FRACTURE TOUGHNESS AND FLEXURAL STRENGTH OF THREE VENEERING PORCELAINS

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ABSTRACT

The purposes of this study were to determine the fracture toughness and flexural strength values of three veneering porcelains.

Materials used in this study were Vita VMK 95, which is veneering porcelain for metal-ceramic restoration, IPS Eris, which is veneering porcelain for Lithia-disilicate-based core ceramic, and Cercon Ceram Kiss, that was veneering porcelain for zirconia-based prosthesis. Specimen preparation and test methods were performed according to ISO 6872-Dentistry- ceramic materials. Flexural strength was determined using ten bar-shaped specimens for each material. Specimens were loaded using four-point bending fixture until fracture occurred. For fracture toughness testing, five bar-shaped specimens were prepared for each material. The single-edge V-notched-beam technique on four-point bending fixture was used with a span length of 20 mm. All specimens were loaded to fracture using a universal testing machine at cross-head speed of 0.5 mm/min. Load at fracture was used to calculate flexural strength and fracture toughness values. Statistical analyses of means flexural strength and fracture toughness were performed using one-way ANOVA and Turkey's multiple comparison tests.

The results showed that fracture toughness values of the three porcelains ranged between 0.89 to 1.04 Mpa•√m. Mean fracture toughness of Vita VMK 95 was significantly higher than that of IPS Eris ($p<0.05$). Means flexural strength of three the porcelains ranged between 39.2 to 80.1 Mpa. No significant differences were found between flexural strength of these three porcelains.

In conclusion, the fracture toughness value of Vita VMK 95 was significantly higher than that of IPS Eris Veneer ($p<0.05$). No significant differences were found between flexural strength of the three porcelains.

KEY WORDS: VENEERING CERAMICS/ FRACTURE TOUGHNESS/ FLEXURAL STRENGTH/ SEVNB

39 pp.

ค่าความทนแรงดัดและค่าความเหนียวต้านการแตกหักของพอร์ซเลนที่ใช้ในทางทันตกรรม จำนวน 3 ชนิด

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บทคัดย่อ

การศึกษานี้มีวัตถุประสงค์เพื่อศึกษาค่าความเหนียวต้านการแตกหักและค่าความทนแรงดัดของพอร์ซเลนที่ใช้ในทางทันตกรรม จำนวน 3 ชนิดคือ Vita VMK 95 ซึ่งเป็นพอร์ซเลนที่เคลือบอยู่บนโครงโลหะเจือ IPS Eris เป็นพอร์ซเลนที่เคลือบอยู่บนโครงที่เป็นลิเธียไดซิลิเกต และ Cercon Ceram KISS เป็นพอร์ซเลนที่เคลือบอยู่บนโครงที่เป็นเซอร์โคเนียมไดออกไซด์หรือเซอร์โคเนีย

การเตรียมชิ้นตัวอย่างและวิธีการทดสอบจะอ้างอิงตามวิธี ISO 6872: Dentistry-ceramic materials หาค่าความทนแรงดัดโดยการใช้ชิ้นตัวอย่างรูปแท่งจำนวน 10 ชิ้นต่อชนิดของผงพอร์ซเลน ชิ้นตัวอย่างจะถูกทดสอบการดัดสี่จุด จนกระทั่งชิ้นตัวอย่างเกิดการแตกหัก สำหรับการหาค่าความเหนียวต้านการแตกหัก ชิ้นตัวอย่างรูปแท่งจำนวน 5 ชิ้นต่อชนิดของผงพอร์ซเลน จะถูกบากเป็นรูปตัววี ก่อนที่จะนำไปถูกทดสอบการดัดสี่จุด โดยจุดรองรับคานด้านนอกที่ใช้มีระยะห่าง 20 มิลลิเมตร ทุกชิ้นตัวอย่างจะถูกทดสอบด้วยเครื่องทดสอบที่ความเร็วในการออกแรงกด 0.5 มิลลิเมตรต่อนาที จนกระทั่งชิ้นตัวอย่างแตกหัก แรงกดที่ทำให้เกิดการแตกหักจะถูกนำมาคำนวณหาค่าความเหนียวต้านการแตกหักและค่าความทนแรงดัด การวิเคราะห์ทางสถิติของค่าเฉลี่ยความเหนียวต้านการแตกหักและความทนแรงดัดจะถูกแสดงโดยการทดสอบ one-way ANOVA และ Turkey's multiple comparison

จากผลการทดสอบพบว่าค่าเฉลี่ยความเหนียวต้านการแตกหักของผงพอร์ซเลนทั้งสามชนิดมีค่าอยู่ระหว่าง 0.89 ถึง 1.04 เมกะปาสคาล• ม$^{1/2}$ ค่าเฉลี่ยความเหนียวต้านการแตกหักของผงพอร์ซเลนของ Vita VMK 95 นั้นสูงกว่าของ IPS Eris อย่างมีนัยสำคัญ($p<0.05$). ส่วนค่าเฉลี่ยความทนแรงดัดของผงพอร์ซเลนทั้งสามชนิดมีค่าอยู่ระหว่าง 39.2 ถึง 80.1 เมะปาสคาล และไม่พบความแตกต่างกันอย่างมีนัยสำคัญของผงพอร์ซเลนทั้งสามชนิด

จากการศึกษานี้สามารถสรุปได้ว่า ค่าเฉลี่ยความเหนียวต้านการแตกหักของผงพอร์ซเลนของ Vita VMK 95 นั้นสูงกว่าของ IPS Eris Veneer อย่างมีนัยสำคัญ ($p<0.05$) และไม่พบความแตกต่างกันอย่างมีนัยสำคัญของค่าความทนแรงดัดระหว่างผงพอร์ซเลนทั้งสามชนิด

39 หน้า.

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LIST OF ABBREVIATIONS

µm.	micrometer
mm.	millimeter
cm.	centimeter
mg.	milligram
g.	gram
ml.	milliliter
N	newton
kN	kiloNewton
SEVNB	single edge v-notch beam
MPa	megaPascal
Ltd.	Limited
Co.	Company
et al.	et alii
cont.	continue
Fig.	Figure
^{o}C degree	Celsius
SD	standard deviation

CHAPTER I

INTRODUCTION

Ceramics has been used in tooth restoration for more than two centuries. Its color and translucency cannot as yet be matched by any material used for fixed partial denture, except other ceramics. For porcelain fused to metal restoration (PFM), it composes of metal as a core and dental porcelain that laminates on metal substructure. For all-ceramic restoration, ceramic core is fabricated instead of metal core. Veneering porcelain is a translucent material that is applied on metal or ceramic substructure to give a natural-look appearance to a prosthesis.

Regarding the application of materials, dental ceramics can be divided into two groups, veneering ceramic or dental porcelain and ceramic core materials. Dental porcelain is an esthetic part of fixed dental prosthesis. Because of its translucent and color, matching between natural tooth and dental restoration can be made. However, its low fracture resistance is the major disadvantage [1-3]. Chipping of veneering porcelain has been reported as a cause of failure for PFM restorations [4]. Modeling method of porcelain is mostly done by mixing porcelain powder with water or modeling fluid recommended by manufacturer. The slurry is applied and shaped in required form on a substructure. Then it has to be sintered in the oven according manufacturer's instructions.

