA test to determine the effects of material type and thermal aging on flexural strength

Source	Type III Sum of Squares	Mean Square	F	P
Material Type	28586825.355	9528941.785	253.845	0.000*
Aging	180.300	180.300	0.005	0.945
Material Type * Aging	71374.188	23791.396	0.634	0.596

* Significant at $p<0.05$

While the material groups were arranged as IPS Empress CAD (155.8 ± 27.2) < Celtra DUO (346 ± 38.3) < inCoris TZI C (1430.7 ± 248.7) < IPS e.max ZirCAD (1447.6 ± 286.3) according to their flexural strengths values (MPa), no statistically significant difference was found between inCoris TZI C and IPS e.max ZirCAD ($p=1.000$). However there was a statistically significant difference between each other two groups ($p<0.05$). All pairwise comparisons were done reported 95% confidence intervals and p-values are Bonferroni-adjusted. Data are mean $\pm$ standard deviation, unless otherwise stated (Table 4.3, 4.4).

Table 4.3: Mean $\pm$ Sd. of flexural strength by material type

Test	InCoris TZI C	IPS e.max ZirCAD	Celtra DUO	IPS Empress CAD	P-value
Flexural Strength	1430 ± 248.7^a	1447.6 ± 286.3^a	346 ± 38.3^b	155.8 ± 27.2^c	0.000*

* Significant at $p<0.05$

The flexural strength means sharing same superscript are not significantly different each other.

Table 4.4: Pairwise comparisons of flexural strength between material groups.

Materials Groups		Mean Difference	p	95% CL for Difference	
				Lower	Upper
Cel	Inc	-1084.0	0.000*	-1250.3	-917.8
	Zir	-1101.6	0.000*	-1267.8	-935.4
	Ips	190.2	0.016*	23.9	356.4
Inc	Zir	-17.6	1.000	-183.8	148.7
	Ips	1274.2	0.000*	1108.0	1440.4
Zir	Ips	1291.8	0.000*	1125.5	1458.0

* Significant at $p < 0.05$

4.2. Surface Microhardness

Below table and graph show the descriptive statistics for Vickers Hardness Number values in material groups with and without thermal aging (Table 4.5).

Table 4.5: Vickers Hardness Number values (VHN) in material groups with and without thermal aging.

Material Groups	Aging	Mean	Standard Deviation	Minimum	Maximum	95% CL for Mean	
						Lower	Upper
Inc	Thermal	1334.70	76.21	1221.67	1431.33	1280.18	1389.22
	Non	1291.93	64.06	1196.33	1383.00	1246.10	1337.76
Zir	Thermal	1299.53	136.93	1167.00	1568.00	1201.58	1397.49
	Non	1288.83	28.81	1256.67	1332.33	1268.22	1309.44
Cel	Thermal	600.75	36.95	526.57	665.27	574.32	627.19
	Non	711.14	42.54	664.03	776.63	680.70	741.57
Ips	Thermal	675.90	55.18	623.80	791.60	636.43	715.37
	Non	549.03	58.08	476.63	653.60	507.48	590.57

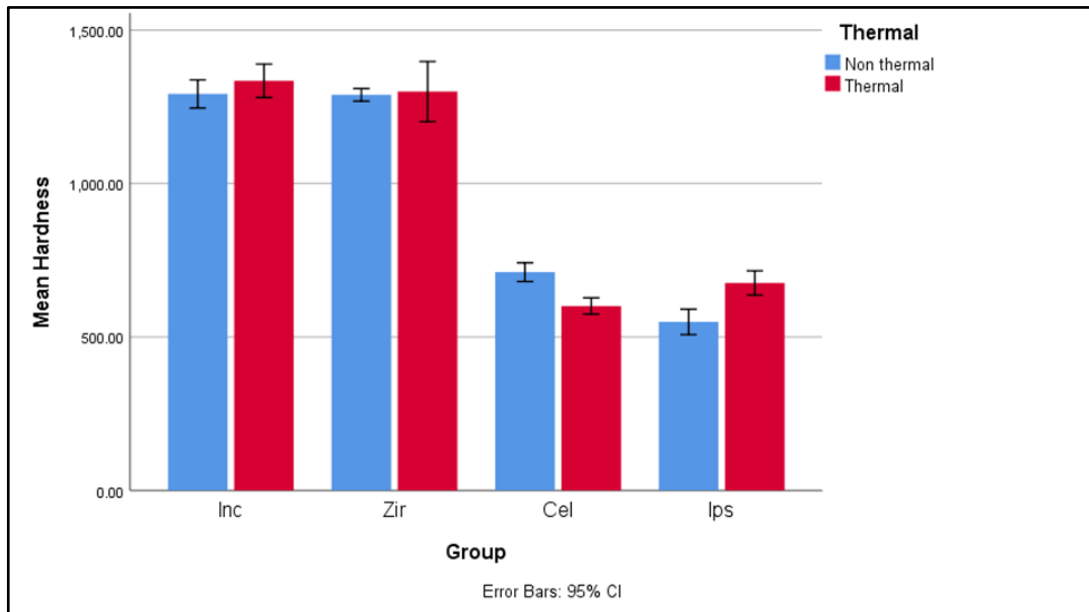


Figure 4.2: Clustered Bar Illustrates Mean of Vickers Hardness Number values (VHN) in material groups with and without thermal aging

The two-way ANOVA test was carried out to examine the effects of material type and thermal aging on surface microhardness. There was a statistically significant interaction between material type and thermal aging on surface microhardness ($p < 0.05$). Therefore, analyses of simple main effects for material type and thermal aging were performed with statistical significance receiving a Bonferroni adjustment (Table 4.6).

There were statistically significant differences in mean VHN values between material groups with thermal aging ($p < 0.05$), and without thermal aging ($p < 0.05$) (Table 4.7).

There was a statistically significant interaction between material type and thermal aging on surface microhardness ($p < 0.05$). Therefore, analyses of simple main effects for material type and thermal aging were performed (Table 4.6).

Table 4.6: Two-way ANOVA test to determine the effects of material type and thermal aging on surface microhardness

Source	Type III Sum of Squares	Mean Square	F	p
Material Type	8988399.856	2996133.285	613.244	.000*
Aging	6117.419	6117.419	1.252	.267
Material Type * Aging	145006.585	48335.528	9.893	.000*

* Significant at $p < 0.05$

There were statistically significant differences in mean VHN values between material groups with thermal aging ($p < 0.05$), and without thermal aging ($p < 0.05$) (Table 4.7 a and b).

Table 4.7 a and b: Simple main effect of material type on surface microhardness (VHN) in thermal and non-thermal aging groups

Aging	inCoris TZI C	IPS e.max ZirCAD	Celtra DUO	IPS Empress CAD	Sig.
Thermal	1334.70 ± 76.21	1299.53 ± 136.93	600.75 ± 36.95	675.9 ± 55.18	0.000*
Non	1291.93 ± 64.06	1288.83 ± 28.81	711.14 ± 42.54	549.03 ± 58.08	0.000*

Aging	Sum of Squares	Mean Square	F	Sig.
Thermal	4641977.221	1547325.740	316.704	0.000*
Non	4491429.220	1497143.073	306.433	0.000*

* Significant at $p < 0.05$

In thermal aging subgroups, while the material groups were arranged as Celtra DUO (600.75 ± 36.95) < IPS Empress CAD (675.9 ± 55.18) < IPS e.max ZirCAD (1299.53 ± 136.93) < inCoris TZI C (1334.7 ± 76.21) according to their VHN values (Table 4.7.a), no statistically significant difference was found between Celtra DUO and IPS Empress CAD ($p=0.113$) and between inCoris TZI C and IPS e.max ZirCAD ($p=1.000$). However there was a statistically significant difference between each other two groups ($p<0.05$) (Table 4.8).

In non-thermal aging subgroups, the material groups were arranged as IPS Empress CAD (549.03 ± 58.08) < Celtra DUO (711.14 ± 42.54) < IPS e.max ZirCAD (1288.83 ± 28.81) < inCoris TZI C (1291.93 ± 64.06) according to their VHN values (Table 4.7.a). No statistically significant difference was found between inCoris TZI C and IPS e.max ZirCAD ($p=1.000$), however there was a statistically significant difference between Celtra DUO and IPS Empress CAD ($p<0.05$) (Table 4.8).

Table 4.8: Pairwise comparisons of Vickers Hardness Number values (VHN) between material groups based on thermal aging

Material Groups			Mean Difference	p	95% CL for Difference	
					Lower	Upper
Thermal	Cel	Inc	-733.9	0.000*	-818.8	-649.1
		Zir	-698.8	0.000*	-783.6	-614.0
		Ips	-75.1	0.113	-160.0	9.7
	Inc	Zir	35.2	1.000	-49.6	120.0
		Ips	658.8	0.000*	574.0	743.6
	Zir	Ips	623.6	0.000*	538.8	708.4
Non	Cel	Inc	-580.8	0.000*	-665.6	-496.0
		Zir	-577.7	0.000*	-662.5	-492.9
		Ips	162.1	0.000*	77.3	246.9
	Inc	Zir	3.1	1.000	-81.7	87.9
		Ips	742.9	0.000*	658.1	827.7
	Zir	Ips	739.8	0.000*	655.0	824.6

* Significant at $p<0.05$

Celtra DUO subgroup with thermal aging had a statistically significantly lower VHN mean value than non-thermal aging Celtra DUO subgroup by 110.4 VHN ($p=0.001$), while IPS Empress CAD subgroup with thermal aging had a statistically

significantly higher VHN mean value than non-thermal subgroup by 126.9 VHN ($p < 0.05$). However, no statistically significant differences were found between thermal aging and non-thermal aging subgroups in Coris TZI C by 42.8 VHN ($p = 0.176$), and same result for in IPS e.max ZirCAD subgroups by 10.7 VHN ($p = 0.733$) (Table 4.9).

Table 4.9: Simple main effect of thermal aging on Vickers Hardness Number (VHN) in material groups

Material Groups	Thermal (Mean $\pm$ Sd.)	Non-Thermal (Mean $\pm$ Sd.)	Mean Difference	p	95% CL for Difference	
					Lower	Upper
Inc	1334.70 $\pm$ 76.21	1291.93 $\pm$ 64.06	42.8	0.176	-19.5	105.1
Zir	1299.53 $\pm$ 136.93	1299.53 $\pm$ 136.93	10.7	0.733	-51.6	73.0
Cel	600.75 $\pm$ 36.95	711.14 $\pm$ 42.54	-110.4	0.001*	-172.7	-48.1
Ips	675.9 $\pm$ 55.18	549.03 $\pm$ 58.08	126.9	0.000*	64.6	189.2

* Significant at $p < 0.05$

4.3. Color difference (ΔE values)

Below table and graph show the descriptive statistics for ΔE values in material groups with and without thermal aging (Table 4.10) (Figure 4.3).

