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CHARACTERISTICS OF COMPOSITE RESIN BLOCKS FOR CAD/CAM

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ศศิพินธุ์ เลาวหุตานนท์ : ลักษณะเฉพาะของก้อนคอมโพสิตเรซินที่ใช้คอมพิวเตอร์ช่วยในการออกแบบและผลิต (CHARACTERISTICS OF COMPOSITE RESIN BLOCKS FOR CAD/CAM) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ทพ. ดร. แมนสรวง อักษรนุกิจ, หน้า.

การศึกษาในครั้งนี้มีวัตถุประสงค์เพื่อเปรียบเทียบคุณสมบัติของก้อนคอมโพสิตเรซิน ก้อนคอมโพสิต-เซรามิก และก้อนเซรามิกที่ใช้คอมพิวเตอร์ช่วยในการออกแบบและผลิต โดยใช้การทดสอบความแข็งแรงดัดขวาง โมดูลัสยืดหยุ่นดัดขวาง ความแข็งผิววิกเคอร์ การสึกแบบวัตถุสองชิ้น และการสึกแบบวัตถุสองชิ้นที่มีสารขัดสี และความแตกต่างของสีเมื่อแช่ชิ้นงานในกาแฟหรือน้ำ ทำการวิเคราะห์ข้อมูลที่ได้ทางสถิติโดยใช้การวิเคราะห์ความแปรปรวนและการเปรียบเทียบพหุคูณแบบทูกี ที่ระดับความเชื่อมั่นร้อยละ 95 ผลการทดสอบชิ้นงานที่เก็บภายใต้อุณหภูมิห้องมีค่าความแข็งแรงดัดขวางอยู่ในช่วง 127-242 MPa โมดูลัสยืดหยุ่นดัดขวางอยู่ในช่วง 9.6-51.5 GPa และความแข็งผิววิกเคอร์อยู่ในช่วง 64-455 การสึกแบบวัตถุสองชิ้นของก้อนคอมโพสิตเรซินที่ใช้คอมพิวเตอร์ช่วยในการออกแบบและผลิตสามชนิดที่ใช้ในการทดลองมีความต้านทานการสึกที่ดีกว่าก้อนคอมโพสิต-เซรามิกและก้อนเซรามิก การสึกแบบวัตถุสองชิ้นที่มีสารขัดสีพบว่าก้อนคอมโพสิตเรซินสี่ชนิดมีความต้านทานการสึกไม่แตกต่างกับก้อนคอมโพสิต-เซรามิกและก้อนเซรามิกอย่างมีนัยสำคัญทางสถิติ และความแตกต่างของสีเมื่อแช่ชิ้นงานในกาแฟหรือน้ำ พบว่าเมื่อแช่ชิ้นงานในกาแฟความแตกต่างของสีจะมีค่าสูงกว่าแช่ชิ้นงานในน้ำอย่างมีนัยสำคัญทางสถิติ เมื่อขัดชิ้นงานที่แช่ในกาแฟมาเป็นเวลา 1 เดือน พบว่าค่าความแตกต่างของสีของก้อนคอมโพสิตเรซินสี่ชนิดมีค่าลดลง เมื่อนำข้อมูลที่วัดได้มาวิเคราะห์ทางสถิติโดยใช้แพร์ทีเทส ที่ระดับความเชื่อมั่นร้อยละ 95 พบว่าค่าความแตกต่างของสีที่วัดได้ของก้อนคอมโพสิตเรซินสี่ชนิดจากทั้งหมดห้าชนิดที่ใช้ในการทดลอง ภายหลังการขัดมีค่าลดลงอย่างมีนัยสำคัญทางสถิติดังนั้นคุณสมบัติของก้อนคอมโพสิตเรซินที่ใช้ในการศึกษาครั้งนี้เหมาะสมที่จะใช้ขึ้นรูปชิ้นงานเพื่อบูรณะฟันหลักซี่เดียวบริเวณฟันกรามน้อย

ภาควิชา ทันตกรรมประดิษฐ์ ลายมือชื่อนิสิต .....

สาขาวิชา ทันตกรรมประดิษฐ์ ลายมือชื่อ อ.ที่ปรึกษาหลัก .....

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SASIPIN LAUVAHUTANON: CHARACTERISTICS OF COMPOSITE RESIN BLOCKS FOR CAD/CAM. ADVISOR: ASSOC. PROF. MANSUANG ARKSORNNUKIT, Ph.D., pp.

This study compared commercial composite resin blocks, composite ceramic block and ceramic block for use in computer-aided design/computer aided manufacturing (CAD/CAM). Flexural strength (FS), flexural modulus (FM), Vicker hardness (VH), two- and three-body wear and discoloration were determined. The data was statistically analyzed using analysis of variance (ANOVA), Tukey's HSD multiple comparisons and pair t test (α=0.05). After dry storage, FS ranged from 127 to 242 MPa, FM from 9.6 to 51.5 GPa, and VH ranged from 64 to 455. The specimens were tested in a ball-on-disc wear device fitted with a zirconia ball (50 N loads, 1.2 Hz, 50 k cycles) in water for two-body and in poppy seed slurry for three-body wear evaluation. Two-body wear for composite resin blocks was small, hybrid ceramic and ceramic blocks showed greater volume loss. Three-body wear was very low for all materials. The color differences (ΔEs) after immersion in coffee of all materials were significantly greater than those after immersion in water. The discoloration found was extrinsic and could be removed effectively in most case with prophylaxis paste polishing. The composite resin blocks for CAD/CAM are considered suitable for fabrication of singles full crown restorations on premolar teeth.

Department: Prosthodontics Student's Signature .....
Field of Study: Prosthodontics Advisor's Signature .....
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CHAPTER I

INTRODUCTION

Computer-aided design/computed-aided manufacturing (CAD/CAM) technologies were introduced to dentistry in the 1980s. During the last decade, CAD/CAM systems in dentistry have rapidly gained importance. CAD/CAM generated dental restorations can achieve standardized manufacturing processes, uniform material quality, reproducibility of restorations, and reduction in production costs. CAD/CAM blocks available for dental restoration include feldspathic glass ceramics, leucite-reinforced glass ceramics, lithium disilicate glass ceramics, zirconia, and composite resin. The interest in fabricating restorations using CAD/CAM systems continues to grow worldwide. Moreover, the demand in nonmetallic restorations from both clinicians and patients has encouraged researchers to seek alternative materials replacing metallic material. Therefore, new generation of ceramic and composite resin for CAD/CAM system were rapidly developed in dentistry.