Ceramic cores have fracture toughness values ranged between 1.2 to 7 MPa$\sqrt{m}$ [5-9] which are higher than those of dental porcelains. However the fracture toughness value of ceramic core is still greatly lower than metal. This can be the reason for lower success rate for all-ceramic prosthesis comparing to the success rate of porcelain fused to metal [10-11]. Nowadays, the compositions of ceramic core have been improved into several chemical formulas such as lithia-disilicate-based ceramic,

alumina-reinforced ceramic, and zirconium dioxide [12-16]. These ceramic core materials have high fracture toughness values but they are more opaque. Several processing techniques are also used for fabricating the core structure such as lost-wax, heat-pressing, CAD-CAM , and slip-cast techniques [1,2,12,13]. According to the results from previous studies, either tests in laboratory or clinical trials, they found that fracture started from the area of connector for the failure of fixed partial denture which could be in the veneering layer [17, 18]. Even strong core material is used to support veneering porcelain, but failure of a dental prosthesis could originate from a weaker layer. In order to obtain data about failure pattern of ceramic materials, information about compositions and properties of porcelain is required.

The objectives of this study were to compare the flexural strength, fracture toughness of three veneering porcelains, Vita VMK 95 for metal-ceramic restoration, IPS Eris Veneer for Empress 2 all-ceramic system and Cercon Ceram Kiss for zirconia-based prosthesis.

CHAPTER II

LITERATURE REVIEW

Dental ceramics are nonmetallic, inorganic materials, primarily containing compounds of oxygen with one or more metallic or semi-metallic elements (aluminum, calcium, lithium, magnesium, phosphorous, potassium, silicone, sodium, titanium, and zirconium). They exhibit chemical, mechanical, physical, and thermal properties that distinguish them from other materials such as metal and polymers. Ceramics also remain stable over period of time that is suitable for use in biological environment. The properties of ceramics are customized for dental applications by precise control of the type and amount of the components used in their production. Dental ceramics exhibit fair to excellent flexural strength and fracture toughness. Although ceramics are strong but these materials are also brittle and may fracture when they are bended.

The main objective of restorative dentistry is to replace teeth lost either from periodontal disease or tooth decay. The replacement is performed to regain both function and esthetics. Regarding the application of materials, dental ceramics can be divided into two groups, veneering ceramic or dental porcelain and ceramic core materials. Dental porcelain is an esthetic part of fixed dental prosthesis. Because of its translucent and color, matching between natural tooth and dental restoration can be made. However, low fracture resistance is its major disadvantage. [1-3] Fracture toughness values of dental porcelains are usually less than 1 MPa$\sqrt{m}$ [1, 4]. Modeling method of porcelain is mostly done by mixing porcelain powder with water or modeling fluid recommended by manufacturer. The slurry is applied and shaped in required form on a substructure. The sintering process is performed in the oven according manufacturer's instructions.

Ceramic cores have fracture toughness values ranged between 1.2 to 7 MPa√m [5-9] which are higher than those of dental porcelains. Improvements in both the composition and method of forming the core of all-ceramic crown have greatly enhanced the ability to produce more accurate and more fracture resistance all-ceramic restoration [15, 16]. Significant progress has also been made for developing less abrasive veneering ceramics [19]. They are commonly referred to as low-fusing ceramics that have been introduced as veneering glasses. Some of these veneering ceramics are claimed to be kinder to opposing tooth enamel either because they are predominantly glass phase material or they contain very small crystal particles [19].

Composition of dental porcelains

A composition of dental porcelain generally corresponds to that of the glasses, except for an increase in alkali content. The addition of greater quantities of soda, potash, and/or leucite is necessary for increasing the thermal expansion of dental porcelain to a level compatible with the metal coping. Conventional dental porcelain is a vitreous ceramic based on a silica (SiO_2) network and potash feldspar ($K_2O.AI_2O_3.6SiO_2$) or soda feldspar ($Na_2O.Al_2O_3.6SiO_2$) or both. The feldspars used in dental porcelains are relatively pure and colorless. Thus, pigments must be added to produce the color appearance of restorative materials that match the adjacent teeth.

Silica (SiO_2) can exist in four different forms: crystalline quartz, crystalline cristobalite, crystalline tridymite, and noncrystalline fused silica. Fused silica is a material with high-melting temperature. Its three-dimensional network of covalent bonds between silica tetrahedral is a basic structural units of a glass network. Fluxes (low-fusing glasses) are often included to reduce the temperature required to sinter the porcelain powder particles together. Low temperature sintering is required, so that an alloy which it is fired does not melt or sustain sag (flexural creep) deformation.

Glass Modifiers

The melting temperature of silica crystal is too high for using as veneering material. Thermal expansion coefficient of silica crystal is also too low for using with dental alloys [20]. Bonds between the silica tetrahedral can be broken by the addition of alkali metal ions such as sodium, potassium, and calcium. These ions are associated

with the oxygen atoms at the corners of the tetrahedral and interrupt the oxygen-silicon bonds. As a result, the three-dimensional silica network contains many linear chains of silica tetrahedral that are able to move more easily at lower temperatures than the atoms that are locked into the three-dimensional structure of silica tetrahedral. This ease of movement is responsible for the increase in fluidity (decreased viscosity), lower softening temperature, and increase in thermal expansion coefficient. Too high a modifier concentration, however, reduces the chemical durability (resistance to attack by water, acids and, alkalis) of the glass. In addition, if too many tetrahedral are disrupted, the glass may crystallize (devitrify) during porcelain firing operations. Hence, a balance between a suitable melting range and good chemical durability must be maintained.

Feldspathic Porcelains

Potassium and sodium feldspar are naturally occurring minerals composed primarily of potash (K_20) and soda (Na_20), respectively. They also contain alumina (Al_20_3), and silica (SiO_2) components. Feldspars are used in the preparation of many dental porcelains designed for metal-ceramic crowns [21]. When potassium feldspar is mixed with various metal oxides and fired at high temperatures, it can form leucite and a glass phase that will soften and flow slightly.

Another important property of feldspar is its tendency to form crystalline mineral leucite when melted. Leucite is a potassium-aluminum-silicate mineral with a large coefficient of thermal expansion (20 to 25 ppm/ ^oC)[1] compared with feldspar glasses (which have coefficients of thermal expansion less than 10 ppm/ ^oC). When feldspar is heated at temperatures between 1150^oC and 1530^oC, it undergoes incongruent melting to from leucite crystals in a liquid glass. Incongruent melting is the process by which one material melts to from a liquid plus a different crystalline material. This tendency of feldspar to form leucite during incongruent melting is used in the manufacture of porcelains for metal bonding.

Feldspathic porcelains contain a variety of oxide components, including SiO_2 (52-62 wt %), Al_2O_3 (11-16 wt %), K_2O (9-11 wt %), Na_2O (5-7 % wt %), and certain additives, including Li_2O and B_2O_3. These ceramics are called porcelains because they contain a glass matrix and one or more crystal phases. They cannot be classified

as glass-ceramics because crystal formation does not occur through controlled crystallization process. There are four types of veneering ceramics. These include (1) low-fusing ceramics (feldspar-based porcelain and nepheline syenite-based porcelain); (2) ultra low-fusing ceramics (porcelains and glasses); (3) stains; and (4) glazes [1]. Type and size of crystal particles, if present, can greatly influence the potential abrasiveness of the ceramic prosthesis [19].

Feldspars are used in the preparation of many dental porcelains designed for metal-ceramic crowns and many other dental glasses and ceramics. The softening of this glass phase during porcelain firing allows the porcelain powder particles to coalesce together. For dental porcelains, the process by which the particles coalesce is called liquid- phase sintering, a process controlled by diffusion between particles at a temperature sufficiently high to form a dense solid. The driving force for sintering is the decrease in energy caused by a reduction in surface area.

Ceramic core materials

There are several ceramic cores available on the market now. Their composition is varied, depending on the method of forming a ceramic core, as follows;

<u>Pressable glass-ceramics</u>

There are few ceramic systems that use this fabrication technique as follows.