Table 4.10: ΔE in material groups with and without thermal aging.

Material Groups	Aging	Mean	Standard Deviation	Minimum	Maximum	95% CL for Mean	
						Lower	Upper
Cel	Thermal	0.54	0.18	0.30	0.85	0.41	0.67
	Non	0.01	0.01	0.00	0.02	0.01	0.02
Inc	Thermal	0.42	0.09	0.31	0.52	0.36	0.48
	Non	0.19	0.14	0.03	0.50	0.09	0.29
Zir	Thermal	0.53	0.31	0.22	1.26	0.31	0.76
	Non	0.04	0.03	0.01	0.09	0.02	0.06
Ips	Thermal	2.78	1.21	1.02	4.30	1.92	3.65
	Non	0.03	0.05	0.00	0.16	-0.01	0.07

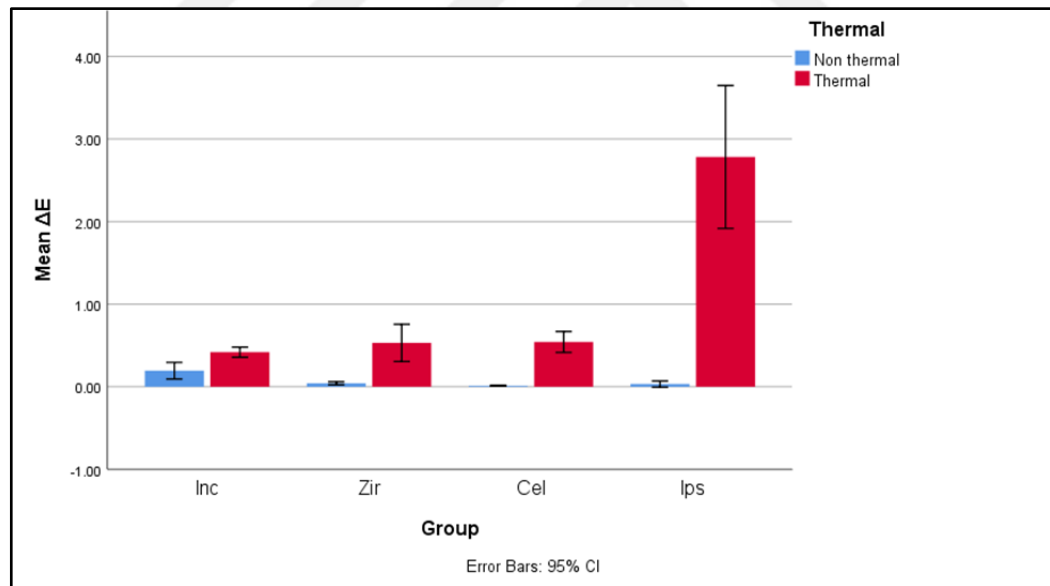


Figure 4.3: Clustered Bar Illustrates Mean of ΔE in material groups with and without thermal aging

The two-way ANOVA was performed to examine the effects of material type and thermal aging on ΔE values. There was a statistically significant interaction between material type and thermal aging on ΔE ($p < 0.05$). Therefore, analyses of simple main

effects for material type and thermal aging were performed with statistical significance receiving a Bonferroni adjustment (Table 4.11).

There was a statistically significant interaction between material type and thermal aging on ΔE ($p < 0.05$) (Table 4.11).

Therefore, analyses of simple main effects for material type and thermal aging were performed (Table 4.12).

Table 4.11: Two-way ANOVA test to determine the effects of material type and thermal aging on ΔE

Source	Type III Sum of Squares	Mean Square	F	p
Material Type	18.758	6.253	30.786	0.000*
Aging	19.960	19.960	98.275	0.000*
Material Type * Aging	20.741	6.914	34.041	0.000*

* Significant at $p < 0.05$

There was a statistically significant difference in mean ΔE between material subgroups with thermal aging ($p < 0.05$), but no statistically significant difference was found without thermal aging ($p = 0.791$) (Table 4.12).

Table 4.12: Simple main effect of material type on ΔE_{ab} * in thermal and non-thermal aging groups

Aging	inCoris TZI C	IPS e.max ZirCAD	Celtra DUO	IPS Empress CAD	Sig.
Thermal	0.42 ± 0.09	0.53 ± 0.31	0.54 ± 0.18	2.78 ± 1.21	0.000*
Non	0.19 ± 0.14	0.04 ± 0.03	0.01 ± 0.01	0.03 ± 0.05	0.791

* Significant at $p < 0.05$

In thermal aging subgroups, while the material groups were arranged as inCoris TZI C (0.42 ± 0.09) < IPS e.max ZirCAD (0.53 ± 0.31) < Celtra DUO (0.54 ± 0.18) < IPS Empress CAD (2.78 ± 1.21) (Table 4.12) according to mean ΔE values, no statistically significant differences were found between Celtra DUO and both inCoris TZI C ($p = 1.000$) and IPS e.max ZirCAD ($p = 1.000$) and also no statistically difference were found between inCoris TZI C and IPS e.max ZirCAD ($p = 1.000$), however mean ΔE

values in IPS Empress CAD subgroup with thermal aging was statistically significantly higher than all other subgroups ($p<0.05$) (Table 4.13).

In non-thermal aging subgroups, according to ΔE values there were no statistically significant difference among the tested groups ($p<0.05$).

Table 4.13: Pairwise comparisons of ΔE values between material groups based in thermal aging groups

Material Groups			Mean Difference	p	95% CL for Difference	
					Lower	Upper
Thermal	Cel	Inc	0.12	1.000	-0.42	0.67
		Zir	0.01	1.000	-0.54	0.56
		Ips	-2.24	0.000*	-2.79	-1.69
	Inc	Zir	-0.11	1.000	-0.66	0.43
		Ips	-2.37	0.000*	-2.91	-1.82
	Zir	Ips	-2.25	0.000*	-2.80	-1.71

* Significant at $p<0.05$

Celtra DUO subgroup with thermal aging had a statistically significantly higher ΔE mean value than non-thermal subgroup by 0.53 ($p=0.010$), and IPS e.max ZirCAD subgroup with thermal aging had a statistically significant higher mean ΔE value than non-thermal subgroup by 0.49 ($p=0.018$). Also, IPS Empress CAD subgroup with thermal aging had a statistically significant higher mean ΔE value than non-thermal by 2.75 ($p<0.05$). However, no statistically significant differences were found between thermal aging and non-thermal aging subgroups in inCoris TZI C ($p=0.270$).

Table 4.14: Simple main effect of thermal aging on ΔE value in material groups.

Materials	Thermal (Mean $\pm$ Sd.)	Non Thermal (Mean $\pm$ Sd.)	Mean Difference	p	95% CL for Difference	
					Lower	Upper
Celtra DUO	0.54 $\pm$ 0.18	0.01 $\pm$ 0.01	0.53	0.010*	0.13	0.93
InCoris TZI C	0.42 $\pm$ 0.09	0.19 $\pm$ 0.14	0.22	0.270	-0.18	0.63
IPS e,max ZirCAD	0.53 $\pm$ 0.31	0.04 $\pm$ 0.03	0.49	0.018*	0.09	0.89
IPS Empress CAD	2.78 $\pm$ 1.21	0.03 $\pm$ 0.05	2.75	0.000*	2.35	3.15

* Significant at $p < 0.05$

4.4. Translucency Parameter (TP)

Below table and graph show the descriptive statistics for translucency parameter (TP) values in material groups with and without thermal aging (Table 4.15).

Table 4.15: TP in material groups with and without thermal aging.

Materials Groups	Aging	Mean	Standard Deviation	Minimum	Maximum	95% CL for Mean	
						Lower	Upper
Ips	Thermal	21.00	0.57	20.23	22.01	20.59	21.40
	Non	20.46	0.92	18.86	21.70	19.80	21.12
Cel	Thermal	20.41	0.54	19.71	21.48	20.02	20.79
	Non	20.09	0.33	19.62	20.84	19.85	20.33
Inc	Thermal	0.78	0.38	0.44	1.46	0.51	1.05
	Non	1.45	1.50	0.48	5.51	0.37	2.52
Zir	Thermal	1.31	0.69	0.57	3.02	0.81	1.80
	Non	1.18	0.72	0.65	3.04	0.67	1.70

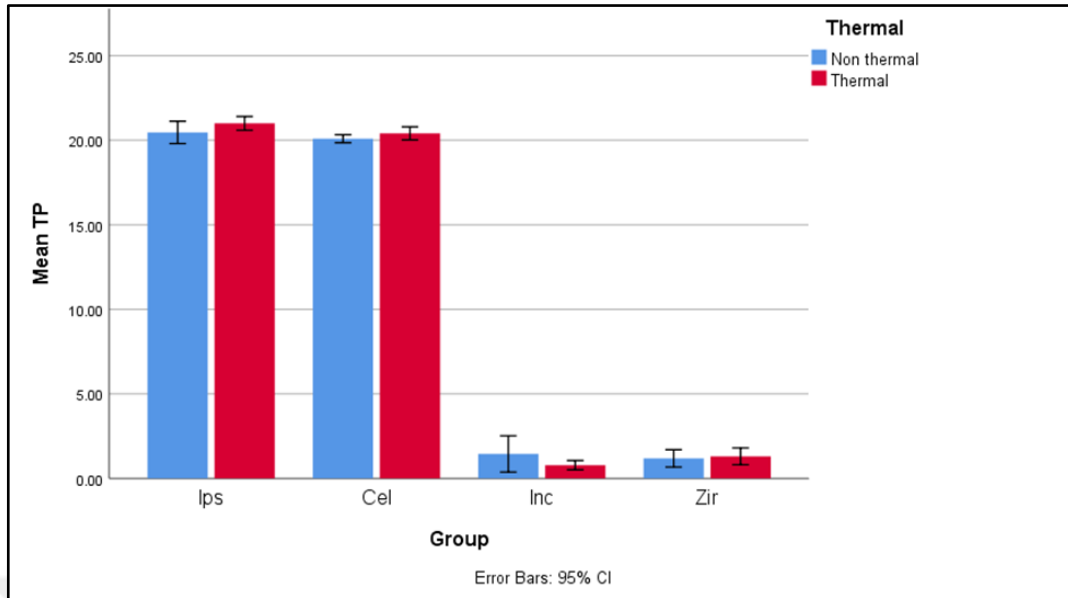


Figure 4.4: Clustered Bar Illustrates Mean of TP in material groups with and without thermal aging.