All-ceramic restorations with the advantages of soft tissue biocompatibility, color stability, improved wear resistance, and excellent light transmitting properties have been developed. In 1965, McLean and Hughes introduced an alumina-reinforced feldspathic core ceramic to improve mechanical and physical properties, which was used for anterior teeth restoration[1]. Other systems have also been developed and launched in the market. However, such as brittleness, crack

propagation, low tensile strength, wear resistance, and marginal accuracy, are the limitations of their use.

In the early 1990s, zirconia had been introduced in prosthetic dentistry for the fabrication of crowns and fixed partial prostheses[2]. The mechanical properties of zirconia are the highest ever reported for any dental ceramic. Zirconia is difficult to mill and time consuming in fabricating prostheses. All-zirconia monolithic restorations can be used for fabrication of posterior fixed partial prostheses. However, zirconia is white opaque material, its application is restricted to the less aesthetically area[3].

Application of restorative resins has expanded from direct restorations to laboratory processed prosthetic work. The composite resin blocks were developed and manufactured for CAD/CAM systems. The CAD/CAM composite resin blocks are industrially polymerized under standardized parameters at high temperature and pressure to ensure the constant quality in the properties. Extensive researches directed toward the improvement of dental material properties and new generation of CAD/CAM composite resin blocks have become available[4]. Recently, composite resin block for CAD/CAM systems was developed as an alternative to the ceramic blocks. One of the advantages is that this material can be repaired and fabricated easier than ceramics[5, 6]. The advantages of CAD/CAM composite resin block over a direct composite restoration are better polymerized material, less porosity, more homogeneity, and without in vivo polymerization shrinkage[5].

There are several CAD/CAM composite resin blocks available in the dental market, but their characteristics have not yet been clearly elucidated. The objective of this study is to investigate the important characteristics of CAD/CAM composite resin blocks compared with an available ceramic block. The null hypotheses were that there would be no differences regarding the characteristics between the CAD/CAM blocks evaluated.

Objectives

1. To investigate the flexural strength and flexural modulus of CAD/CAM blocks
2. To investigate the hardness of CAD/CAM blocks
3. To investigate the filler content, filler morphology and element analysis of CAD/CAM blocks
4. To investigate the two- and three-body wear of CAD/CAM blocks
5. To investigate the color stability of CAD/CAM blocks

Hypotheses

The null hypotheses were that:

There would be no significant differences in flexural properties among the CAD/CAM blocks investigated.

There would be no significant differences in hardness among the CAD/CAM blocks investigated.

There would be no differences in filler micro-morphology among the CAD/CAM blocks investigated.

There would be no differences in filler element among the CAD/CAM blocks investigated.

There would be no significant differences in quantitative wear and no differences in micro-morphology of the worn surfaces in two-body wear among the CAD/CAM blocks investigated.

There would be no significant differences in quantitative wear and no differences in micro-morphology of the worn surfaces in three-body wear among the CAD/CAM blocks investigated.

There would be no significant differences in color stability among the CAD/CAM blocks investigated.

Research design

Laboratory experimental research

Part I Mechanical properties of composite resin blocks for CAD/CAM

ABSTRACT

This study compared commercial composite resin blocks with one ceramic block for use in CAD/CAM. Four composite resins, one composite ceramic, and one feldspar-ceramic block were investigated. Flexural strength (FS), flexural modulus (FM), and Vickers hardness (VH) were determined under three conditions: dry storage; immersion in water at 37°C for 7 days; and immersion in water at 37°C for 7 days followed by 10,000 thermocycles. Flexural properties were determined using a three point bending test according to ISO 6872:2008 using a universal testing machine (TRAPEZIUM X, Shimadzu Corp., Kyoto, Japan). Vicker hardness test was measured using a micro hardness testing machine (MVK-H2, Akashi, Kawasaki, Japan) with a load of 300 gf and 15s dwelling time. After dry storage, FS ranged from 127 to 242 MPa, FM ranged from 9.6 to 51.5 GPa, and VH ranged from 64 to 455. After water 7 days, FS ranged from 121 to 197 MPa, FM ranged from 7.8 to 52.8 GPa, and VH ranged from 59 to 454. After water 7 days followed by 10,000 thermicycles, FS ranged from 118 to 294 MPa, FM ranged from 97.2 to 54.9 GPa, and VH ranged from 57 to 449. Two-way ANOVA was performed for FS, FM and VH followed by Tukey's multiple comparison (α<0.05). Results demonstrated that the materials degraded after water immersion and thermocycling, but their properties were within the acceptable range for fabrication of single restorations according to the ISO standard for ceramics (ISO 6872:2008).

INTRODUCTION

CAD/CAM technologies were introduced in dentistry during the 1980s. During the last decade CAD/CAM systems in dentistry have rapidly gained importance and popularity. CAD/CAM generated dental restorations meet standardized manufacturing processes with uniform material quality, reproducibility of restorations and reduction in production costs. CAD/CAM blocks available for dental restoration include lithium disilicate glass ceramics, leucite-reinforced glass ceramics, feldspathic glass ceramics, aluminum-oxide and yttrium tetragonal zirconia polycrystals[7], composite resin[8] and titanium that is mostly used for implant abutments[9]. Practitioners' interest in fabricating restorations using CAD/CAM systems continues to grow worldwide. Moreover, the demand for nonmetallic restorations from both clinicians and patients has encouraged researchers to seek alternative materials.

The first CAD/CAM ceramic blocks were launched to the dental market in 1985[10], having a flexural strength of approximately 120 MPa[11]. These blocks were intended for fabrication of inlays, onlays and veneers. Improved ceramic blocks containing approximately 30 volume % of fine distributed crystals of leucite were developed in 1991[12]. This ceramic material reportedly had enamel-liked abrasion characteristics[13]. Therefore, it was considered suitable for fabrication of inlays, onlays and monolithic anterior crown and veneers[14]. Single tooth restorations constructed with this material demonstrated an overall failure rate of approximately 22% after 4 years in service[15]. A recent study of this ceramic material reported a flexural strength

of 103 MPa when tested using a three-point bending test[16]. This value is slightly higher than the flexural strength proposed by International Organization for Standardization (ISO) 6872:2008 for single unit restorations[17].

The first composite resin block for CAD/CAM systems was launched in 2000, and was originally a product polymerized by light activation using factory processes[8, 18]. Concurrently with the development of ceramic blocks for CAD/CAM, composite resin technology made considerable progress with the development of nanofilled and nanohybrid composite resins. The new nanofiller-containing composite resins exhibited equivalent mechanical properties to the clinically proven microhybrid type composites and were thus recommended for direct posterior restorations[19]. This development led to the application of these materials for indirect restorations through combined light and heat polymerization modes[20]. Nevertheless, the properties of composite resin still depend largely on the resin matrix used; the filler type, shape and size; and on the filler/matrix interface coupling[21].