- IPS Empress is a leucite-based glass-ceramic that contains about 35%(vol.) of leucite crystals.
- IPS Empress 2 core material consists of 70%(vol.) of Lithia disilicate crystals in a glass matrix and can be layered with a glass containing some dispersed apatite crystals.
- OPC is leucite-containing ceramic and OPC 3G contains Lithia disilicate crystals. The ultralow fusing temperature of the veneering porcelain suggests a low level wear rate of opposing enamel.

Glass-infiltrated ceramics (Slip-cast technique)

These systems use slurry of a core ceramic material to paint on a porous refractory die. Heating is carried out according to the manufacturer's recommendations to produce a partially sintered coping or framework. This partially sintered core is infiltrated with glass at 1100° c for 4 hrs to eliminate porosity and to strengthen the slip-cast core. The crown is then covered with veneering porcelains. Ceramic systems that use this fabrication technique are as follows

- In-Ceram Alumina core consists of 70 wt% alumina infiltrated with 30wt% sodium lanthanum glass.
- In-Ceram Spinel core consist of glass-infiltrated magnesia alumina silicate ($MgAl_2O_4$) which improve the translucency of the final restoration.
- In -Ceram Zirconia core consist of 67 wt%aluminum oxide (Al_20_3)and 33 wt% zirconium oxide (ZrO_2 , Ce-stabilized).

Because of its low glass composition, acid etching and silanization can not be used to improve the bonding with resin cements. The bonding of In -Ceram systems obtained primarily from mechanical bonding.

CAD-CAM Ceramics

Computer-aided design and manufacturer (CAD-CAM) is one of the most popular techniques. All-ceramic restoration is made by computer-aided designing and milling of ceramic ingots. There are many systems that use CAD/CAM as a method of fabrication (Table 1).

The Procera system

This system is composed of densely sintered, high purity aluminum oxide or zirconia core and compatible veneering porcelain. After the prepared tooth is scanned and the data is transmitted to the milling unit to produce an enlarged die. The slurry paste of aluminous oxide or zirconium oxide is applied on the die and the coping is milled and sintered at high temperatures. The coping is then veneered with aluminous porcelain.

The Cerec system

The Cerec system is available in the market for several years with the latest improved Cerec 3D system introduced in the 2000s. Materials that can be used with this system are Vita mark II , In-Ceram (zirconia, Alumina, Spinell), Procad and Triluxe.

LAVA system

The framework ceramic of this system consists of zirconium dioxide. The frameworks are fabricated using scanning, computer-aided framework designing and milling from presintered zirconia blanks. High strength restorations with excellent fit are produced by this system.

The Cercon system

For this system, a wax pattern of desired framework is scanned and manipulation with CAM Processing. The latest material is used in Cercon system made from zirconium oxide (ZrO_2) and yttrium oxide (Y_2O_3) about 99%.

Table 1. CAD-CAM and Copy-milled ceramics used for all-ceramic prostheses

Systems	Materials
Cercon base	Presintered ZrO_2
LAVA frame	Presintered ZrO_2
Procera AllCeram	Presintered Al_2O_3
VitaBlocs Mark II	Feldspathic porcelain
VitaBlocs Spinell	sintered $MgOAl_2O_3$
VitaBlocs Alumina	sintered Al_2O_3 followed by glass infiltration
VitaBlocs Zirconia	sintered Al_2O_3/ ZrO_2

Nowadays, all-ceramic restorations are possible substitutes for the strong but less esthetic porcelain fused to metal restoration. Combining the strength of ceramic cores and superior esthetics of a weaker veneering porcelain can result in a reliable and more biocompatible restoration [22]. The introduction of zirconium dioxide or zirconia led to the design of fixed all-ceramic partial dentures without any limitation regarding the size of the fixed partial denture [23]. Its qualities, strength, transformation toughening mechanism, white color, chemical and structural stability have made zirconia a reliable core material [24].

Various studies indicated that strength of veneer ceramic dictated the strength of layered restorations [25]. Veneering porcelain has low mechanical strength. Although they are strong under compression, ceramic materials are brittle and unable to withstand tensile stress. The strength of ceramic restorations may be further compromised by complex distribution of tensile stresses that occurred during functional loading of restoration. If tensile stresses are not seriously considered in the design of the structure, failure can occur at unexpected low stresses [26, 4].

White et al. (2005) studied about flexural strength of eight groups of layerd zirconia and feldspathic porcelain [25]. The result revealed that the moduli of rupture of feldspathic porcelain ranged between 77 to 85 MPa. Moduli of rupture of zirconia specimens, ranging between 636 to 786 MPa, were significantly higher than those of feldspathic porcelain. The elastic moduli of the porcelain and zirconia materials were 71 and 224 GPa, respectively. Crack propagation following initial cracking often involved porcelain-zirconia interface breaking, as well as bulk porcelain and zirconia fracture [25].

Interface between core and veneering materials has been reported as failure origin in failed restorations as well as in laboratory testing [27-29]. In a laboratory study, zirconia-based crowns failed basically by delamination of the veneer from intact core structure, while crowns made of layered lithia-disilicate core material failed by fracture of both the core and veneer materials [30]. Failure of brittle ceramics is related to structural flaws, which tend to concentrate stresses and can act as fracture initiation sites [31]. There are various causes and types of structural flaw which could be located at the surface, in the bulk of the material, or at core–veneer interface [32]. As dental ceramics are brittle, they have limited ability to absorb

elastic energy; thus tensile stresses and structural flaws can result in premature failure under low functional stresses [33].

Differences in thermal expansion coefficients (TEC) of the core and veneer ceramics can cause fracture of restoration due to residual stress developed in a structure [34]. Poor wetting of the core by the veneer ceramic, firing shrinkage of veneer ceramic, transformation of zirconia crystals at core–veneer interface due to thermal influences or stress loading, and inherent flaws formation during various fabrication steps are also the factors that affect failure of the restorations [35]. Various veneering ceramics are specially developed for zirconia-based core material. A special liner may be used to modify the color of the core. Omitting this liner did not weaken zirconia–veneer bond strength but it increased a chance of interfacial failure [34]. Besides veneer ceramics used for standard layering technique, new ceramic veneers can be hot-pressed onto a zirconia core. This technique is claimed to produce a veneer layer that has better properties than that made from conventional sintering technique.

The clinical failure of all-ceramic restorations is related to their brittleness and low fracture toughness. The lack of sufficient clinical studies has led manufacturers and dental operators to place great emphasis on mechanical properties to predict the clinical performance of these materials. In this regard, the most relevant mechanical properties are strength and fracture toughness. Strength values in ceramics are affected by several factors, such as a flaw distribution and the test methodology. Such variability makes comparison of materials tested under different conditions inappropriate. Fracture toughness, which is independent of flaw distribution, is considered to be a more consistent property. Wen et al. (1995) also suggested that fracture toughness is more fundamental than strength, since it characterized bulk structure without involving the flaw size [34].

From previous studies, failed clinical prostheses were investigated. Failure might be resulted from 1) the presence of critical structure flaws 2) thermal incompatibility between core and veneering materials 3) improper crown and bridge design 4) stress in layered structures and 5) non-standardized processing techniques [35]. Most of the improvements emphasized into core material but approximately 70-80% of failure originating from interface between the core and veneer ceramics [37].

Various studies indicated that the strength of veneer ceramic dictated the strength of layered core-veneered restoration [4, 25, 26]. The strength of these restorations may be further compromised by complex distribution of tensile stressed. The common clinical failure such as chipping and delamination of veneering ceramic may be resulted from low core veneered bond strength or thermal mismatch and low strength of veneering ceramic [1].

Strength is defined as maximum stress that a structure can withstand without sustaining a specific amount of plastic strain (yield strength) or stress at the point of fracture (ultimate strength)[1]. The strength is dependent on several factors including; [38]

1) strain rate
2) shape and size of the test specimen
3) surface finish (that controls the relative size and number of surface flaws)
4) test methods
5) environment in which material is tested.