The two-way ANOVA test was run to examine the effects of translucency parameter (TP) by material type and thermal aging. The interaction effect between material type and thermal aging on translucency parameter (TP) was not statistically significant ($p=0.095$). Therefore, analyses of main effects for material type and thermal aging were conducted, which indicated that the main effect of thermal aging was not statistically significant ($p=0.664$), while the main effect of material type was statistically significant ($p<0.05$) (Table 4.16).

Table 4.16: Two-way ANOVA test to determine the effects of material type and thermal aging on TP.

Source	Type III Sum of Squares	Mean Square	F	p
Material Type	7460.987	2486.996	4014.889	0.000
Aging	0.118	0.118	0.190	0.664
Material Type * Aging	4.103	1.368	2.208	0.095

* Significant at $p<0.05$

All pairwise comparisons were performed, where reported 95% confidence intervals and p-values are Bonferroni-adjusted. Data are mean $\pm$ standard deviation, unless otherwise stated.

While the material groups were arranged as inCoris TZI C (1.11 ± 1.12) < IPS e.max ZirCAD (1.24 ± 0.69) < Celtra DUO (20.25 ± 0.46) < IPS Empress CAD (20.73 ± 0.79), according to mean TP values no statistically significant difference was found between inCoris TZI C and IPS e.max ZirCAD ($p=1.000$), and between Celtra DUO and IPS Empress CAD ($p=0.339$). However, there was statistically significant difference between each other two groups ($p<0.05$) (Table 4.17 and 4.18).

Table 4.17: Mean $\pm$ Sd. of TP by material type

Test	inCoris TZI C	IPS e.max ZirCAD	Celtra DUO	IPS Empress CAD	P-value
TP	1.11 ± 1.12^a	1.24 ± 0.69^a	20.25 ± 0.46^b	20.73 ± 0.79^a	0.000*

* Significant at $p<0.05$

Table 4.18: Pairwise comparisons of TP between material groups

Materials Groups		Mean Difference	p	95% CL for Difference	
				Lower	Upper
Cel	Inc	19.14	0.000*	18.46	19.81
	Zir	19.00	0.000*	18.33	19.68
	Ips	-0.48	0.339	-1.16	0.19
Inc	Zir	-0.13	1.000	-0.81	0.54
	Ips	-19.62	0.000*	-20.29	-18.94
Zir	Ips	-19.49	0.000*	-20.16	-18.81

* Significant at $p<0.05$

4.5. Opalescence Parameter (OP)

Below table and graph show the descriptive statistics for opalescence parameter (OP) values in material groups with and without thermal aging (Table 4.19).

Table 4.19: OP in material groups with and without thermal aging.

Materials Groups	Aging	Mean	Standard Deviation	Minimum	Maximum	95% CL for Mean	
						Lower	Upper
Cel	Thermal	12.98	0.45	11.95	13.40	12.65	13.30
	Non	12.96	0.20	12.67	13.15	12.82	13.10
Ips	Thermal	10.01	0.72	8.58	10.73	9.50	10.53
	Non	10.61	1.73	6.51	12.12	9.37	11.85
Inc	Thermal	0.62	0.36	0.29	1.23	0.37	0.88
	Non	0.54	0.38	0.16	1.22	0.26	0.81
Zir	Thermal	0.73	0.54	0.34	2.19	0.34	1.12
	Non	0.84	0.69	0.34	2.65	0.35	1.34

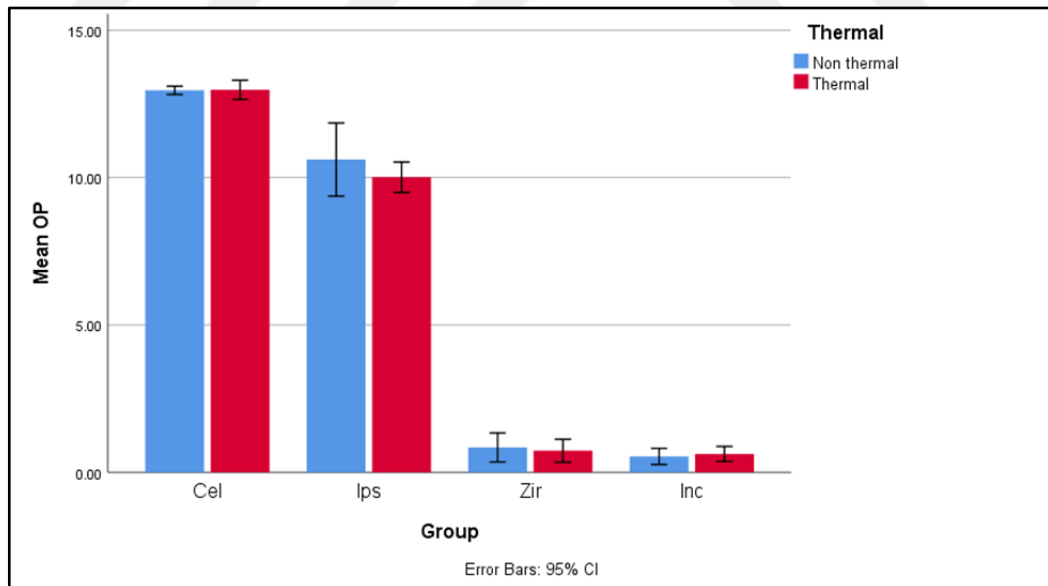


Figure 4.5: Clustered Bar Illustrates Mean (OP) in material groups with and without thermal aging

The two-way ANOVA test was accomplished to examine the effects of material type and thermal aging on opalescence parameter (OP). The interaction effect between

material type and thermal aging on OP was not statistically significant ($p=0.502$). Therefore, analyses of main effects for material type and thermal aging were performed, which indicated that the main effect of thermal aging was not statistically significant ($p=0.384$), while the main effect of material type was statistically significant ($p<0.05$) (Table 4.20).

Table 4.20: Two-way ANOVA test to determine the effects of material type and thermal aging on OP

Source	Type III Sum of Squares	Mean Square	F	p
Material Type	2471.626	823.875	1369.699	0.000*
Aging	0.462	0.462	0.768	0.384
Material Type * Aging	1.430	0.477	0.792	0.502

* Significant at $p < 0.05$

All pairwise comparisons were run where reported 95% confidence intervals and p-values are Bonferroni-adjusted. Data are mean $\pm$ standard deviation, unless otherwise stated.

While the material groups were arranged as inCoris TZI C (0.58 ± 0.36) < IPS e.max ZirCAD (0.79 ± 0.61) < IPS Empress CAD (10.31 ± 1.33) < Celtra DUO (12.97 ± 0.34) according to mean opalescence parameter (OP), no statistically significant difference was found between inCoris TZI C and IPS e.max ZirCAD ($p=1.000$), however there were statistically significant difference among each other tested groups ($p<0.05$) (Table 4.21 and 4.22).

Table 4.21: Mean $\pm$ Sd. of OP by material type.

Test	inCoris TZI C	IPS e.max ZirCAD	Celtra DUO	IPS Empress CAD	P-value
OP	0.58 ± 0.36^a	0.79 ± 0.61^a	12.97 ± 0.34^b	10.31 ± 1.33^c	0.000*

* Significant at $p<0.05$

Table 4.22: Pairwise comparisons of OP between material groups.

Materials Groups		Mean Difference	p	95% CL for Difference	
				Lower	Upper
Cel	Inc	12.39	0.000*	11.72	13.05
	Zir	12.18	0.000*	11.51	12.85
	Ips	2.66	0.000*	1.99	3.32
Inc	Zir	-0.21	1.000	-0.87	0.46
	Ips	-9.73	0.000*	-10.40	-9.07
Zir	Ips	-9.53	0.000*	-10.19	-8.86

* Significant at $p < 0.05$

4.6. Scanning Electron Microscope (SEM) Evaluation

During the specimen preparation procedure and after flexural strength and micro hardness tests high kinetic energy developed affected the thin sample of CAD/CAM restorative material lead to crack formation.

From each subgroup of tested materials one specimen was selected randomly and examined under the scanning electron microscope (SEM) for microstructure changes and crack propagation or other defects.

Figure 4.6 showed the original microstructure of four samples from, Celtra DUO, IPS Empress CAD, IPS e.maxZir CAD and inCoris TZI C before mechanical tests.

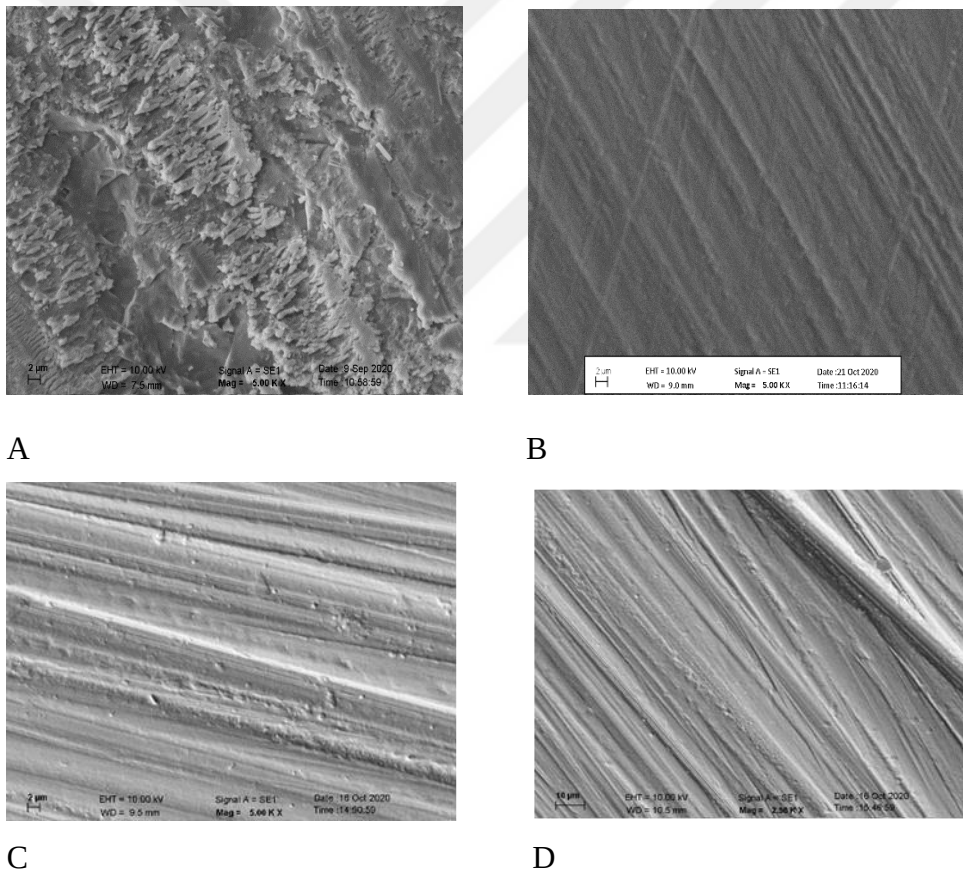


Figure 4.6: SEM images of original microstructure of four different materials.