Significant improvement in the mechanical characteristics of composite resin blocks for CAD/CAM was achieved by applying heat-polymerization under high pressure to hybrid, nanofilled and nanohybrid composite resins[5]. Additionally, restorations produced from CAD/CAM composite resin blocks can be more easily fabricated and repaired than restorations made from CAD/CAM ceramic blocks[5, 22].

An approach leading to the improvement of CAD/CAM ceramic blocks was the substitution of the brittle glass phase with polymer to form a polymer-infiltrated

ceramic network. This modification improved the flexural strength and strain at failure[23]. Some reports even claimed that this type of material showed similar properties to natural tooth substance[24].

Several CAD/CAM composite resin block materials are available in the dental market. In Japan, CAD/CAM-produced composite resin premolar crowns were approved by the Japanese social insurance system in early 2014. However, the properties of these new materials have not yet been clearly elucidated. The objective of this study was therefore to investigate some important characteristics of CAD/CAM composite resin blocks when compared with a clinically proven ceramic block. The null hypothesis was that there would be no difference in characteristics among the CAD/CAM blocks.

Part II *In vitro* evaluation of the wear resistance of composite resin blocks for CAD/CAM

ABSTRACT

The aim of the study was to investigate two- and three-body wear of CAD/CAM blocks. Four composite resins, one hybrid ceramic and one feldspar ceramic block material were examined. Six specimens each were tested in a ball-on-disc wear device fitted with a zirconia ball (50 N load, 1.2 Hz, 50 k cycles) in water for two-body and in poppy seed slurry for three-body wear evaluation. Volume loss after each 10 k cycle was quantified using a digital CCD microscope. The wear data was statistically analyzed using ANOVA and Tukey's HSD post-hoc comparisons (α=0.05). Linear regression analyzes for the relationships between volume losses and numbers of sliding cycles were calculated for each block material and each wear mode. Two-body wear for composite resin blocks was small and hybrid ceramic while ceramic blocks showed larger volume loss. Three-body wear was very low for all materials. All CAD/CAM block materials investigated displayed low wear compared to previous data reported for direct posterior composites carrying out wearing the same wear test. The block materials are considered suitable for fabrication of single full crown restorations on premolar teeth.

INTRODUCTION

Patients' demands for esthetic restorations are increasing, at least in the visible area of the mouth. Porcelain-fused to metal or ceramic crowns have been the first choices for long time. However, since the introduction of CAD/CAM technology in dentistry almost 40 years ago by Duret et al.[25], and in particular after introduction of the CEREC system, developed by Mörmann et al.[26] CAD/CAM produced inlays, crowns and bridges have gained increasing attention. CAD/CAM block materials are available in the market as ceramic, hybrid ceramic, and composite resin materials[7]. The introduction of the first composite resin CAD/CAM blocks in 2000 was a milestone[8]. Concurrent with the rapid development and improvement of direct composite resin materials, recent composite CAD/CAM blocks are fabricated by high-pressure high-temperature polymerization resulting in improved mechanical characteristics[5] which might make them suitable as materials for single crown restorations. Although resin-ceramic block materials were introduced on the market more than a decade ago, published scientific evidence proving their efficacy is very limited. Long-term clinical data from controlled randomized studies do not currently exist to the best of our knowledge.

In April 2014, the Japanese social insurance system approved premolar crowns produced from CAD/CAM composite resin block materials. Consequently, several composite resin CAD/CAM block materials emerged on the market. Hence, it

is expected that patients in the future will frequently request such full coverage restorations.

In a recent publication, Lauvahutanon et al.[27] investigated mechanical properties of several composite resin CAD/CAM blocks and concluded that the materials examined are acceptable for fabrication of single restorations according to the ISO standard for ceramics (ISO 6872:2008)[17]. Apart from mechanical, physical and chemical characterization that is partly detailed by the manufacturers, wear resistance is another important property. However, so far there is very little information available regarding wear resistance. From an *in vitro* wear evaluation of five CAD/CAM resins, only one product presented wear values comparable with glass-ceramic when tested under two-body wear condition[28].

Therefore, the aim of the present *in vitro* study was to investigate two- and three-body wear characteristics of CAD/CAM composite resin blocks in comparison to a hybrid ceramic and a feldspar ceramic block. The null hypotheses were that there would be no difference in volume loss due to wearing and no difference in morphologic appearance of the worn block surfaces.

Part III Color stability of CAD/CAM composite resin blocks after immersion in coffee

ABSTRACT

This study evaluated discoloration (ΔE) of CAD/CAM blocks after immersion in coffee and water. Five composite resin blocks, one composite ceramic block, one PMMA block, and one ceramic block and four conventional restorative composite resins were evaluated. The CIE *L*a*b** values of 2.0-mm-thick disk-shaped specimens were measured using the spectrophotometer on white and black backgrounds (n=6). The ΔEs after one day, one week, and one month after immersion in water or coffee were calculated and separately analyzed by two-way ANOVA with immersion solution and immersion periods as main factors, followed by Tukey's multiple comparisons (α<0.05). After 1-month immersion in coffee and water, ΔEs ranged from 1.4 to 7.9 and from 0.3 to 1.3, respectively. Two-way ANOVA of ΔEs revealed that the two main factors, immersion solution and immersion periods, and their interaction were significant except for ΔEs of composite ceramic block and PMMA block. The ΔEs after immersion in coffee were significantly greater than those after immersion in water. When selecting materials for clinical use ΔE values of CAD/CAM composite resin blocks should be considered to ensure long-term esthetic appearance. The discoloration found was extrinsic and could be removed effectively in most cases with prophylaxis paste polishing. Maintenance polishing can remove

most of the surface stains from all CAD/CAM block materials and from the majority of the light-cure composite resins.

Introduction

Application of composite resin has been expanded from direct restorations to laboratory processed prosthetic works[20]. Composite resin block materials have been developed and manufactured for CAD/CAM systems since 2000[8, 18]. Currently, there are several new products of CAD/CAM composite resin blocks available. Such new CAD/CAM composite resin blocks are industrially polymerized composite resins under standardized parameters at high temperature and pressure to achieve satisfactory properties at microstructure level and high degree of conversion. As a result, material characteristics were improved compared to direct restorative composite resin[5]. Composite resin block materials' mechanical properties were evaluated and suggested suitable for single restorations according to the applicable ISO standard[17, 27].

However, the success of restorations depends not only on mechanical and physical properties but also on the esthetic appearance[7, 29, 30]. Tooth-colored restorative materials should feature excellent color match and color stability during clinical service[31, 32].