However, strength of brittle materials such as ceramics may appear to be low when large flaws are present or if stress concentration area exists because of improper design of prosthetic component. Under this condition, a clinical prosthesis may fracture at a much lower applied force because the localized stress exceeds the strength of the material [1]. The mathematical formula for computing the flexural strength is as follows:

$$\sigma = \frac{3Pl}{2bd^2} \qquad (1)$$

Where

σ is the flexural strength (MPa)

l is the distance between the supports

b is the width of the specimen

d is the depth or thickness of the specimen

P is the maximum load at the point of fracture

Strength values are often relied upon as indicators of structural performance for brittle dental materials. Strength, however, varies depend upon several factors as previous mentioned. It is not an inherent material property, and strength data alone can not be effectively extrapolated to predict structural performance. Strength data is meaningful when it is considered with knowledge of material microstructure, processing history, testing methodology, testing environment and failure mechanisms [38].

Any processing step can affect the size, orientation or distribution of flaws of dental ceramics. In order to be clinically relevant for strength testing, test specimens should have the same type and distribution of flaws as the target structure service (e.g. crown, inlay or veneer [39]. Strength-controlling intrinsic defects are inevitably introduced into material during processing. Mechanically defective microstructural regions in ceramic, including area of porosity, agglomerates, inclusion (extraneous debris) and large-grained zone, can all be processing-related [40]. Machining, grinding, and air-oxide abrasion can determine the size and number of extrinsic surface flaw, yielding a range of "strengths"

Rice et al. (1981) reported that laboratory preparation of strength specimen could be uniquely different from actual restorations with respect to decisive processing variables. Test specimens are often machined or polished over large portions of the surface. The direction of surface grinding can affect strengths; bars ground parallel to their tensile axis are generally stronger than perpendicular ground bars [41].

Campbell et al. (1989) reported that removal of the outer 50-100 μm. "ceramimg skin" was shown to increase strength of glass ceramic crown (Dicor,Dentsply), due to removal of flaws in this layer of ceramic [42]. Strength is time-dependent for the many environmentally sensitive brittle materials. Strength reduction due to slow crack growth in the presence of water is well documented [43]. Such materials would have lower strength when tested under reactive environments (e.g. decrease in strength with increase in humidity). An increase in strength is also related to and increase in stress rate [42].

There are several strength test methods, however three methods are widely used as follow;

1) Three-point bending test

2) Four-point bending test

3) Biaxial bend test

The simplest possible strength techniques that can be used over a wide range of test variables are those that involve bending [44]. Three-point and four-point bend tests are frequently used because no special grips are required, and simple sample shapes (bars or rods) can be used. However, these tests have distinct disadvantages. Bending creates a stress gradient in the specimen, and only a relative small volume is exposed to high tensile stress. Also, the specimens are very sensitive to edge or surface machining damage. Thus, the test is deceptive in that it appears easy to set up and conduct, but too often the strength-controlling flaws in the bend test are not the same as for a component in service [44].

Biaxial bend test is useful since the edges of the sample (a frequent source of failure origin) are not stressed, and biaxial loading is commonly encountered in service [45]. Disc-shaped specimens are easily fabricated and by having a circular ball-bearing race support the sample, slightly warped specimens can be tested since the support balls are free to rotate. However, as with all bend tests, the maximum stress is on the surface, and volume flaws are generally strength controlling.

Fracture toughness is a property which describes the ability of material containing a crack to resist fracture [46]. Fracture toughness is a quantitative way of expressing a material's resistance to brittle fracture when a crack is present. If a material has a large value of fracture toughness, it will probably undergo ductile fracture. Standard fracture mechanics equation is as follows

$$K_{Ic} = Y\sigma c^{1/2} \qquad (2)$$

When

K_{Ic} is Fracture toughness (MPa $m^{1/2}$)

Y is Geometric constant

σ is Fractural strength

c is Critical crack size

Brittle fracture is characteristic of materials with a low fracture toughness value. Dental porcelains and ceramics are brittle materials, which generally fail in tension due to their limited ductility, which restricts the ability to absorb a great deal

of elastic strain energy before fracture [46]. A major weakness of this material is the sensitive to flaws, which may have developed as a result of thermal, chemical or mechanical process, and act as local stress raisers. At certain critical applied stress, a crack can originate from flaw and propagate, engendering final catastrophic fracture. So in the last decade in the field of dental porcelains and ceramics research, much attention has been paid to improve fracture toughness of dental materials. However, determination of K_{Ic} is technically rather sensitive and obtained values and ranking maybe different depending on the techniques and procedures used [47].

There are several fracture toughness test methods which are

- Indentation fracture IF
- Surface crack in flexure SCF
- Single edge pre-cracked beam SEPB
- Chevron notch CN
- Single-edge-V-notched-beam SEVNB

Most of studies evaluated the fracture toughness of dental porcelains by indentation fracture (IF method) [48].This method became popular in the dental field because of ease of use and small specimen sizes; however, it was not included in an ASTM standard because of inconsistent in results. Surface crack in flexure SCF method, a semi-elliptical surface crack, a sharp precrack was formed via Knoop indentation prior to the test. Single edge pre-cracked beam SEPB, a straight-through pre-crack is introduced in a beam shaped specimen via a bridge-loading technique. In both SCF, SEPB, K_{Ic} is calculated by measuring the flexural strength and the pre-cracked size. The fracture toughness evaluated by IF, SCF and SEPB methods are similar for dental porcelains contributed by a vitrous phase. For porcelain containing leucite phase the IF, SCF result was similar and lower than SEPB [49]. For all three methods fracture toughness increased with increasing leucite content, indicating that the main toughening mechanism was crack deflection around leucite agglomerates. The transition of K_{Ic} value from short (IF and SCF) to long (SEPB and SEVNB) pre-cracked is likely to exist in porcelains reinforced with leucite, dictated by spartial distribution of leucite particles [49]. For design and failure analysis, K_{Ic} determined by SCF method is preferred, since fracture of dental restoration usually starts from small surface cracks. An inference from the above conclusion is that increasing the

intrinsic toughness of the glassy metrix and/or improving the homogeneity of the leucite particle dispersion would enhance the resistance to short crack propagation [49].

Recently, the single-edge-V-notched-beam (SEVNB) method, where a saw cut is tapered to a sharp V-notch using razor blade sprinkled with diamond paste, was reintroduced. The fracture toughness was measured with this method on five advanced technical ceramics in an international round robin. It showed that the repeatability and reproducibility of this method was very good. Most participants had no difficulties conducting the measurements and rated the SEVNB method as user friendly, easy and cheap to conduct, reliable, accurate and worthwhile for standardization [50].

The improvement of the in-service reliability of ceramics can be achieved by increasing its fracture toughness [51]. Several toughening mechanisms with differing effectiveness have been used to improve the properties of dental ceramics. The most relevant mechanisms, described by Swain and subsequently by Evans, can be classified by: crack deflection; zone shielding; contact shielding and crack bridging[52]. Crack deflection occurs when a crack is deflected from its trajectory as a result of residual stresses, fracture-resistant second phase and grain boundaries. The reorientation of the crack plane away from normal to the applied tensile stresses causes dispersion of its energy which corresponds to an increase of the fracture toughness of the material. A shielding zone, which brings about a reduction of stress intensification at the crack tip, can result from microcrack and transformation toughening. Microcrack toughening can occur in ceramics that contain high-localized residual stresses. These residual stresses can occur in a region with thermal expansion anisotropy in polycrystalline materials with elongated grains and/or thermal expansion or elastic mismatch in polyphase materials and/or in transforming materials. Microcracks normally occur along the lowest energy path, such as the lower modulus and toughness glassy phase in a glass–ceramic. Transformation toughening is characteristic of zirconia-based ceramics. In materials where contact shielding is involved, the crack deviation and the dissipation of its energy are due to the physical contact between the opening faces of a growing crack which may result in friction between interlocking grains (pull-out of grains). The stresses at the crack

tip are also reduced because of the closure forces resulting from such crack bridging sites.