- A. The crystalline microstructure of IPS Empress CAD without zirconia particles
- B. The glass with completely dissolved zirconia of Celtra DUO
- C. IPS e.max ZirCAD
- D. inCoris TZI C

For the Celtra DUO zirconia reinforced lithium silicate with the inclusion of 10% zirconium oxide, the crystallites are more and small in size and not differentiated from the matrix. This result in ultrafine microstructure leads to a positive effect on the optical and mechanical properties of the material made it more translucent and has a higher opalescence parameter.

The group of non-thermal aging samples showed well-aligned parallel crystals with no spacing, on the contrary to the thermally aged group where the arrangement of the crystals was more irregular.

The image for IPS Empress CAD shows a more crystalline structure and less homogenous microstructure than Celtra DUO which gave it average flexural strength.

The white spots seen in both the groups are the coloring agent. The cracks lines were interrupted by the crystals in both groups after the flexural strength test and indentations of surface microhardness test (Figure 4.7).

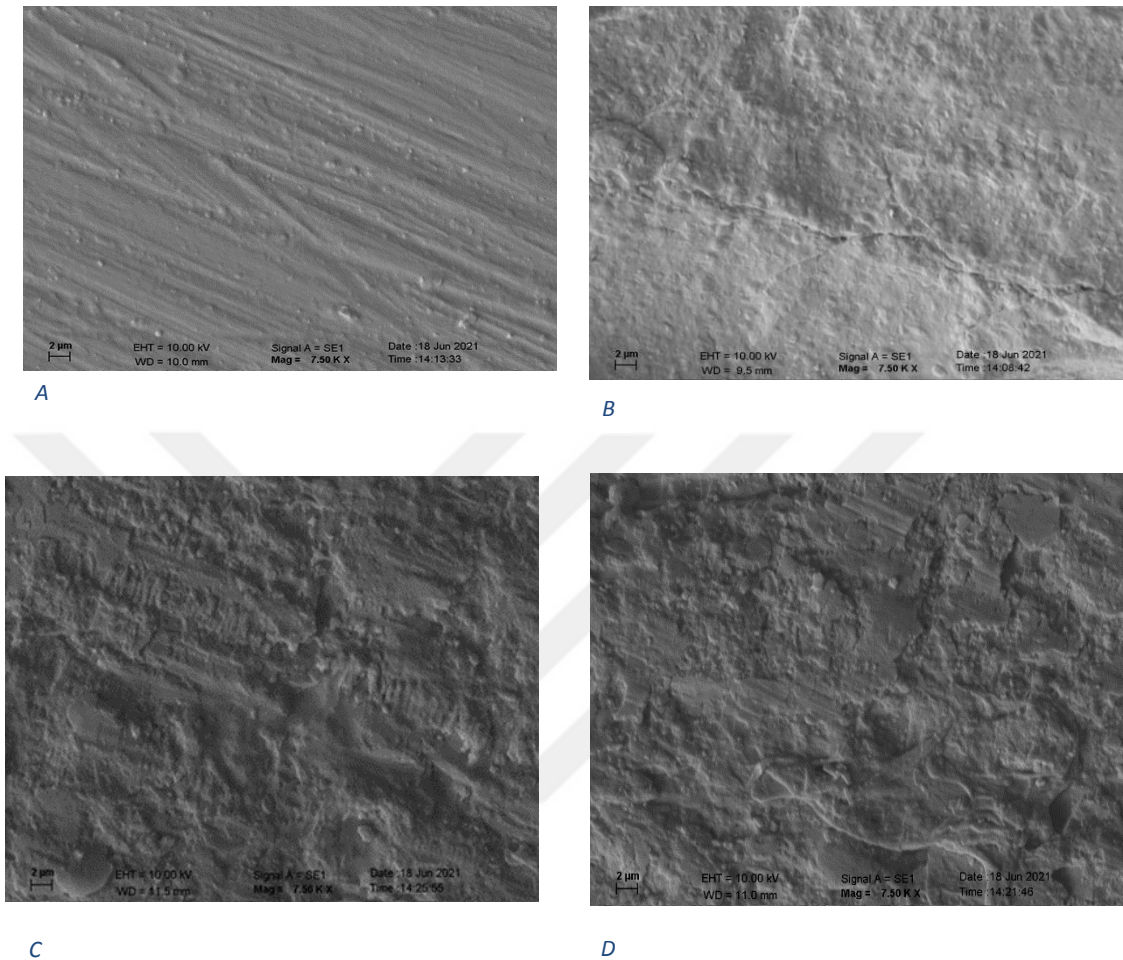


Figure 4.7: One sample from each group of glass ceramics after flexural strength and hardness tests, under x7500 original magnification

A Celtra DUO non thermocycling subgroup.

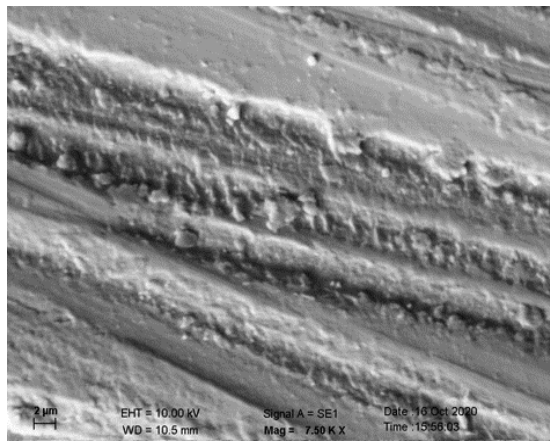
B-Celtra DUO thermocycled subgroup.

C- Ips Empress CAD non thermocycling subgroup.

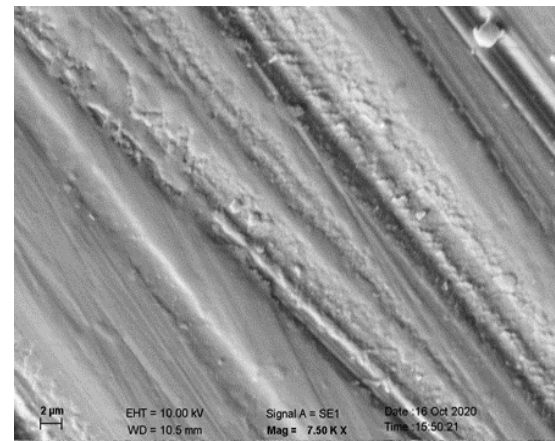
D- IPS Empress CAD thermocycling subgroup.

The monolithic zirconia samples for both types inCoris TZI C, IPS e.max ZirCAD due polycrystalline structure with small size crystals and minimal porosity showed diffuse compact appearance with interrupted crack propagation after mechanical testes.

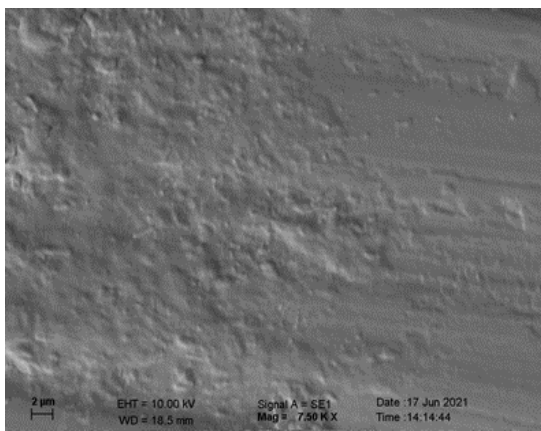
The thermocycling subgroups for both have more homogenous matrix in the shape and the crystals less prominent and there is clear notches of indentions with prominent white spots for coloring agent (Figure 4.8).



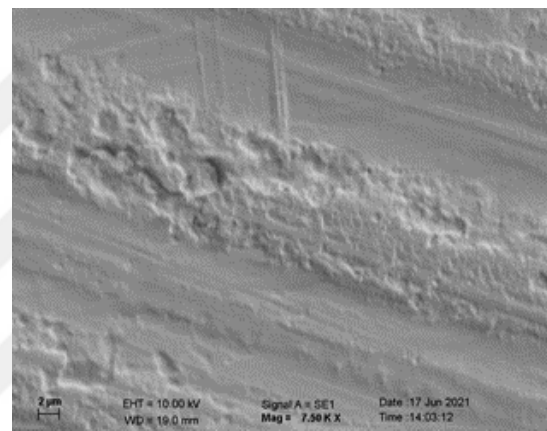
A



B



C



D

Figure 4.8: One sample from each group of zirconia material after flexural strength and hardness tests, under x7500 original magnification

- A. inCoris TZI C non-thermocycling subgroup
- B. inCoris TZI C thermocycling subgroup
- C. IPS e.max ZirCAD non-thermocycling subgroup
- D. IPS e.max ZirCAD thermocycling subgroup

5. DISCUSSION

Since several years ago, as material of choice for different dental restorations, various kinds of ceramics have been used depending on their favorable mechanical and esthetic properties. In despite to their high biocompatibility and translucency still their brittleness , limited resistance to crack spread and low tensile strength considered as main drawback for ceramic.¹⁷⁷

With the constantly evolving of ceramic and zirconia restorative materials, the monolithic restorations particularly, monolithic zirconia appeared as a feasible dental restorative material, these materials exhibited a 100% survival rate after 36 to 68 months in a study by Moscovitch et al.¹⁷⁸ and there is no cracks or fracture within monolithic zirconia restorations reported.