Restorative materials are exposed in the oral cavity and subjected to multiple and frequent influencing factors such as temperature, humidity, food, beverage and smoking. Such influencing factors may be associated with discoloration of dental restorative materials. It is known that composite resin restorations show a tendency to discoloration in the oral environment[33, 34]. Previous studies demonstrated that

composite resins were susceptible to discoloration due to various extrinsic and intrinsic factors[35]. Extrinsic factors include the influence of staining solutions such as coffee, tea, cola and red wine, while intrinsic factors are related to the resin matrix composition, initiator system, kind and duration of polymerization, conversion of the matrix monomers, particle size and oxidation of the unreacted carbon double bonds[36-42]. Due to its high potential for staining, resin-based materials' evaluation on the effect of immersion in coffee is considered a reasonable test procedure to estimate a materials' tendency to discoloration[36]. Moreover, discoloration of resin-based materials might also be attributed to water sorption. A correlation between color stability and water sorption was demonstrated[43].

To date, there are no studies published comparing and evaluating the discoloration tendency and change of translucency of recent CAD/CAM composite resin blocks after immersion in discoloring media. Therefore, the purpose of this study was to evaluate the discoloration susceptibility and translucency parameters of CAD/CAM composite resin blocks compared with direct resin composites after immersion in coffee. The null hypothesis was that there would be no significant difference regarding discoloration and translucency change among the CAD/CAM composite resin blocks after immersion in coffee.

CHAPTER II

LITERATURE REVIEW

CAD/CAM technology

The CAD/CAM is the abbreviation of computer aided design/computer aided manufacturing. The technology for CAD applied the use of computer system to assist in the creation, modification, analysis, or optimization of a design. CAM applies to the use of computer systems that plan, manage, and control in the manufacturing operations. In the early 1980s, The first dental chairside CAD/CAM system was developed and named the CEREC system[44]. In 1983, the CAD/CAM technology in fabricating composite veneered restorations was introduced. This system was later known as the Procera system[45]. Since then, many systems have been introduced into the dental market.

There are three general processes involved with the CAD/CAM system. The first process is to record the geometry of patient's dentition and soft tissues to a computer. For many years dentists have been using the conventional impression techniques to create stone models and deliver to dental laboratory. Most recently, intraoral scanners or cameras have been introduced that allow dentists to perform the scanning process without the need for conventional impression and stone models. The second process involves the utilization of the scanned information in a CAD software program. The program is utilized to propose a volume model of the

desired prosthesis on the virtual model of the dentition. Additional software editing tools allow customizing of the restoration into specific shape and size. The third process is to utilize the digital proposal of the prosthesis to direct a machining device to fabricate the desired outcome[46, 47].

The American Dental Association specifies that a dental restoration must fit its abutment within a 50 μm range. This requirement has directed the CAD/CAM system to have very accurate data collection technique, sufficient computing power to process and design complex restorations, and very precise milling system.

During the last 2 decades, exciting new developments have led to the success of dental CAD/CAM technology. Several methods have been used to collect 3-dimentional data of the prepared tooth using optical cameras, contact digitization, and laser scanning. Replacement of conventional milling discs with a variety of diamond burs has resulted in major improvements in milling technology. Integration of these technologies has resulted in the introduction of several highly sophisticated CAD/CAM systems: CEREC 3D and inLab (Sirona Dental systems, Charlotte, NC); DCS Precident (Popp Dental Laboratory, Inc, Greendale, WI); Procera (Nobel Biocare, Yorba Linda, CA); Lava (3M ESPE, St Paul, MN); Cercon Smart Ceramics (Dentsply Ceramco, York, PA); Everest (Kavo Dental, Lake Zurich, IL); Densir (Decim, Skelleftea, Sweden); DentaCad (Hint-ELS Canada Inc, Ontario, Canada) and Evolution D4D (D4D Technologies, Richardson, TX).

CAD/CAM technology provides several advantages from the dental laboratory perspective. CAD/CAM systems offer automation of fabrication procedures with increased quality in a shorter period of time. Dental CAD/CAM systems have the potential to minimize the inaccuracies in technique and to reduce hazards of infectious cross-contamination associated with conventional multi-stage fabrication of indirect restorations. However, the capital costs of these CAD/CAM systems are quite high.

Materials used for CAD/CAM system

Meanwhile, an increase in gold alloy price, allergic reactions to nickel-chromium alloy and esthetic demand forced researchers to find other alternative materials for prosthetic restoration. CAD/CAM blocks available for dental restoration include feldspathic glass ceramics, leucite-reinforced glass ceramics, lithium disilicate glass ceramics, zirconia, and composite resin. CAD/CAM materials used for fabricated prosthetic restoration can be divided in two types, monolithic material and bi-layered material. This review is focused only on monolithic material.

Monolithic materials

Glass ceramics

Glass ceramics are multiphase materials with a glassy matrix and crystalline component.

1.1 Feldspathic glass ceramics

Feldspathic ceramics are silica-based ceramics with low to moderate crystalline leucite filler ($K_2O.Al_2O_3.4SiO_2$) roughly 5-25% by volume, which is created by firing feldspar at 1,150°C[12].The high glass content in the feldsparthic ceramics results in excellent aesthetic properties which resembling the natural tooth structure[48]. Leucite particles are used to provide high translucency and to alter the coefficient of thermal expansion, as well as to improve the material strength by inhibiting crack propagation. However, the original feldspathic ceramics have a random distribution and large size of leucite particles, which contributes to the low fracture strength, ranging between 70 and 100 MPa[49].

In 1985, the first Vita Mark I block (VITA Zahnfabrik, Bad Säckingen, Germany) was developed for the Sirona CAD/CAM system (Sirona Dental system, Bensheim, Germany). This material had a flexural strength of approximately 120 MPa[11]. and was intended to be used for fabrication of inlays, onlays, and veneers.

New generations with about 30% by volume fine grain (10 to 20 μm) and distributed particles were developed (Giordano and McLaren, 2010) in 1991 as a Vita Mark II block. The fine crystal microstructure and the CAD/CAM processing technique produce the enamel-like abrasion characteristic of Vita Mark II dental restorations[13]. According to the manufacturer, the flexural strength of Vita Mark II is approximately 150 MPa, making it suitable for fabrication of inlays, onlays, and monolithic anterior

crown and veneers[14]. Most recently, single crowns made from Vita Mark II showed a survival rate of 83.78% after an observation period of 4 years[15].

1.2 Leucite-reinforced glass ceramics

The glass matrix in leucite-reinforced ceramic is based on an alumino-silicate glass. A high proportion of leucite crystal ranging from 35% to 45% by volume, is used to reinforce the glass ceramic and improve its mechanical properties[50]. The flexural strength of leucite-reinforced glass ceramic is approximately 105-129 MPa and the restorations are highly translucent[51].