The increasing demand for all ceramic restoration has led to the improvement of veneering material for better compatibility with all ceramic core. Thus the information for the mechanical properties especially fracture toughness and flexural strength are needed for being an important data to develop a high quality of ceramic restoration.

CHAPTER III

MATERIALS AND METHODS

Preparation of the specimens

Three veneering porcelains used in this study are listed in Table 1 with their code letters. Bar-shaped specimens of each type were prepared according to the recommendations of the manufacturers and ISO 6872: Dentistry-ceramic materials [53]

Table 2 Veneering porcelains investigated in this study.

Product	Code	Type	Manufacturer
Vita VMK95	VM	Leucite containing feldspathic porcelain	VITA Zahnfabrik, Germany
Cercon Ceram Kiss	CC	Low-fusing glass	Dentsply Ceramco USA
IPS Eris Veneer	EV	Low-fusing fluorapatite glass	Ivoclar Vivadent (Schaan, L)

Ten bar specimens of each group (VM, CC, EV) were prepared. The slurry of porcelain powder was vibrated and condensed into a silicone mold, 2.2 mm deep, 4.2 mm wide and 25 mm long. The mould was filled and excess water at the free surface of the specimen was removed with absorbent paper. After condensation, the specimen was removed from the mold and transferred to a firing tray. The specimens were sintered in a porcelain furnace (Programat P100, Ivoclar Vivadent) according to the firing cycle of each manufacturer.

All specimens were finished sequentially using 30 µm and 15 µm diamond disks. After polishing, the polished specimens were 2.0 mm thick, 4.0 mm wide, and 25 mm long. The parallelism and flatness of opposing surfaces of each specimen was checked with a micrometer to a tolerance within $\pm$0.05 mm. The test pieces were thoroughly rinsed with water to remove all grinding debris.

Four-point bending test

The four-point bending test was performed using universal testing machine (Model 4465, Instron Corp, Canton, MA) on four-point bending fixture (20 mm outer span, 10 mm inner span) at cross head speed of 0.5mm./min. The following formula was used to calculate flexural strength;

$$\sigma = \frac{3PL}{4bd^2} \quad (3)$$

Where

- P is the load at failure, in Newtons;
- L is the inner test span (center to center distance between inner support rollers), in millimeters;
- b is the width of the specimen, i.e., the dimension of the side at right angles to the direction of the applied load, in millimeters;
- d is the thickness of the specimen, i.e., the dimension of the side parallel to the direction of the applied load, in millimeters;

SEVNB method

Fracture toughness of each material was determined using the SEVNB method. Five specimens of each group were prepared with a depth of 4.0 mm, 3.0 mm in thickness and 25 mm in length (Figure 1). Before testing, each specimen was cut to get the V-notches at the center of each specimen's tensile surface. The V notches

could be produced either by hand or by automated means. The suggested procedure could be performed in three steps:

1) Mount five specimens and two dummy specimens (used to protect test specimens during saw cutting and polishing of the starter notch) as close together as possible on to flat holder that allow uniform cutting in diamond saw. Face one 3 mm. wide side up to receive the starter notch (this side would be in tension during fracture test). Draw a pencil line along the measured center of the beam lengths for orientation of the saw cut (Figure 2).

2) Mount the holder in diamond saw. Use a blade having thickness as close to or only slight larger than the thickness of the razor blade. Saw a starter notch along the length of the pencil line to a uniform depth over all specimens of approximately 0.5 mm (Figure 3). Clean the specimens, especially the notch, following the saw cut to remove debris prior to polishing the notch.

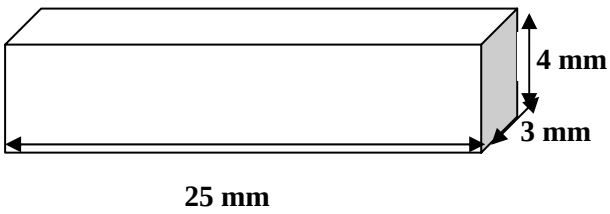


Figure 1 Specimen configuration

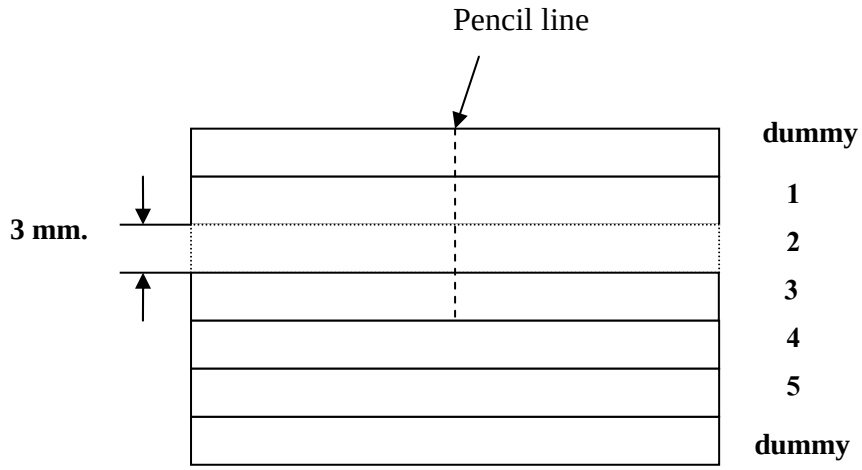


Figure 2 Schematic diagram of a specimen before receiving the starter notch

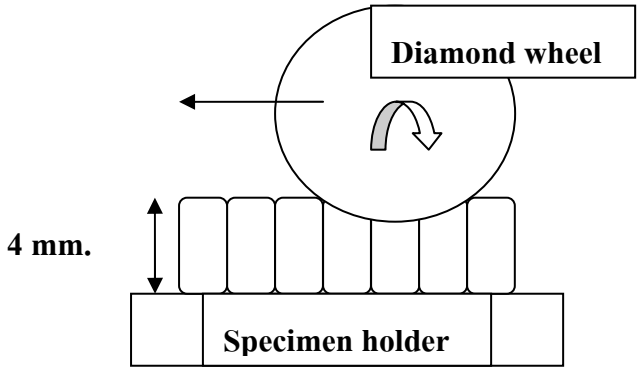


Figure 3 Schematic diagram of preparing the starter notch with diamond wheel

3) Polish a second deeper notch into this slot with a razor blade sprinkled with diamond paste having a maximum grain size between 3 µm. and 6 µm. Put razor blade into starter notch and apply light force and polish using a gentle back-forth motion as straight as possible. Using a light microscope examine both ends of the V-notch occasionally for evenness of depth. The final V-notch depth should be uniform and lie between 0.8 mm. and 1.2 mm. Remove the specimens from the holder and clean them with acetone in an ultrasonic bath. Dry the specimens well.

4) Place the 3mm width face with the V-notch down. Load the specimens with a crosshead speed of 0.5 mm/min at room temperature in air. Record the thickness (B) and width (W) of each specimen from measurement made using a micrometer capable of measuring to three decimal places. The depth of V-notches are measured using a calibrated microscope with a magnification $\geq$ 50X. Read the depth a_1, a_2 and a_3 to three significant figures (figure4).