For testing of the actual dental restorative material performance, the long term clinical studies is the best for their evaluation but, these studies time consuming and involved ethical approvals and long clinical procedure therefore, the laboratory testing or in-vitro studies provide scientific data helps in material evaluations.^{179,81}

In the current study, all the test parameters, color of tested materials, and specimen's preparations were standardized to restrict the variable factors. And all the materials tested in the studies have to fulfill the requirements for Class 2 of the ISO standard classification.¹⁸⁰ Where the obtained a result of mean flexural strength greater than the requirement of 100 MPa which is an acceptable for clinical applications.¹⁸¹

The load in the mouth varies from one region to another, it is increasing from about 90-340 N in the anterior teeth area to about 220-450 N in the region of premolar teeth to about 400-900 N in the region of molar teeth.¹⁸² Therefore, all tested materials in

the present study during the mechanical tests procedure subjected to the load did not exceed the maximum load determined in previous studies.

The thermocycling in a wet environment was used for aging subgroups from each brand of the tested material to imitate the oral cavity condition and to be similar to the fatigue process in the clinical situation as used in many previous studies.^{183,142,184}

While surface finishing and polishing may affect the outcome of the biaxial bending strength test for all materials tested in the present study, each sample has been finished and polished to a uniform one surface.¹⁸⁵

This present in-vitro study directed to compare four types of monolithic ceramic materials available on the market for the CEREC CAD/CAM system from many aspects regarding their mechanical and optical properties.

Although, an in-vitro study was limited, its result provided important information about the materials behaviors which can help in clinical applications of dental restorative materials.¹²⁴

The current in-vitro study evaluated and compared the mechanical properties (flexural strength and surface microhardness), and optical properties (color difference, translucency and opalescence) of two types of monolithic zirconia (inCoris TZI C, and IPS e.max ZirCAD), a zirconia reinforced lithium silicate (Celtra DUO) and a leucite-reinforced glass ceramic (IPS Empress CAD) ceramic materials with and without thermal aging of the specimens.

The result of this study disclosed that the mechanical properties (flexural strength and surface microhardness) and optical properties (color difference, translucency and opalescence) showed statistically significant differences between material types, and

there is thermocycling effect on the materials sub groups in hardness and color difference test. Therefore, the null hypotheses have been partially rejected.

Regarding the mechanical properties evaluations, the present results demonstrated that, there is no statically significant difference for the main effect of thermal aging on flexural strength in all the groups of materials while the main effect of material type was statistically significant ($p < 0.05$). So for that we will compare the main groups for the material types only and there will be no comparison for subgroups.

Among all the tested groups IPS e.max ZirCAD was recorded the highest value of flexural strength about 1447.6 ± 286.3 MPa followed by inCoris TZI C having 1430.7 ± 248.7 MPa (the mean values for thermal and non-thermal subgroups) and no statistically significant difference was found between inCoris TZI C and IPS e.max ZirCAD ($p = 1.000$) in both subgroups for thermal and non-thermal aging.

The lowest value for flexural strength was recorded for IPS Empress CAD as 155.8 ± 27.2 MPa (the mean value for thermal and non-thermal subgroups) followed by Celtra DUO as 346 ± 38.3 MPa (mean value for both subgroups of thermal and non-thermal aging). This indicated that the monolithic zirconia groups were higher flexural strength and surface micro hardness than reinforced lithium silicate and lithium disilicate. These finding were compatible with earlier studies that evaluate the Y-TZP ceramic when subjected to high stress concentration.^{186,187}

The flexural strength of Celtra DUO specimens was recorded as 346 ± 38.3 MPa (mean value for thermal and non-thermal subgroups) where no statistically significant difference existed between thermal and non-thermal aged sub-groups of the same material. This result was matching with the manufacturer value (360 MPa) and agree with Wendler et al¹⁸⁸ who revealed that the Celtra DUO has an intermediate range of strength in compared with zirconia and silicate ceramics.

In this present study, the two types of monolithic zirconia (IPS e.max ZirCAD and inCoris TZI C) have a small amount of alumina oxide about 0.35 wt% which responsible for intensifying the aging resistance of 3YTZP ceramic materials as proved by previous studies^{20,189}

However, according to many previous studies the lowering of the amount of alumina to 0.1 or 0.05 wt% can enhance the translucency and at the same time lowered the hydrothermal stability.^{190,22}

Moreover, the microstructural homogeneity, low porosity and density of sintered monolithic zirconia lead to increasing flexural strength and enhance their optical properties.^{191,192}

These findings are consistent with the present study's results where SEM images for inCoris TZI C and IPS e.max ZirCAD monolithic zirconia showed more microstructural homogeneity in both the thermocycling subgroups and non-thermocycling subgroups and this is due to that material blocks subjected to high temperature during sintering before cutting of the specimens but these changes did not have any statistical significant difference in the flexural strength test and effect other tests.

Also, these results of this current study can be attributed to the many factors that greatly influence the mechanical and optical properties of two types of monolithic zirconia as well as the other two types of reinforced monolithic ceramic materials, this factor includes the phase composition, distribution of the crystalline phases and Y-TZP ceramic grain dimensions which are restricted by boundaries of the grains in micro structure.^{185,193,194}

In a study by Ban et al.¹⁹⁵ surface roughness of seven dental ceramic type finished with three diamond grinding tools and two diamond pastes was measured. The scanning electron micrographs of the dental ceramics has shown that after final sintering at 1350°C,

a tetragonal zirconia type with a high density body size about 0.3 mm and less phase transformation has been shown.

These proved previous studies results, gave explanation to the SEM images in the current study, which showed the surface morphology of each monolithic zirconia-based ceramic evident interrupted micro cracks in compact homogenous structure. This feature was missed in lithium silicate ceramics where IPS Empress CAD surface showed small cavities occupied by leucite crystals, and amount of micro-cracks in the glassy matrix (Figure 4.6).

The thermocycling procedure in the present study was used to expose the tested specimens to the changes of temperature that imitate oral environment where the several studies established that 10,000 cycles correspond approximately to 1 year of in vivo functioning, this calculation is based on the theory that such cycles might occur 20 to 50 times per day.¹⁹⁶ Many studies agreed, that the static tests like thermal cycling test can gave results on a longer period of time than that of dynamic tests, but it is mostly not proved results, while dynamic tests better for simulate the masticatory force to which dental restorative materials are subjected in the patient mouth.¹⁷⁴ It can be considered as limitations of current study, where we used static type of thermocycling with limited thermal cycles. Water storage and an aqueous oral environment promote subcritical crack growth and leads to a transformation of Y-TZP into monoclinic structure through stress-corrosion type mechanism in many studies prior to thermocycling. However, no systemic study of aging with dental ceramics has nevertheless been done under real clinical conditions until now.^{197,198} Also, there were not a lot of studies gave data about flexural strength and hardness before and after thermocycling cycling to compare it with present study.

Keuper et al.¹⁹⁹ stated that the constant rate in the 3Y-TZP layer is about 0.5 to 1 mm per year for low-temperature degradation. This rate is well defined in humid conditions and begins after the application of moisture, the similar rates has been seen at low partial water pressures, but several times before the degradation begins, the delay ranged from zero to 7 months.

Carvalho et al.²⁰⁰ concluded that aging in the water alone did not impact the flexural strength of a feldspathic ceramic by different number of temperature cycles as a result of their study, examined the effect of aging on the flexural strength of feldspathic ceramic by means of different thermocycling protocols. In the present study we used different materials with less thermal cycling. Lughy and Sergo²⁰¹ in their review summed up the scientific data and concluded that stabilizer type (oxides), grain size and rest stress are the key factors affecting zirconia aging. The static type of thermal cycling test was used in the current study for aging the tested material, which did not effect on the monolithic zirconia, but effect on the zirconia reinforced material for mechanical test and also effect the color difference of all tested material. This explained by that the result of static type of thermocycling test truly depended on the amount of stress generated and the mechanism of failure which appeared more effective in testing of bond strength rather than highly strength monolithic material without bonding materials.¹⁹⁶ Another cause is that the effects of thermal cycling process might be attributed to the diversity and composition of tested materials rather than time consuming.¹⁷⁴

Many studies proved that the most common and efficient way to imitate promoted low-temperature degradation in vitro is hydrothermal treatment at 134°C than thermocycling method for zirconia-based material,^{202,203} but this degree of temperature cannot be reached in normal oral conditions which we try to simulate it in the present study. Li et al.²⁰⁴ and Cenci et al.²⁰⁵ considered that the temperature on the tooth surface cannot reach the actual temperatures for hot and cold fluids ingested due to the buffering effect in the oral environment.

Ban has reviewed that numerous studies have shown that well-controlled zirconia has sufficient oral stability even after a slight transformation due to their high initial strength and adequate transformation durability.²⁰⁶

Chevalier et al.²⁰⁷ showed that 1 hour in autoclave aging at 134°C was the same as 3–4 hours in vivo at 37°C. He also premises that the transformation durability would

take 25 years to reach the 20% monoclinic content of zirconia and confirms that long-term stability.

Flexural strength examines the material's resistance under bending, which is a common type of force in prosthetic restorations, in the current study the flexural properties of monolithic zirconia showed no significant effect by thermo-cycling and this is maybe attributed to the amount of zirconia and alumina oxides in two types of monolithic zirconia.

Although, zirconia when transferred from the tetragonal to monoclinic phase after cooling down from high sintering temperatures, which resulted in an increase of around 3 to 5 percent in grain volume, causing surface roughening, micro cracking and mechanical retrenchment.^{208,41}

However, the change in the dimension of grains creates compressive pressure that inhibits crack growth by a phenomena called toughening transformation.⁵⁷ Also, the addition of metal oxides and crystalline structure playing a meaningful role in enhancing the mechanical characteristic especially the flexural strength.⁶⁶ While, the monolithic zirconia used in the present study sintered by conventional sintering process the polycrystalline zirconia structure becomes more compact at temperature between 1350°C and 1550°C and the porosity decreased, this explained the high value for flexural strength and surface microhardness test results for the tested monolithic zirconia specimens.

Consistent with the present study there are several studies that have shown high fracture resistance of monolithic restorations produced from lithium disilicate and leucite-reinforced glass-ceramics. Nakamura et al.²⁰⁹, as a result of his in-vitro study, demonstrated that the crowns fabricated from monolithic zirconia showed higher fracture resistance than monolithic lithium disilicate crowns.