To overcome the disadvantages of powder/liquid ceramics, such as micro-porosity and shrinkage, the IPS Empress ceramic (Ivoclar Vivadent, Schaan, Liechtenstein) was introduced in 1990; the most widely used leucite-reinforced pressable ceramic[12]. The ceramic ingots, supplied by the manufacturer in a variety of shades, can be pressed under heat (1050-1080°C) and pressure (0.3-0.4 MPa). Fine leucite crystal in a glassy matrix; with a size range between 1-5 μm, and heat-pressing techniques have contributed to the increased material flexural strength of 160-180 MPa[52]. This ceramic material is indicated for inlays, onlays, veneers, or crown restorations in anterior teeth[53].

IPS Empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein) is the CAD/CAM machinable version of IPS Empress ceramic. It was introduced in 2006 with a flexural

strength of about 160 MPa and designed to be either with chairside or in lab-side CEREC systems to fabricate veneers, inlays, onlays, and anterior crowns[12].

1.3 Lithium disilicate glass ceramics

In order to be able to construct anterior three-unit all-ceramic bridge restorations, a glass ceramic based on lithium disilicate ($Li_2Si_2O_5$) has been developed. Densely arranged needle-like lithium disilicate crystals at a concentration of 70% by volume[54] with a length of 4 μm and a diameter of 0.5 μm are uniformly distributed in a glass matrix. This interlocking structure hinders crack propagation and elevates the flexural strength of lithium disilicate ceramic to 300-400 MPa[55], which is more than twice the strength of glass ceramic[56].

Ivoclar Vivadent introduced the first lithium disilicate ceramic (IPS Empress II, Ivoclar Vivadent, Schaan, Lichtenstein) in 1998 in ingot form to be used with the press technique at approximately 920°C. Further improvement in physical properties and translucency of lithium disilicate glass ceramic was provided with the introduction of IPS e.max Press (Ivoclar Vivadent, Schaan, Lichtenstein). The use of this material is recommended for fabrication of monolithic inlays, onlays, and posterior crowns, or as a core for crowns and anterior three-unit fixed partial prostheses[57, 58]. An innovation in lithium disilicate glass ceramic was developed in 2005 under the name of IPS e.max CAD (Ivoclar Vivadent, Schaan, Lichtenstein) for milling techniques. The IPS e.max CAD block is a partially crystallized block consisting of 40% lithium meta-silicate crystals, allowing the material to be easily milled.

According to the manufacturer's data, the flexural strength of fully crystallized IPS e.max CAD is about 360 MPa. This material is not only indicated to fabricate monolithic inlays, onlays, single crowns, and anterior fixed partial prostheses, but also for short-span posterior fixed partial prostheses[59].

Yttrium-tetragonal zirconia polycrystal ceramics (Y-TZP)

Pure zirconia is polymorphic and exhibits three crystallographic phases at different temperatures: the cubic phase is stable from 2680°C to 2370°C, the tetragonal phase is stable from 2370°C to 1170°c, and the monoclinic phase is stable form 1170°C to room temperature. Phase transformation is associated with a substantial volume increase (4%), and causes high internal stress, which can induce severe cracking[60]. Addition of minor components such as magnesium oxide (MgO), calcium oxide (CaO), or yttrium oxide (Y_2O_3) to pure zirconia provides formation of multiphase materials known as partial stabilized zirconia (PSZ) at room temperature[61]. Yttrium-stabilized zirconia is the most popular and frequently used form of zirconia for biomedical application.

The recent introduction of zirconia-based ceramics as restorative dental materials has generated considerable interest in the dental restorative material. The mechanical properties of zirconia are the highest ever reported for any dental ceramic. This may be used in posterior fixed partial dentures and also substantial reduction in core thickness. All-zirconia monolithic restorations are indicated for fabrication of posterior single crown restorations in patients with para-functional

habits or limited occlusal space[62]. However, as zirconia is high value white opaque material, the desired tooth shade is developed by staining the restoration prior to sintering. Due to the inferior in aesthetic properties of monolithic zirconia, its application is restricted to the less aesthetically area[3]. Because of the high hardness properties of the monolithic zirconia ceramic, the wear of antagonist was extensively examined under *in vitro* conditions[63]. One in vitro study tested the wear of five zirconia materials using steatite and human enamel as the antagonist. Wear on the zirconia ceramic could not be observed, moreover, the wear rates of the enamel antagonists were also not determined. But the study reported the cracks or even fractures of the enamel antagonist at the ridges in all tested zirconia ceramics group[64].

Composite resin

The applications of composite resins have been expanded, from a direct to the indirect laboratory-processed restorative material[5, 65]. Today, the use of indirect composite materials has increased substantially. This might be related to their improvements in the properties of materials. The indirect composites can be subdivided into two groups: the indirect CAD/CAM processed composite resin blocks and the conventional laboratory-processed composite resins. The CAD/CAM composite resin blocks are industrially polymerized under standardized parameters at high temperature and pressure to ensure the constant quality in the properties. Composite resin for CAD/CAM systems was developed as an alternative to the

ceramic blocks. The major advantage of this material is repairable and easier in maintenance compared to ceramic[5, 6]. Also compared to direct composite resin, CAD/CAM composite resin has less porosity, less polymerization shrinkage and more homogeneity[5].

Through the extensive research directed toward improving the properties of dental materials, the new generation of CAD/CAM composite resin blocks has become available in dental market. These materials are claimed to be suitable for permanent dental restorations. Several manufacturers have introduced their products in the market. The followings are some of the materials available in the markets.

Block HC (Shofu Inc., Kyoto, Japan)[66]

This material is formulated using silica, silicate and Zr-silicate with a total filler content by weight of approximately 61%. The monomer compositions are UDMA (urethanedimethacrylate) and TEGDMA (triethylene glycol dimethacrylate)[66].

Cerasmart (GC corp., Tokyo, Japan)[67]

Cerasmart is the latest generation of CAD/CAM composite resin block from GC Corporation. It is introduced to the dental market in first trimester of 2014. This material is formulated using silica nanoparticles of 20 nm diameters and Ba-glass of 300 nm with total filler content by weight of approximately 71%. The monomer compositions are Bis-MEPP (2,2-Bis(4-methacryloxypolyethoxyphenyl)propane), UDMA

and DMA (dimethacrylate). According to manufacturer information, Cerasmart has a flexural strength of 240 MPa[67].

Gradia (GC Corp., Tokyo, Japan)[68]

Gradia was introduced to the dental market before Cerasmart. This material is formulated using silica, fluoroaluminosilicate glass and prepolymerized filler with a total filler content by weight of approximately 76%. The monomer compositions are UDMA and Polyfunctional methacrylate copolymer[68].