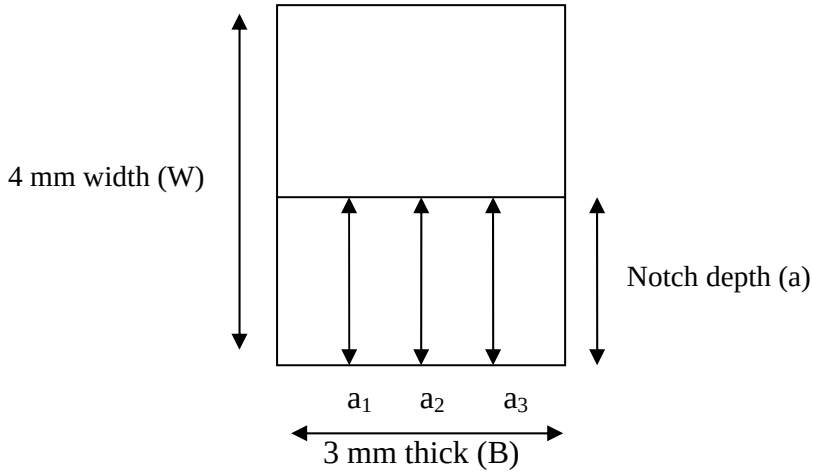


Figure 4 Schematic of the depth of V-notches a_1-a_3 must be measured

The average V-notch depth and the fracture toughness K_{Ic} was calculated using the following equations.

1) Average (a) and relative (α) V-notch depths were calculated for each specimen.

$$a = (a_1 + a_2 + a_3) / 3 \quad (4)$$

$(a_{max}-a_{min})/a \leq 0.1$ (this relationship was assumed to be satisfied)

$$\alpha = a/W \quad (5)$$

Where

a average notch depth (m)

a_{max} maximum among a_1, a_2 and a_3 (m)

a_{min} minimum among a_1, a_2 and a_3 (m)

α relative V-notch depth (-)

2) Fracture toughness, K_{Ic} was calculated for each specimen using the following equations.

$$K_{Ic} = \sigma \sqrt{a}\, Y = \frac{F}{B\sqrt{W}} \cdot \frac{S_1 - S_2}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y \quad (6)$$

$$Y = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2} \quad (7)$$

Where

K_{Ic} fracture toughness (MPa√m)

σ fracture strength (MPa)

F fracture load (MN)

B specimen thickness (m)

W specimen height (m)

S_1, S_2 support span ($S_1 > S_2$) (m)

Y stress intensity shape factor (-)

The means flexural strength and fracture toughness data of all groups were analyzed using one-way ANOVA and the Tukey post hoc test. The alpha value was set at 0.05.

CHAPTER IV

RESULTS

Flexural strength

The flexural strength values and standard deviations of three veneering porcelains are shown in Table 2. These values ranged from 39.2 MPa for IPS Eris to 80.1 MPa for Vita VMK95. There was no significant difference between means of three dental porcelains ($p>0.05$)

Fractur toughness

The fracture toughness values and standard deviations of three veneering porcelains are shown in Table 2. These values ranged from 0.89 MPa•√m for IPS Eris to 1.04 MPa•√m for Vita VMK95. The results from statistical analysis showed that mean fracture toughness of Vita VMK95 was significantly higher than IPS Eris ($p<0.05$)

The representative SEM micrographs of a uniform V-notch depth of three dental porcelains are shown in Figure 7 (a-c). From the SEM pictures, there were some porosities inside the specimen at polished surface.

Table 3 Means fracture toughness and flexural strength of three veneering porcelains.

Veneering porcelains	Mean fracture toughness (MPa•√m)	Mean flexural strength (MPa)
Vita VMK95	1.04 ±0.09	65.2 ±7.4
Cercon Ceram Kiss	0.97 ±0.05	63.9 ±13.1
IPS Eris	0.89 ±0.05	59.4 ±14.5

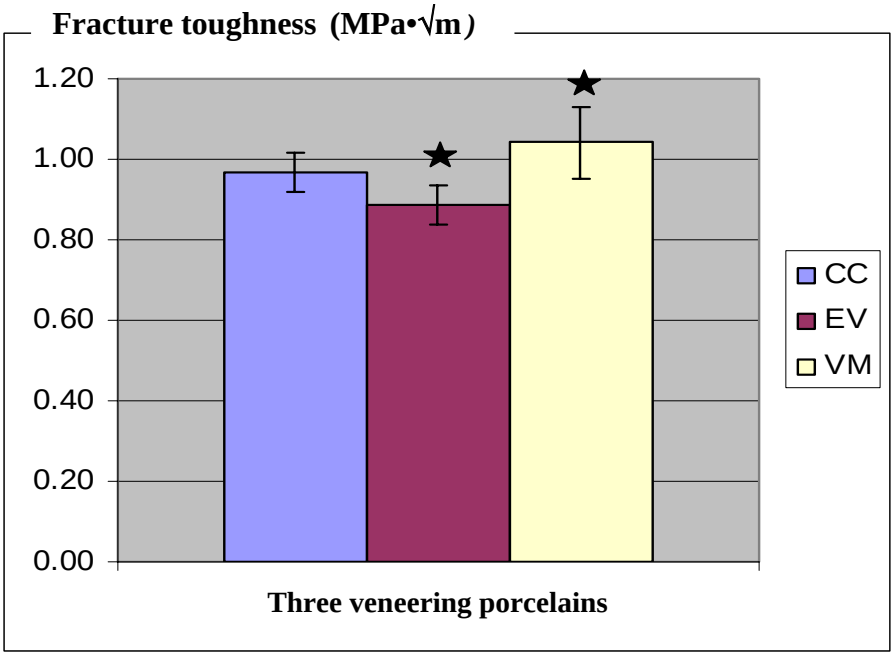


Figure 5. Means fracture toughness of three veneering porcelains.

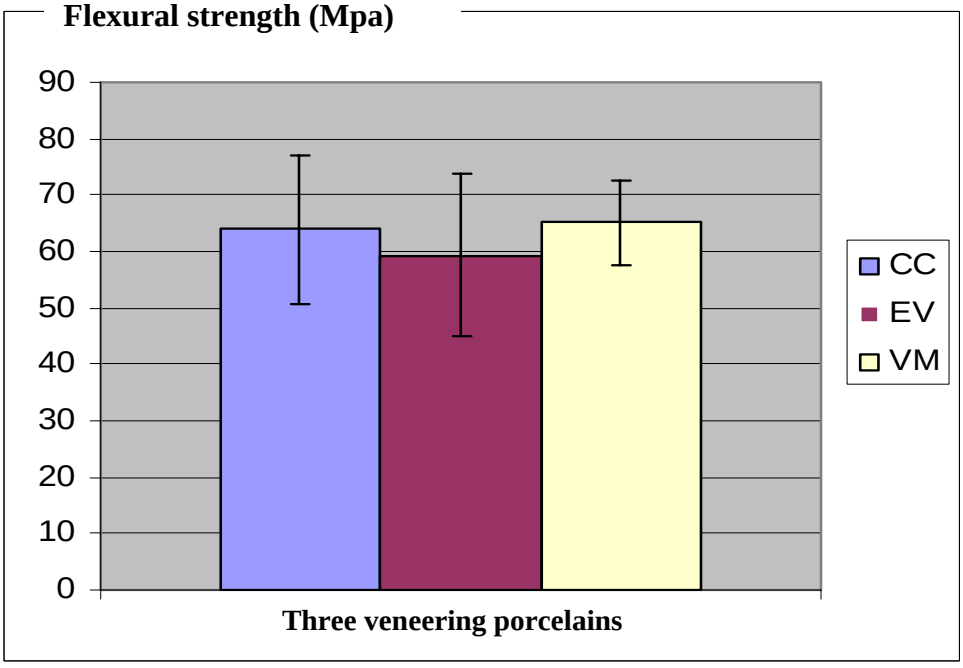
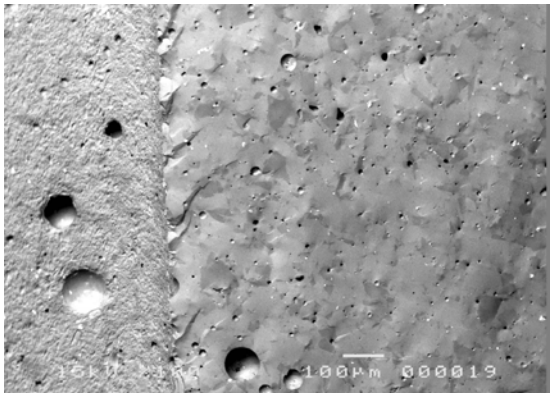
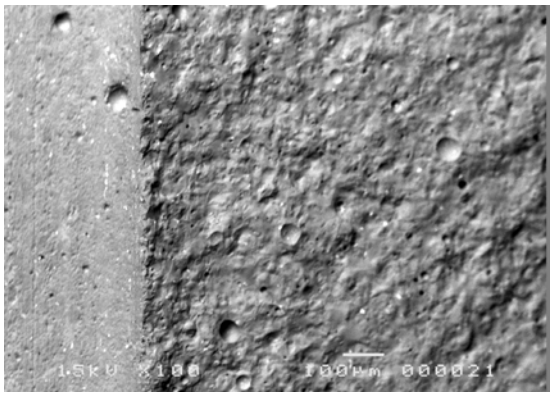