Johansson et al.²¹⁰ evaluated the difference between fracture resistance of monolithic zirconia and lithium disilicate at the same thickness (1.8 mm) after thermo-

cycling and cyclic loading. They disclosed that the zirconia crowns recorded higher strength compared to lithium disilicate.

Sonmez et al.¹⁸⁴ examined five groups of CAD/CAM materials including IPS Empress CAD and proved that thermocycling aging process appears to affect composite polymers materials ceramic more than glass ceramic materials.

Many earlier studies in accordance with our study proved that zirconia ceramic materials had better mechanical properties than lithium disilicate and lithium silicate, particularly flexural strength and fracture strength, while there were a lower optical characteristics than lithium disilicate and lithium silicate for zirconia.^{211,85,95}

Guazzato et al.¹⁹³ investigated nine all-ceramic materials for the strength, fracture toughness, and microstructure and proved that zirconia ceramics has a fracture toughness and flexural strength at least three times than that of adhesive glass-ceramic materials and the zirconia materials contain 3Y-TZP (3 mol% yttria-stabilized tetragonal zirconia polycrystals) provided unmatched mechanical properties.

Surface microhardness is defined as the resistance to permanent deformation of the material. This feature can influence the material's wear resistance. So, the restorative materials which has increased surface hardness values can withstand the uneventful wear of antagonist natural dentation, however, restorative materials with less hardness might be exposed to unrestrained wear from the opposing natural teeth, firm particles of food or aggressive action of tooth brushing.²¹²

The surface microhardness tests such as Vickers indentation test is an important indicator for dental prostheses. The different brands of zirconia-based materials have different hardness values according to their composition, additive oxides colorant,²¹³ microstructure and also sintering procedure.²¹⁴

In the present study regarding the surface microhardness test, there was a statistically significant difference between the material type and thermal aging for glass

ceramics groups. In the thermocycling subgroups, the lowest surface microhardness value recorded by Celtra DUO was 600.75 VHN and 711.14 VHN for non-thermocycled subgroup. IPS Empress CAD recorded 675.9 VHN for thermocycled subgroup and 549.03 VHN for non-thermocycled subgroup. That means the non-thermal treated Celtra DUO specimens higher than IPS Empress CAD and the thermal treated specimens of IPS Empress CAD higher than Celtra DUO. The Celtra DUO and IPS Empress CAD were statistically significant affected by thermocycling process, while no statistically significant difference among the two types of monolithic zirconia materials (in inCoris TZI C, $p=0.176$; and in IPS e.max ZirCAD, $p=0.733$). The thermal aging subgroups showed that IPS Empress CAD group had a statistically significantly higher surface microhardness than non-thermal aged subgroups by 126.9 VHN ($p<0.05$). This result attributed to the polycrystalline structure of leucite reinforced ceramic material with 40% leucite which becomes more homogenous and compact after thermal treatment and interrupted the crack propagation as mentioned under the electron microscope (Figure 4.7 C,D).

In this study IPS Empress CAD did not obtain higher result than zirconia reinforced Celtra DUO and this finding is agreed with a previous study by Cesar et al.²¹⁵ who released that leucite contents can show improvement in flexural strength of the material, but could not increase the resistance of crack growth.

Guazzato et al.¹⁸⁷ investigated the effect of sandblasting, grinding, polishing and heat treatment on the yttria stabilized tetragonal zirconia polycrystals ceramic (Y-TPZ) and concluded that fine polishing can remove the compressive stress layer and thus reduce the mean flexing force. The polishing of unfired Celtra DUO following machine cutting of the specimens may help to improve its strength and remove deformed areas surrounding surface defects.⁵²

Vasylykiv et al.⁹⁶ who proved that the hardness increases with density, which already become higher after sintering of zirconia and the addition of 0.2-0.35 % alumina to the dense yttria-stabilized tetragonal zirconia increased their hardness. however, the tetragonal zirconia ceramic contain 0.7 mass% Al_2O_3 with a 1.5-3 mol% Y_2O_3 stabilizer

content showed a significantly average hardness property values in comparison with the samples with the higher yttria-stabilizer contents, 0.2-0.35 % alumina doped zirconia ceramics and alumina-free zirconia. This study supported our result where the monolithic zirconia in present study have <1.0% for IPS e.max ZirCAD and 0.35% for inCoris TZI C alumina oxides, this enhance their mechanical properties especially surface hardness and flexural strength.

Tetragonal zirconia when subjected to indentions fissure initiated, the material undergo inner volumetric transformation interfere with crack propagation that is the unique transformation toughening of zirconia which responsible for their distinguishing mechanical properties.¹⁹¹ These statements explain the result of present study while the monolithic zirconia shows the highest value for microhardness test even after thermocycling process.

In line with present findings, Shen et al.²⁰² has assessed the hardness of monolithic zirconia after hydrothermal aging, showing that both 3Y-TZP and 5Y-TZP restorations have no impact on the surface hardness of the complex oral conditions.

Zirconia reinforced lithium silicate glass ceramics have been launched as CAD/CAM blocks which are obtained in a recrystallized (Celtra CAD) or totally sintered stage (Celtra DUO) and indicated for inlays, crowns, onlays and veneers. Celtra DUO has similar microstructures of VITA Suprinity. While Celtra DUO has large size of the Li_2SO_3 phase than Suprinity, in Celtra DUO ($\sim 1 \mu\text{m}$ in length) while in Suprinity (up to $\sim 0.5 \mu\text{m}$).²¹⁶ ZrO_2 -containing glass-ceramic lithium silicate materials uses diphosphorus pentoxide (P_2O_5) as a nuclear agent for metasilicate crystallization, these materials contain zirconia particles about 10% by weight scattered in a fine glassy matrix of about 500-800 nm. The Celtra DUO blocks are a type of that materials which available in fully sintered or recrystallized form and indicated for crowns, onlay and inlays due to remarkable improved optical properties.²¹⁶

According to manufacturer states, the Celtra DUO blocks has a flexural strength average 360 MPa and in many recent studies the Celtra DUO presented intermediate strength compared with zirconia ceramics and silicate ceramics at the same time the

addition of ZrO_2 to lithium silicate increase the strength of the material at the comparable level with lithium disilicate.⁶⁹

The Celtra DUO manufacturer declared that crystallization is not requisite, and the material can be polished after grinding, and adhesively cemented, making it particularly appropriate for the chairside process, also they mentioned that sintering may help increasing of its flexural strength^{188,54} in the present study Celtra DUO used without firing and the samples have polished by silicon papers to keep one smooth polished surface.

Lawson et al.⁵² assessed the mechanical characteristics of several CAD/CAM materials, demonstrated that the increase in strength seen with the fired Celtra DUO did not result from further crystallization because there was no change of crystalline micro structure observed in the SEM of the Celtra DUO. The glassy dental ceramics has a potential for healing cracks at high temperature by flowing the viscous glass around the crack edges lead to impeding of crack propagation.²¹⁷ In this way, sintered Celtra DUO improved its strength. In the present study we used it without sintering, this explained why the hardness for the Celtra DUO decreased after thermocycling and effected by recurrently cycling of cold and hot water.

Moreover, the polishing of the specimens played role for improving the strength of glass-ceramic materials and can remove destruction zone after surface damage by cutting.²¹⁸ Therefore, surface polishing of unfired Celtra DUO after machine cutting may help to enhance its strength without thermocycling.

The index of brittleness which determined by means of Vickers surface microhardness diagonal indent has direct relation to the degree of marginal chipping.²¹⁹ The results of this study have shown cracks emerging from the Vicker's diagonal indents made in unfired Celtra DUO, indicating that these materials are more likely to chip at thin margins.⁵²

Most likely, the variance values among non-thermal aged IPS Empress CAD and Celtra DUO are different because of their composition difference. Celtra DUO contains highly spread (10 percent of its weight). Zirconia submicron grain size (ZrO_2) that is dissolved in a viscous vitreous matrix, which is responsible for the nuclei formation of crystal phase provides high strength zirconia-reinforced ceramics improved load resistance in comparison with the leucite reinforced ceramics.^{220,221} In current study, Celtra DUO showed high hardness than IPS Empress CAD where SEM image of the zirconia-reinforced lithium silicate gave an explanation by displaying an uniform and densely arranged bean like crystals (Figure 4.6).

According to the study of Ramos et al.²²², the lithium disilicate and lithium silicate exhibited equivalent values for fracture toughness. So, the zirconia contents in the zirconia reinforced lithium silicate have not increased the toughness of the fractures or slowed down the propagation of crack. The current results did not agree with that, because Celtra DUO with zirconia reinforcement record high value of surface hardness than IPS Empress for non-thermal aged sub group.

Although Ramos et al.²²² shows the microstructure similarity of lithium disilicate in comparison with zirconia reinforced lithium silicate, while the lithium silicate crystals cannot be detected without etching in zirconia reinforced lithium silicate. And it was observed clearly after hydrofluoric acid-etching where it appears as larger and rounded in shape compared with lithium disilicate crystal. While some studies used etching and others did not.^{173,223} In the present study, those features did not seen in non-etched samples by SEM images, and clearly appearance of the direction of polishing strokes have been seen. Since, our study relies on a comparison of the material properties in the clinical situation without any chemical effect to their contents, as the etching procedure can lead to the dissolvent of all glassy matrix from the materials and this may affect our intended results from this research.

In addition, Yu Zhang et al.²²⁴ in their in-vitro study evaluated monolithic ceramic material for their mechanical properties with more interest to zirconia ceramic and lithium disilicate glass-ceramic, demonstrated that glass-ceramic cannot match

zirconia ceramic for mechanical properties and even highly translucent zirconia cannot reach the color matching of monolithic glass ceramics which have superior fracture resistance to the porcelain veneered coping zirconia for chipping and crack propagation mode.

A clinical study by Belli et al.²²⁵ evaluated the fracture rate of zirconia restorations including many types of zirconium oxide materials. The fabricated crowns of IPS e.max ZirCAD MT in the study resist about (150N/170 N) which is similar or higher than the chewing load of natural teeth (100 and 150 N), and the zirconium oxide materials reported no clinical fracture for IPS e.max ZirCAD bridges.²²⁵ These results are agreed with present results in the current in-vitro study, given that we simulate the circumstance of the clinical study by the thermo-cycling process where the IPS e.max ZirCAD recorded a high value of surface microhardness and for flexural strength among all the tested material groups.