Lava Ultimate (3M ESPE, St. Paul, MN, USA)[69]

Lava Ultimate is made from resin nano-ceramic material for chairside fabrication with CEREC and E4D systems.

The material is formulated using both nanoparticles and nanocluster fillers with a total nanoceramic material content by weight of approximately 80%. Nanoparticles contain two types: silica nanoparticles of 20 nm diameter, and zirconia nanoparticles of 4 to 11 nm diameter. Nanocluster particles contain zirconia-silica nanocluster particles comprise of 20 nm silica particles and 4 to 11 nm zirconia particles. The average nanocluster particle size is 0.6 to 10 micrometers. The nanoparticles and nanocluster particles are treated with a silane coupling agent using a proprietary method. The addition of nanomer particles to the formula containing nanoclusters reduces the interstitial spacing of the filler particles, leading to higher nanoceramic content. The reinforced matrix (resin plus nanoparticles) is significant harder and much more wear resistance than pure resin. The monomer compositions

are Bis-GMA (bisphenol A diglycidylether methacrylate), Bis-EMA (ethoxylated bisphenol-A dimethacrylate), UDMA and TEGDMA. This material is processed multiple hours in a special heat treatment process. Lava Ultimate is suitable for the fabrication of permanent single restoration, such as crowns, inlays, onlays, and veneers. According to manufacturer information, Lava Ultimate has a flexural strength of 200 MPa. This material is stronger than previously introduced Paradigm MZ100 block (3M ESPE, St. Paul, MN, USA)[69].

Paradigm MZ100 block was introduced to the dental market in 2000[18]. This material is a BisGMA and TEGDMA resin-based composite with zirconia-silica spheroidal filler (85% by weight) with an average particle size of 0.6 μm. According to manufacturer reports, the flexural strength of MZ100 ranges between 150 and 160 MPa, whereas the elastic modulus is reported to be approximately 15 to 20 GPa[18].

Vita Enamic (Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany)[70]

In 2013, a novel polymer infiltrated hybrid ceramic dental material for the CAD/CAM system has been developed by VITA Company. Vita Enamic is a hybrid dental ceramic with a dual network. A porous feldspathic ceramic was reinforced by a polymer network material. It was claimed that its composition mechanical properties that were intimately resemble the natural tooth properties[23]. According to manufacturer information, the flexural strength and elastic modulus of Vita Enamic are reported to be approximately 150 to 160 MPa and 30 GPa, respectively[70].

Characteristics of CAD/CAM blocks

Mechanical properties

In the current dental literature, several studies evaluated distinct properties of dental materials, which can influence and predict their performance[71, 72]. Dental products have been rapidly developed the number of studies designed to evaluate their characteristics are also increasing. Practitioners are aware of the importance of previous laboratory and clinical trials before using the materials in clinical practice. Therefore, the knowledge of the material properties is essential to support the indication of the dental restorative materials and to ensure a long-term clinical performance[72]. In the oral environment, restorations are subjected to stress from mastication action. These forces act on teeth and/or material producing different reactions that lead to the deformation, which can ultimately compromise their durability over time[73].

Several *in vitro* tests are proposed to evaluate different properties[71-73]. In order to seek for standardized testing protocols, International Organization for Standardization (ISO) recommends and presents the main guidance for dental materials laboratory testing. However, ISO standard has no specific guideline for CAD/CAM materials. Therefore, it is important to assess the following properties which were considered relevant. Since the CAD/CAM composite resin blocks are the mixture of ceramic and polymer, the mechanical properties of the test guideline derived from both ISO

4049:2009 polymer-based restorative materials and ISO 6872:2008 dental ceramic are following proposed. The followings are some of the important mechanical properties.

Flexural strength and flexural modulus

The flexural strength of the material is its ability to bend before it breaks. It is obtained when the ultimate flexibility of one material is achieved before its proportion limit[71, 74]. Flexural forces are the result of forces generated in clinical situations and the dental materials need to withstand repeated flexing, bending, and twisting[71]. A high flexural strength is desired once these materials are under the action of chewing stress that might induce permanent deformation[72].

Dental CAD/CAM ceramics for restorations are generally categorized on the basis of their flexural strength, which is not the only characteristic determining the clinical performance. The strength values scatter within a certain range depending on materials microstructure, intrinsic flaws, geometry and finishing of specimens. The uncertainty of ceramic materials in structural applications is their brittleness and consequent limitations for mechanical reliability[75].

Strength is an important mechanical property that determines the performance of brittle materials[76]. However, microcracks and defects that grow inherently during the thermal or mechanical processes can significantly influence strength measurement. Strength is therefore considered a condition property[76, 77]. Different methods are available to assess the tensile strength of the ceramic materials. These testing method includes three-point bending[77-79], four-point bending,

the nondestructive test method[80], and the biaxial flexural test[81], which includes the ring on ring[82], ball on ring[81], and piston on 3 ball tests[78, 79]. The three-point flexural test is the standard for strength testing of dental ceramics and composite resins. However, the drawback of this test is inherent sensitivity to flaws and defects near specimen edges[83].

Require information on the materials such as the elastic modulus and Poisson's ratio, which are fundamental properties for a material and essential to dental applications. Elastic modulus represents the stiffness of a material within the elastic range when tensile or compressive forces are applied[84]. It is also an indication of the amount of reversible deformation that occurs in a structure when a load is applied to it. Within the elastic range, the ratio of the lateral strain to the axial strain is called Poisson's ratio[73].

Composite resin restorative materials are homogenously dispersed particulate reinforced polymer matrix composites. The elastic modulus of dental composites is one of the key mechanical properties which may influence the length of service of these materials. The elastic modulus relates to stiffness of a material and is defines mathematically as the slope of the stress-strain curve within the proportional limit. The modulus of elasticity is directly related to the amount of deformation when the material subjected to external forces. Hence, the dental composites used in posterior restorations must possess an adequate modulus value in order to

withstand the high masticatory force[85]. Moreover, it is a function of whole sample rather than of a weak part or of the surface only[86].

The elastic modulus and other mechanical properties as flexural strength, tensile strength, diametral tensile strength, fracture toughness, hardness, wear resistance etc., are importance in determining the resistance to occlusal forces[86].