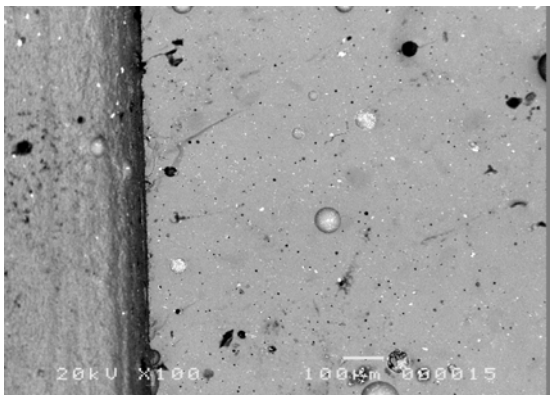
Figure 6. Means flexural strength of three veneering porcelains.



(a) IPS Eris



(b) Vita VMK 95



(c) Cercon Ceram Kiss

Figure 7.The SEM micrographs of a uniform V notch cut.

CHAPTER V

DISCUSSION

Fracture toughness (K_{Ic}) is an important material property. Its value characterizes the resistance of a material against a propagating crack. The higher the K_{Ic} value, the better is the mechanical behavior of a component fabricated out of this material. Unfortunately, dental porcelains have low K_{Ic} values because their main composition is based on glass composition. Nevertheless, those ceramic materials are still used in dental applications because of their esthetic advantages.

Vita VMK95 contains leucite crystals in its composition according to results from previous studies [21, 54]. There are two reasons for having leucite in dental porcelain, to increase its linear coefficient of thermal expansion and to strengthen the porcelain structure. In this study, the mean fracture toughness of Vita VMK 95 was significantly higher than that of IPS Eris but was comparable to Cercon Ceram Kiss. According to results from previous study, an increase in K_{Ic} of dental porcelain was observed as a result of increasing leucite content [54, 55].However, not only the leucite content of dental porcelain that affected its mechanical properties, crystal size and shape had to be taken into consideration also [56] For VMK 95, crystal size is approximately 17 μm according to data provided by the manufacturer. The amount of leucite in dental porcelain is usually less than 22 vol% [57]. The amount of crystals incorporated into the materials is limited. An increase in crystal volume fraction would decrease the translucency of a material. Esthetics will be compromised for this increase. Coefficient of linear thermal expansion of leucite is also quite high comparing with dental alloys. Increasing leucite content would raise a coefficient of linear thermal expansion of dental porcelain that would create a thermal expansion mismatch between dental alloy and veneering porcelain. And fracture of dental porcelain would be the result from this mismatch. Microcracks around leucite crystals were also reported in some studies [55]. These microcracks result from the thermal expansion mismatch between high expansion leucite crystals and low expansion glass

matrix. However, crystal size less than 4 μm will cause less microcrack than larger crystal [55]. Microcrack toughening mechanism is proposed when this crack can inhibit crack propagation. In contrast, if this microcrack causes failure as a critical flaw for crack propagation, it would lower strength of a material.

Means flexural strength of three veneering porcelains ranged from 59 MPa to 65 MPa. No significant differences were found between means of these porcelains. As previously mentioned, composition of dental porcelain was based on composition of glasses. Therefore, their flexural strengths were comparable to glasses. Even higher fracture toughness was observed for VITA VMK 95, but its flexural strength was comparable to those of the remaining materials. However, porosities were observed on the fracture surfaces of all materials. These porosities might be the reason for low strength of these veneering materials. (Fig. 8-10)

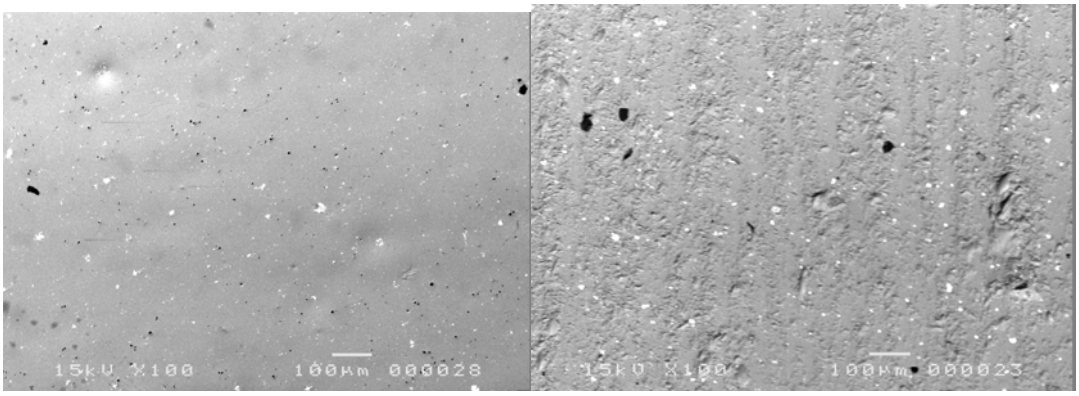


Figure 8 SEM pictures show the specimen polished surface (left) and fractured surface (right) of Cercon Ceram Kiss.

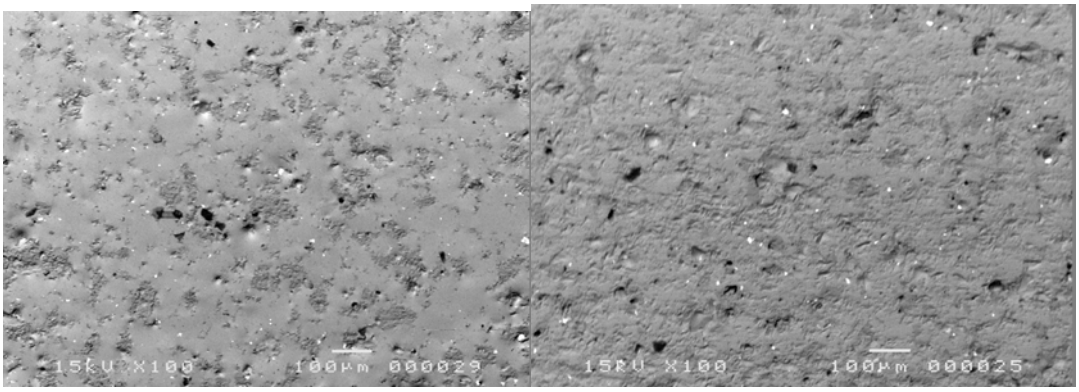


Figure 9 SEM pictures show the specimen polished surface (left) and fractured surface (right) of IPS Eris Veneer.