In addition, the flexure forces for all the different IPS e.max ZirCAD products higher than ISO 6872:2015 with a minimum of 500 MPa for dental ceramic (class 4) or 800 MPa (class 5).¹¹⁸

Holland et al.²²⁶ mentioned that the strength of glass-ceramic was accomplished by precipitating of crystals. This is can be noted in the SEM images of IPS Empress CAD samples (Figure 4.6), where the crystals appear more prominent under high magnification.

Lawson et al.⁵² spell out that the zirconia-reinforced lithium silicate materials (Celtra DUO) and lithium disilicate (IPS e.max CAD) showed high strength and hardness than polymer-infiltrated ceramic and resin nanoceramic. The result of the present study showed average hardness and strength on lithium silicate but less than monolithic zirconia materials.

Concerning mechanical and physical properties of monolithic zirconia crowns, the previous studies clarify the superiority for monolithic crowns compared to veneered framework crowns and lithium silicate ceramic crown.⁶¹ The third generation of

monolithic zirconia ultra-translucent brand has no transformation toughening when aged at the same time presented significantly inferior mechanical properties.⁶¹

However, in many in vitro studies the mechanical stability of monolithic zirconia was determined for the crown and FDPs.^{64,85} In the present study, the aging of the sample by thermo-cycling did not affect their flexural strength and translucency properties, these results disagree with many previous studies that mentioned, tetragonal zirconia meta stability increasing the aging of ceramic zirconia oxide in presence of moisture by phenomena called low-temperature degradation (LTD).²²⁷ And agree with Putra et al.²⁰³ who investigated the effect of hydrothermal treatment of monolithic zirconia on the light transmission and demonstrated that a statistically significant difference of TP over aging time depended on the commercial product types.

Recently, many types of monolithic zirconia used in most posterior and full arch restorations, to avoid the delamination and chipping of veneered zirconia restorations, yet their opacity pose a big problem particularly for anterior teeth restorations, where matching the color of adjacent natural tooth is important ,and to overcome the problems of zirconia opacity and enhance it is translucency, new zirconia with reduction of grain size, porosity, and the amount of adding alumina has been produced.⁹⁵ In recent translucent monolithic zirconia brands, there is noted obvious drop in the mechanical properties particularly flexural strength but still higher than glass ceramic LDS restorative materials.²²⁸

Regarding the evaluation of optical properties for zirconia and zirconia reinforced glassy ceramic materials ,in the present study we evaluate monolithic tested materials by using a spectrophotometer device, to measure the color difference (ΔE), translucency parameter (TP) and opalescence parameter (OP) values according to the CIE $L^*a^*b^*$ color scale.

The two types of monolithic yttria tetragonal zirconia materials tested in this study showed lower optical features than Celtra DUO and IPS Empress CAD and this difference attributed to the amount of oxides content in IPS e.max ZirCAD and inCoris TZI C.²²⁹

According to earlier studies, the translucency of zirconia is related to its, additives microstructure, thickness and sintering temperature.¹⁹⁰ The conventional zirconia ceramic has an opaque appearance because of its typical pores (range 200-400 Nm) and density (0.05 percent).¹⁹¹

The study of Lawson who compare the translucency and strength of traditional and high-speed sintering CAD/CAM zirconia blocks, with lithium disilicate confirmed that optical characteristics of monolithic materials affected by the material type.¹⁹² That result is in conformity with our findings from this study where the optical properties of tested materials effect by the type and compositions of material. Nevertheless, the grain size not only the responsible factor for increase the of zirconia ceramics translucency. There is a complex process of reflection, refraction, scattering, absorption and transmission may occur when the light goes through the zirconia restorations.^{229,23}

Several studies evaluated the color of teeth and restorative materials that indicated the ΔE value should be greater than 1 ($\Delta E > 1$) to be able to detect color change clinically.²³⁰ Paravina et al.²³¹ proved that the perceptual threshold of $\Delta E = 1.2$ has recognized within 95%. In the intra-oral conditions the acceptability will be limited in the range of 3.3-3.7 ΔE CIE $L^*a^*b^*$ units.²³²

In this thesis study, the average ΔE values were evaluated according to O'Brien's clinical match tolerance threshold values (Table 2.4).

O'Brien et al.²³² concluded that the clinical perception of color is perfect if ΔE value is between 0-0.5; if ΔE value between 0.5-1, is very good; if the ΔE value is between 1-2, is good; if the ΔE value is between 2-3.5, there is a clinically acceptable color change; and if the ΔE value is greater than 3.5, the color is clinically unacceptable. In our study, the differences in the color acceptability and perceptibility of dental restorations were determined by using ΔE where $\Delta E < 3$ which is generally undetectable to the human eye.²³⁰ ΔE ranged from 0.42 to 0.54 for all the groups and there was statistically significant difference in all material tested after thermocycling except monolithic zirconia inCoris TZI C. The mean ΔE in IPS Empress CAD with thermal aging was (2.78 ± 1.21) which

is statistic significantly higher than all other subgroups ($p < 0.05$), and this might be associated with polychromatic character of the IPS Empress CAD ceramic material or due their leucite microstructure, where the effect of hydrothermal treatment on the translucency of monolithic restorations investigated only in a few studies.²³³ There is no similar study to compare.

Translucency is one of the main factors that controlled esthetic appearance and it is very critical point in the selection of dental materials. Yu et al.¹⁶⁰ declared that the TP value may vary according to the tooth measurement zone and measurement procedure used in the study. In the present study, the Celtra DUO and IPS Empress CAD recorded the highest values for TP and OP, this result was agreed with studies stated that the number of crystals within the glassy matrix controlling the degree of translucency of glass-ceramic.²³⁴ IPS Empress CAD due to it is highly content of silica (60-65 wt.%). This ceramic showed improved translucent property.⁹¹ Also, the difference between the refractive index of both the glassy matrix and crystals which represents the quantity of speed reduction of light when passing through a medium, play important role for this change, the glassy matrix has the similar refractive indices to the leucite and lithium.¹⁹¹

In the present study, the linear well-organized crystals structure with the high transmittance glass ceramic noted clearly in the SEM images of IPS Empress CAD material which played an important role in increasing the translucency of this material among all tested materials. The residual pores and impurities in the zirconia present as volumes with refractive index differences that cause optical dislocation on zirconia surface, resulting in reduced translucency.²³⁵ Yu Zhang²³ compared the pores with the size of the wavelength of visible light (400-700), they found the big size pores of zirconia has a great effect on the light scattering lead to decrease it is translucency, this can be one cause of low translucent zirconia restorative material in current study. In the material with additive agent like alumina or yttrium oxides which added to improve the stability of ceramics may increase the light scattering and reduce the translucency.²³⁶ This might be another cause explain why the monolithic zirconia in the present study recorded low translucency value than lithium silicate.

Therefore, these results are compatible with results of in vitro study by Husain et al.²³⁷ who stated that despite the translucent zirconia showed improvement in their optical properties still lower when compared with lithium disilicate materials.

Shena et al.²⁰² found that the TP of 3Y-TZP significantly reduced when the thickness of the samples more than 1.0 mm, and the 3Y-TZP zirconia presented the best translucency with 0.5 mm thickness, since the esthetic improvements are going to be considerably small for samples with 1.0 mm of thickness. In our study the sample thickness was 1.2 mm for all the types of the materials this explain the opacity of monolithic zirconia samples.

In agreement with the present study, Subaşı et al.²³⁸ stated that the monolithic zirconia showed smallest color change among studied groups and its color change was not affected by the thickness after subjected the specimens to coffee thermocycling. Also, he demonstrated that the translucency ascending from lithium disilicate, reinforced lithium silicate and lastly preshaded monolithic zirconia. The study done by Baldissara et al.²³⁹ confirmed that the lithium disilicate glass ceramic manifested high translucency than zirconia based core materials. And that proved by result of current study.

In accordance with the present study, Church et al.²²⁸ evaluate the translucent property and strength of highly translucent four monolithic zirconium materials including inCoris TZI C and compared these materials with high translucent (HT) lithium disilicate material (IPS e.max CAD HT) and the results of their study revealed that the translucent zirconia materials showed flexural strength significantly higher than lithium disilicate materials while zirconium oxide materials have lower translucent than lithium disilicate at the same thicknesses.

The inCoris TZI C (Dentsply Sirona) contain 3 mol% by weight polycrystal of yttria partially stabilized tetragonal zirconia (3Y-TZP) with less concentration of alumina oxide and limiting porosity by a higher temperature sintering to improve its translucency.⁶⁴

While inCoris TZI C, according to manufacturer considered as one of the highly translucent CAD/CAM monolithic zirconia product for crowns and FDPs.⁶⁴ In the results of the present study when compared inCoris TZI C with the new zirconia reinforced ceramic Celtra DUO, showed low translucency parameter and has the approaching value of TP and OP parameter with IPS e max ZirCAD. This degree of opacity regarding the inherent double refraction of non-cubic zirconia phases of inCoris TZI C which resulting in light scattering from pores and grain boundaries. The 3Y-TZPs zirconia contains about 0.25% alumina oxide and showed strengths exceeded 1GPa in flexure.⁶⁴

In order to produce stable monolithic zirconia include better transparent phase increased amount of yttria 4 mol% (4Y-PSZ) and or 5 mol% (5Y-PSZ) with cubic zirconia has been used which remarkable increased the translucency but lead to deficiency in the toughness and strength properties²⁴¹, while in this present study the IPS e.max ZirCAD Y-PSZ (> 25%) recorded comparable value to inCoris TZI C in optical and mechanical properties where there are no cubic phase in their structures so, recorded high value for flexural strength and microhardness tests and low TP, OP value compared to other tested materials.