Surface hardness

Surface characteristics are one of the determinant factors when the restorative materials are used in clinical oral environment. Surface characteristics can influence on polishing ability, on the scratch occurrence and on the wear resistance[73, 87]. Surface hardness is a parameter frequently used to evaluate material surface resistance to plastic deformation. Hardness is not instrinsic material properties dictated by precise definition terms of fundamental units of mass, length and time. A hardness property value is the result of a defined measurement procedure[73]. The usual method to evaluate hardness value is to measure the depth or area of indentation left by an indenter of a specific shape with a specific force applied for a specific time. There are four common standard test methods for evaluate the hardness of material: Brinell, Rockwell, Vickers, and Knoop[73]. Each of these methods is divided into a range of scales, defined by a combination of applied load and indenter geometry.

The Brinell hardness test method consists of indenting the material with a 10 mm diameter hardened steel or carbide ball subjected to a load. It is the oldest

method to measure surface hardness and is applicable to test metal and alloys. The Rockwell hardness test method consists of indenting the test material with a diamond cone or hardened steel ball indenter. This method is useful to evaluate surface hardness of plastic materials used in dentistry[73, 87].

The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and angle of 136 degrees between opposite faces subjected to a load of 1 to 100 Kgf. The load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The edge of the indentation is calculated. It is suitable to be applied to determine the hardness of small areas and for very hard materials[73].

The Knoop hardness is more sensitive to surface characteristics of the material. The Knoop indenter is a diamond ground pyramidal form that produces a diamond shaped indentation having approximate ratio between long and short diagonals of 7:1. The depth of indentation is about 1/30 of its length. When measuring the Knoop hardness, only the longest diagonal of the indentation is measured and used to calculate the Knoop hardness number[72]. Knoop hardness test is applied to evaluate enamel and dentin structures. One of the major difficulties is the requirement of a high polished flat surface that is more time-consuming and more care taking compared to other tests[72, 73].

Hardness tests are important applicability in dentistry. Hardness test can also evaluate the degree of mineralization of a dental substrate and the degree of conversion of composite resin and resin cement using Vickers hardness and Knoop hardness test[72].

The hardness of values of composites has also been considered to provide an indication of their wear resistance properties. However, the complexity of the wear process of composites has caused conflicting reports regarding the correlation between the hardness of a material and its wear resistance[88]. Dental materials textbooks often suggest that a general relationship exists[73]; however, some studies reported that there was no relationship between hardness and abrasion resistance of composite resin[89, 90].

In the development of depth-sensing indentation methodology, which involves the continuous tracking of applied load and indenter's displacement, the elastic properties of the material can also be deduced. This technique relies on the fact that the materials undergo elastic recovery when the indenter is withdrawn from the indented material. With the advancement in technology, many commercially available indentation test systems are capable of measuring load and displacement with superior accuracy and precision. Apart from instrumentation, research has been carried out to improve and refine the indentation methodology so as to make this measurement technique a reliable and accurate to determine the elasto-plastic properties of materials.

Thermocycling

In vitro tests continue to be an indispensable method for the initial screening of dental materials. Thermal cycling is one of the most widely used procedures in simulating the physiological aging experienced by biomaterials in clinical practice. Consequently, it is routinely employed in experimental studies to evaluate material's performance. According to ISO 11405 recommendations (International Standards Organization, 1994) and several studies reported temperatures of 5°C and 55°C to test dental materials, were as the closest to the physiology of the oral cavity[91, 92].

There are still no reports that have been found about the number of thermocycles per unit time representing the duration in vivo. The number of cycles is usually randomly set which makes it difficult to compare published results. Although the International Organization for Standardization, in 1994 considered a protocol of 500 cycles as appropriate in simulating the aging of biomaterials, many study suggested that 500 cycles are a limited number of representing an adequate aging time[93, 94]. Gale and Darvell suggested that approximately 10,000 thermal cycles corresponded to 1 year of physiological aging in the oral cavity[95]. This estimate is based on the hypothesis that such cycles might occur 20 to 50 times a day and it is accepted by many studies[96].

Dwelling time is the period of time that the specimen is immersed in a bath of a particular temperature. It corresponds to a latency period, which is required by the oral capacity to reach its normal temperature again, after consuming hot or cold

food and drink. Unfortunately, the choice of dwelling time in experiment studies appears arbitrary and no effect of dwell time on the results has been clearly established[97]. Several studies indicated approximately dwelling time 60 seconds[98, 99].

Wear characteristics: two- and three-body wear

The term of "wear" refers to the net loss of a material from its surface under operational conditions. The phenomenon is dependent on many different factors occurring almost simultaneously. In the oral cavity, many factors contribute to the wear of enamel and dentin, such as the nature of the occlusal contacts with antagonist teeth (attrition), chewing of food, tooth brushing with tooth paste, inhalation of dust (abrasion), acidic attack due to the consumption of certain fruits and beverages, inhalation of acids or vomiting and regurgitation of gastric juice as in the case of bulimia and anorexia nervosa (erosion)[100].

Prediction of the expected clinical performance of new materials from laboratory tests is highly desirable[101]. The material wear characteristics are best determined though clinical trials; however, clinical trials are expensive and time consuming. Therefore, in vitro wear testing is important to evaluate the new materials before clinical usage[102].

Many articles on in vitro wear of composite resins have been published, using a variety of different wear testing devices to simulate two- and/or three body wear[103-108]. Therefore, the combination of at least two different wear settings is recommended to assess the wear resistance of materials[104]. It is still important to

analyze the mechanisms resulting from two-body wear at direct contact between opposing tooth surfaces (attrition), and from three-body wear (abrasion), occurring when a food bolus is compressed between antagonist teeth and abrading particles of food slide over the restoration surface[109-111]. In vivo, wear conditions are affected by both mechanisms. It is adviced that the wear simulating devices should be designed to simulate both two- and three- body wear. The ISO technical specification 14569-2:2001 "Wear by two- and/or three-body contact" recommends both natural grains such as poppy seed and synthetic material polymethyl methacrylate (PMMA) as abrasive particles.

Different methods are available for the quantification of wear. In one long-used wear testing technique, a component is weighed, placed in service, removed and weighed again, periodically over the life of the part. The weight of material lost is considered a measure of how much the part has been "worn." While this method serves as a general indicator of wear, it unfortunately gives little indication of the wear mechanism or of any change in the component's functionality. Two-dimensional measurement techniques, such as stylus profiling, characterize wear based on a single trace measurement across the sample surface. The most widely specified surface parameter, average roughness (Ra), was developed based on 2D stylus measurements. Ra, however, makes no distinction between peaks and valleys, nor does it provide information about spatial structure, both of which are critical to understand surface performance. Other 2D parameters have been developed which

are sensitive to these types of surface characteristics, but they are still based upon the limited, single slice of the surface. Most recently, 3D surface measurement instruments have led to sophisticate methods for visualizing and quantifying wear. Among these methods is optical profiling, which uses the interference of light to measure surface shape and roughness. This non-contact method can resolve features from micrometer-scale roughness through millimeter-scale step heights, operating at the scales typical of mechanical wear. Among the several methods of determining wear, three-dimentional (3-D) scanning is the most suitable method, since quantitative data may be measured, including volume loss, depth, surface and area of wear[102].