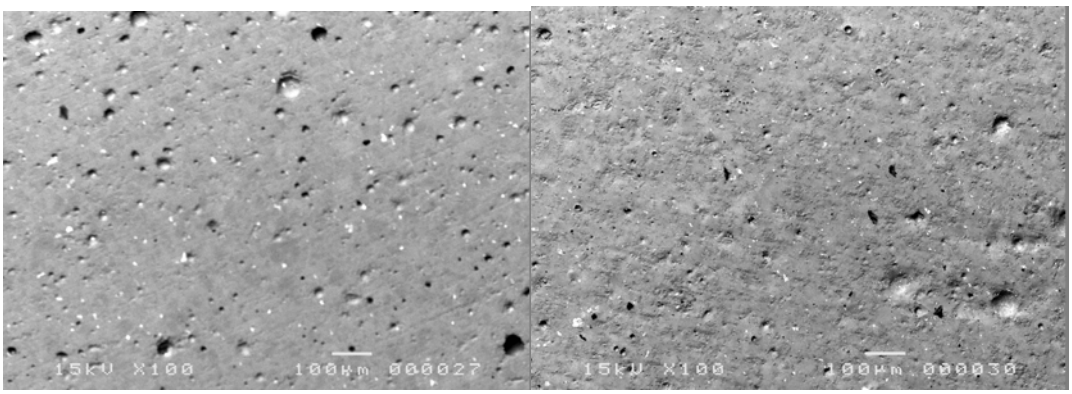


Figure 10 SEM pictures shown the specimen polished surface (left) and fractured surface (right) of IPS Eris Veneer.

Veneering porcelain is the esthetic part of crown and bridge, so there are many limitations that have to be taken into consideration when improving or adjusting the composition or processing method because it may affect porcelain translucency. However, the property of porcelain can influence the overall function and performance of dental fixed partial prosthesis because it is the part that directly contact to natural tooth while chewing. Fracture can occur at a veneering layer and core structure. Porcelain fracture can cause from its low strength or CTE mismatch. Study of physical and mechanical properties of dental porcelain will provide information that can be used for determining a success or failure mechanisms for dental prostheses.

Besides the failure from porcelain fracture, another failure that cause from porcelain is enamel wear of natural tooth. Several previous studies revealed that rate of natural tooth wear from contacting with porcelain veneered crown and bridge was higher than that when contacting with natural tooth. Prevention of natural tooth wear by using low surface hardness, polished and glazed porcelain surface. Nowadays most of information of natural tooth wear was from laboratory research which might not be clinically relevant. Future clinical study is required for dental material characterization and development.

CHAPTER VI

CONCLUSION

1. The fracture toughness value of Vita VMK 95 was significantly higher than IPS Eris Veneer. ($p<0.05$)
2. There was no significant difference between f means flexural strength of three veneering porcelains. ($p>0.05$)
3. Porosities were observed on the fracture surfaces of all materials. These porosities might be the reason for low strength of these veneering materials.

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APPENDIX

Appendix

Fracture toughness' data

	B(m)	W(m)	a1(m)	a2(m)	a3(m)	a	α	Y	K(MPa√m)
C-1	**0.00293**	**0.00399**	**0.000831**	**0.000833**	**0.000833**	**0.000832**	**0.208605**	**1.319151**	**0.87**
C-2	**0.003**	**0.00401**	**0.000811**	**0.000813**	**0.00082**	**0.000815**	**0.203159**	**1.330647**	**0.99**
C-3	**0.00299**	**0.00403**	**0.000808**	**0.000809**	**0.000804**	**0.000807**	**0.200248**	**1.336882**	**0.98**
C-4	**0.00302**	**0.00399**	**0.000803**	**0.000811**	**0.000801**	**0.000805**	**0.201754**	**1.333647**	**0.99**
C-5	**0.00301**	**0.004**	**0.000813**	**0.0008**	**0.000808**	**0.000807**	**0.20175**	**1.333657**	**0.99**
E-1	**0.00307**	**0.00407**	**0.000802**	**0.000961**	**0.00085**	**0.000871**	**0.214005**	**1.307968**	**0.98**
E-2	**0.00306**	**0.00406**	**0.0009**	**0.000916**	**0.00091**	**0.000909**	**0.22381**	**1.288198**	**0.84**
E-3	**0.00306**	**0.00407**	**0.00093**	**0.000954**	**0.00094**	**0.000941**	**0.231286**	**1.273568**	**0.86**
E-4	**0.00301**	**0.00399**	**0.00087**	**0.000875**	**0.000873**	**0.000873**	**0.218713**	**1.298389**	**0.87**
E-5	**0.00304**	**0.00403**	**0.000985**	**0.000912**	**0.000955**	**0.000951**	**0.235897**	**1.26473**	**0.89**
V-1	**0.003**	**0.00399**	**0.00103**	**0.001002**	**0.001001**	**0.001011**	**0.253383**	**1.232452**	**0.95**
V-2	**0.00301**	**0.00403**	**0.00103**	**0.001044**	**0.001035**	**0.001036**	**0.257155**	**1.225737**	**1.13**
V-3	**0.00301**	**0.004**	**0.000983**	**0.001009**	**0.001**	**0.000997**	**0.249333**	**1.23976**	**0.94**
V-4	**0.003**	**0.00399**	**0.00118**	**0.001181**	**0.00118**	**0.00118**	**0.295823**	**1.161488**	**1.06**
V-5	**0.003**	**0.00398**	**0.000954**	**0.001**	**0.000975**	**0.000976**	**0.24531**	**1.247118**	**1.13**

Flexural strength's data

	P(N)	W(mm)	T(mm)	F(MPa)
V1	**87.438**	**4.11**	**2.04**	**76.681**
V2	**83.586**	**4.01**	**2.09**	**71.579**
V3	**56.471**	**3.99**	**1.99**	**53.609**
V4	**58.136**	**4.01**	**1.99**	**54.914**
V5	**65.687**	**4.03**	**2.04**	**58.750**
V6	**82.125**	**4.04**	**2.09**	**69.806**
V7	**77.81**	**4.11**	**2.03**	**68.912**
V8	**70.202**	**3.96**	**2.01**	**65.819**
V9	**74.332**	**4.02**	**2.08**	**64.108**
V10	**77.832**	**4.01**	**2.08**	**67.294**
C1	**51.486**	**4.06**	**2.03**	**46.160**
C2	**66.986**	**3.98**	**2.05**	**60.074**
C3	**77.61**	**3.99**	**2.07**	**68.092**
C4	**74.485**	**4.06**	**2**	**68.798**
C5	**66.367**	**4.05**	**2.00**	**61.451**
C6	**83.301**	**4.01**	**2.05**	**74.146**
C7	**51.218**	**3.97**	**1.97**	**49.864**
C8	**83.192**	**4.07**	**2.01**	**75.890**
C9	**49.56**	**3.98**	**1.96**	**48.621**
C10	**90.013**	**3.92**	**2**	**86.109**
E1	**67.21**	**4.04**	**2.05**	**59.379**
E2	**90.019**	**4.01**	**2.04**	**80.913**
E3	**60.179**	**4.03**	**2.07**	**52.275**
E4	**59.432**	**4.04**	**2.03**	**53.547**
E5	**66.369**	**4**	**2.02**	**60.995**
E6	**48.662**	**3.96**	**1.95**	**48.475**
E7	**46.175**	**3.95**	**1.96**	**45.645**
E8	**39.314**	**3.96**	**1.95**	**39.163**
E9	**81.107**	**4.00**	**2.04**	**73.085**
E10	**90.01**	**4.01**	**2.05**	**80.118**

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