Leucite porcelain materials are among the most esthetic dental ceramics and still widely used clinically for inlays, onlays, crowns, partial crowns and ceramics veneers fabrications.²²⁶

IPS Empress CAD has been evaluated in the current study and showed high value for optical properties when comparing with zirconia reinforced glass-ceramic and monolithic zirconia ceramics. As mention in it is manufacture instruction it containing about 40% leucite crystals embedded in a feldspathic glass-ceramic considered a monolithic successor of CAD-CAM restorative material, and the crystalized surface of a monolithic glassy matrix having CeO₂ and TiO₂ nucleating agent which gave moderate strength. IPS Empress CAD displays a flexural strength of 185 MPa depending on the manufacture information.²²⁶

Kelly et al.²⁴² in their review stated that zirconia with its high crystalline properties has less translucent than lithium disilicate materials. While the degree of opacity of zirconia act as advantage for masking underlying discolored tooth structure or dark post restorations.²⁴³

Vichi et al.¹²² in their in-vitro study compared groups of fifteen low translucency ceramic materials include materials for sintered IPS e.max ZirCAD, Sirona InCoris ZI, and lithium disilicate, and glass-infiltrated in-ceramic alumina. All materials groups were evaluated for translucency and found that the contrast value at 1.0 mm started from 0.89 for Sirona InCorisZI to 0.78 for IPS e.max ZirCAD and these materials can be used for the posterior restorations, based on it is high strength values. the previous study examined different levels of light transmission, that were lower significantly than that of the lithium disilicate materials, which used as a control group, and this result approved by current study where the monolithic zirconia InCorisTZI C and IPS e.max ZirCAD showed lower translucency compared with lithium silicate ceramic materials used in the study.

Sravanthi et al.²⁴⁴ evaluated and compared the translucent nature of three manufactured all-ceramic materials of alumina, zirconia and lithium disilicate, which had been commercially available, showed significant translucent differentiation between the three, with decreasing lithium disilicate, alumina, and zirconia ranges. The authors of the study suggested lithium disilicate crowns for matching highly translucent natural teeth, alumina crowns for average translucent teeth, and zirconia for opaque teeth.

Results from another study by Ya-Ming Chena et al.²⁴⁵ confirmed our findings, who analyzed the relative translucence and bending strengths of four different ceramic dental core materials and stated that the IPS Empress and In-ceram Alumina were more translucent than zirconia based material.

Regarding the electron microscopic evaluation (SEM) of the specimens, however Surface defects, which may potentially become a larger crack, cannot affect the marginal integrity of restorative materials, they are considered to weaken the restorations.²⁴⁶

Another study by Wendler et al.¹⁸⁸ explained the presence of numerous pitting and cracking in specimen's surface of ceramic materials showed by scanning electron microscope images are related to thermal incompatibility between phases of zirconia and localized residual stresses that are mitigated during cooling after sintering of the material. This finding is in accordance with present result of SEM images for monolithic zirconia groups in the current study.

The other damages that cleared in the surface image of the specimens might be caused by diamond coated cutting instrument upon cutting of the specimens even when we used highly polished specimens.²⁴⁷ In this present study, the commercially available CAD/CAM blocks for inCoris TZI C, and IPS e.max ZirCAD was sintered in a furnace according to their manufactured instructions before cutting with a diamond blade under constant water irrigation, this procedure may lead to creation of some scratching and surface damaging as noted in SEM images.

Kaizer et al.¹⁷³ investigated the translucency, microstructure, and wear resistance for monolithic translucent zirconia-based ceramic crowns (inCoris TZI), and proved that the sintering temperature affects the size and density of the grains of ceramic materials, and this influence the optical properties and hardness of ceramic.⁶⁵ This compatible with the current study results, where the translucency measured on flat surface of the standardized thickness of monolithic zirconia ceramic restorative materials after sintering in porcelain furnace.

Completely crystalline 3Y-TZP ceramics (IPS e.max ZirCAD LT) by sintering, it consists of a metastatic tetragonal phase of about 98%. While the grain size controlling the transformation tendency, the recent 3Y-TZP ceramics are made of uniform and homogeneous grains. The proper temperature during sintering generates a typical grain size of 0.5 μ m, which enables t-to-m transformation at crack margins, lead to high toughness, also caused 3Y-TZP materials more opaque than other groups of lithium silicate ceramic materials which not subjected to sintering in the same study.¹¹⁸ This is explain the homogeneous shape of monolithic specimens in the current study SEM image (Figure 4.6, 4.8).

The new translucent zirconia ceramics recently introduced contain cubic zirconia and tetragonal phase in order to enhance the optical properties are significantly weaker and cannot be appraised as a complete alternative to Y-TZP ceramics with their excellent mechanical properties.¹⁴⁷

Regarding the thermal stability of zirconia-based ceramic restorative materials, studies showed that well-controlled zirconia ceramics have enough long term stability in the oral environment even after noted tenuous transformational durability.²⁰⁶

Studies on lithium disilicate have disclosed controversial results on the formation and persistence of thermostable phase during the early crystalline phase, showed that metastable phase has no significant effect on the thermodynamical stability of the material.²⁴⁸ All these studies support the present study where the monolithic zirconia groups did not effect by the thermocycling procedure.

Contrary to the result of current study, Porojan et al.²⁴⁹ concluded that thermal cycling significantly increases translucency of ZLS and lithium disilicate glass-ceramic material, with the most damaging impact on samples pressed and polished. Micro and nano roughness in vitro ageing were noted with a negative correlation. Glazed samples for all types of materials are characterized by significant reddened surfaces. On the nano level, thermal cycling substantially smoothed ZLS material also the thermocycling smoothed the surfaces in SEM and topography, but the difference in microstructure results in a differently dissolved environment with consequences for micro roughness. This difference attributed to that they used glazed sample with 1.5 mm thickness while in our study we used 1.2 mm thick samples without glazing with short duration thermocycling procedure.

The opalescence was the anther optical properties which tested in this thesis study, the scattering of the short wavelength of the visible light in the reflected color gave an object a bluish manifestation and orang brown coloration during color transitions and this is called the opalescence of the object.²³¹

Dental enamel is considered an opalescence material and gives the reflected color a bluish appearance and the transmitted color an orange-brown appearance when subjected to the light scattering of the short wavelength of visible light. As determined by Yong-Keun Lee²⁵⁰ in a previous study that OP value for bovine tooth enamel was 7.6–22.7 and for human natural enamel was 19.8–27 and the perfect and most esthetic restorative materials should have the same opalescence of the natural tooth, and should have a good masking effect of underline tooth structure.

The OP values in the current study were obtained using a dental spectrophotometer. OP used to evaluate and compared the opalescent value for all tested ceramic restorative material where the highest OP value for Celtra DUO (2.97 ± 0.34) followed by IPS Empress CAD (10.31 ± 133) and the lowest OP value found for monolithic zirconia groups from inCoris (0.58 ± 0.36) followed by IPS e.max ZirCAD (0.79 ± 0.61). These results showed that OP for our tested material was lower than the criteria for normal enamel. Take in consideration, that we used LT blocks for our study.

In agreement with our study, Sang Cho et al.²⁵¹ found all the OP values resulted from their study were low than the criteria of previous studies where the mean value of bovine enamel OP was from 10.6 ± 1.4 to 19.0 ± 2.1 , and the mean value of OP human natural enamel was 22.9 ± 1.9 .

In disagreement with Porojan et al.²⁴⁹ who examined OP values, found there is significant differences between all groups. ZLS samples with values higher than lithium disilicate and decrease in opalescence following thermal cycling for ZLS, and positive correlation was found between TP and OP.

These differences regarding the measurement protocols like thickness and size of samples and the proportional aperture sizes for spectrophotometer should affect the OP value, where the thickness of bovine enamel specimens in the previous studies, about from 0.7 to 1.1 mm.²⁵⁰ and in our study 1.2 mm.

The improved opalescence properties of dental ceramic materials are combined with the natural looks of materials and it is reaction to the light as natural tooth material.¹⁷⁶ Also the micro particles glass in some opalescent ceramics which scatter the incident light can improve the esthetic properties like translucency and brightness.²⁵²

There is a clear correlation between the OP and TP values in the current study, this compatible with the previous study by Yong-Keun Lee et al.²⁵³ that found there is a correlations among OP, ΔE , Δb^* and Δa^* for transmittance and reflectance measurements were recorded for composites specimens. While the opalescence is correlated to translucency that means the low translucency of monolithic restorations may have influenced their opalescence in the current study.

As many previous studies proved that the property of masking dark background was affected by translucency and opalescence of restorations when translucency was in the similar values.²⁵³⁻²⁵⁴ The highly opalescent materials can be used as veneer restoration for discolored teeth which allowing for full masking without removing excess tooth structure.²⁵⁵

Considering all the results of this limited current study,

1. First null hypothesis as ‘there will be no difference among four different monolithic CAD/CAM materials’ mechanical properties (flexural strength and surface microhardness) with and without thermocycling’ was partially rejected. There was no difference in terms of mechanical properties between monolithic zirconia materials.
2. Second null hypothesis as ‘there will be no difference among four different monolithic CAD/CAM materials’ optical properties (color difference, translucency and opalescence) before and after thermocycling’ was partially rejected. There was difference among some materials in terms of optical properties before and after the thermocycling.
3. Third null hypothesis as ‘there is no effect of thermocycling on the mechanical properties (flexural strength and surface microhardness) and optical properties (translucency and opalescence) for tested monolithic CAD/CAM restorative

materials' was partially rejected. Thermocycling affects the surface microhardness and color difference of all tested materials.

All the tested restorative ceramic materials in the current study are indicated for veneers, inlay, onlay and crowns. The higher crystalline properties of zirconia-based ceramics make it less translucent when compared to glass matrix ceramic restorative materials.



6. CONCLUSION

The conclusion of the current in vitro study are as following:

1. The four studied monolithic restorative ceramic materials have different compositions that affect their mechanical and optical properties.
2. The monolithic zirconia restorative materials (IPS e.max ZirCAD and inCoris TZI C) have higher mechanical properties than glass ceramic materials.
3. The flexural strength of any tested material was not affected by thermocycling procedure simulating one year.
4. The surface microhardness of both reinforced monolithic glass ceramic materials (IPS Empress CAD and Celtra DUO) were affected by thermocycling simulating one year, while the surface microhardness of monolithic zirconia materials (IPS e.max ZirCAD and inCoris TZI C) were not affected.
5. The reinforced monolithic glass ceramic materials have superior optical properties than monolithic zirconia ceramics.
6. The color change (ΔE) was increased for all tested materials after thermocycling.
7. The two different types of reinforced glass-ceramic restorative materials (IPS Empress CAD and Celtra DUO) showed similar TP and OP values.
8. The two different types of monolithic zirconia ceramic restorative materials (IPS e.max ZirCAD and inCoris TZI C) showed similar mechanical properties, TP and OP values.
9. The TP value for reinforced glass ceramic restorative materials (IPS Empress CAD and Celtra DUO) are within the range of natural teeth TP values.
10. The OP value for all tested groups showed lower values than natural tooth OP values.

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