Filler content measurement, filler morphology and element analysis

Filler weight content can be determined by the standard ash method[112]. Microstructural characterization and determination of the material properties are the first steps to understand the behavior of the materials used in restorative dentistry. Scanning electron microscopy (SEM) is a useful tool to provide information topography and microstructure parameters (stereology) such as particle size and shape. When SEM is associated with Energy dispersive spectroscopy (EDS), the information is enhanced by semi-quantitative chemical data of the material. This information is important in the material's properties. The proposed X-ray mapping technique provides a unique method for the dispersion assessment of mineral filled systems and it can be performed on any SEM equipped with an EDX spectrometer

and access to a computer running image analysis software. The method is a useful in addition to the current procedures available for the measurement of dispersion quality and should prove invaluable for the study of filled polymeric systems[113, 114].

The microstructure and properties of composite resin have been extensively studied. As for the filler particles, the SEM has been successfully used to evaluate their numbers, shapes, and sizes[115, 116]. The filler type, shape and amount, as well as the efficient coupling of fillers and resin matrix, contribute to the material properties. Properties such as compressive or flexural strength, hardness and Young's modulus improve as the filler content increases. The investigation of filler technology is particularly important because filler content has been shown by several studies to strongly correlate with the mechanical and physical properties of composite resin[112, 117-119].

Energy dispersive X-ray spectroscopy (EDS) is a powerful analytical technique used for performing qualitative and quantitative elemental analyses of materials by measuring the characteristics of re-emitted X-ray. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are based on the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer.

Color stability

The success of dental restorations depends on mechanical properties, physical properties and optical properties to guarantee the success of the restorations[29]. Furthermore, aesthetics restorative material should mimic the appearance of natural tooth, and this fact is directly related to the material's color match and color stability[31, 32]. Tooth color material restoration should maintain its long-term color stability in order to avoid replacement of restorations. Regardless of their chemical composition, dental resins tend to absorb liquids. Restorations in oral cavity are exposed to several parameters, such as temperature, humidity, food, beverage and smoking habits. There are many factors associated with the discoloration of the dental materials in the oral environment. However, it is known that composite resin restorations have a tendency to discolor when exposed to the oral environment. The color stability of the composite resin is due to exogenous and endogenous reasons[6, 36]. The exogenous reasons include the influence of staining solution such as coffee and red wine. The endogenous reasons include the system of initiator system, duration of polymerization, resin matrix composition, conversion of the matrix monomers, particle size and hardness and oxidation of the unreacted carbon doubles bonds[120-122].

Previous studies have shown that composite resins are susceptible to color instability when exposed to various staining media, especially red wine, coffee, cola, tea and whisky[123, 124]. Moreover, restorative material discoloration might be attributed

to water sorption degree and matrix resin hydrophilicity. If a composite resin can absorb water, it can also absorb other fluids resulting in color alteration. Additionally, filler particle size and distribution as well as resin matrix composition have been shown to play an important role in the composite resin color stability and water sorption degree[113]. Some studies reported high surface roughness of composites, even after finishing, due to irregularly arranged inorganic filler particles, which could result in easier staining over time[125].

Two of the most widely used systems for describing color are the Munsell System and the International Commission on Illumination (CIE) color/order system. The CIE or Commission Internationale De L'Eclairage (translated as the International Commission on Illumination), is the body responsible for international recommendations for photometry and colorimetry. In 1931 the CIE standardized color order systems by specifying the light source (or illuminants), the observer and the methodology used to derive values for describing color[126].

The CIE system is the international standard for color measurements. It incorporates a standard observer and a light source. The widely recognized CIE L*a*b* color order system is commonly used in dental research. In this system, the location of a particular shade in the color space is defined by three coordinates: L*, a*, and b*. The L* describes the lightness of the object being assessed. The a* value defines the color on the red-green axis, and the b* defines the color on the color

yellow-blue axis. The measure of the total color difference between two objects is described by ΔE. It is defined as follows:

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

It was possible to compare the color change after staining immersion treatment by the ΔE parameter of CIEL*a*b* system[6, 126, 127].

Visual color difference thresholds can be used as a quality control tool and guideline for selecting of esthetic materials. The clinical relevance of color change is considered perceptible and acceptable. These factors are subjective and vary among persons. The perceptibility threshold is the magnitude of color difference that is visually detectable by human eye and the acceptability threshold is the magnitude of color difference that constitutes and unacceptable between tooth-color restorative materials[128-130].

According to individual ability of human eye to appreciate differences in colors, three different intervals were used to distinguish changes in color values: $\Delta E < 1$ imperceptible by the human eye; $1.0 < \Delta E < 3.3$ considerate appreciate only for skilled person, both clinically acceptable; $\Delta E > 3.3$ easily observed, these color changes values are not clinically acceptable[131].

In clinical situation, the prospective multi-center study has performed to establish the clinical relevant on color perception of five group observers: dental students, dental auxiliaries, dental technicians and non-dental professionals. This is

to find the difference between perceptibility threshold (PT) and acceptability threshold (AT) among groups of observers and among research sites. This study used dental ceramic in simulated clinical setting. The results found that CIELAB 50:50% PT was ΔE_{ab}=1.2; means that the color difference perceptibility for 50% of observers and 50:50% AT was ΔE_{ab}=2.7; means that the color difference acceptability for 50% of observers [132].

CHAPTER III

MATERIAL AND METHODS

Part I Mechanical properties of composite resin blocks for CAD/CAM

CAD/CAM blocks

The six CAD/CAM block brands, four composite resin blocks (BLO, CER, GRA, ULT), one composite ceramic block (ENA), and one conventional feldspar ceramic block (VIT) as a reference material, are examined in the present study and listed in Table 1. Their characteristics as claimed by the respective manufacturers are shown in Table 2.

Table 1 Materials examined[27]

Type	Brand	Code	Manufacturer	Shade/ Size	Batch
Composite resin	Block HC	BLO	Shofu Inc., Kyoto, Japan	A2-LT/M	111301
	Cerasmart	CER	GC Corporation, Tokyo, Japan	A3 LT/14	1308261E
	Gradia Block	GRA	GC Corporation, Tokyo, Japan	A3/14	1308012
	Lava™ Ultimate	ULT	3M ESPE, St. Paul, MN, USA	A3-HT/ 14L	N494437
Hybrid ceramic	Vita Enamic®	ENA	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	2M2T/ EM-14	37740
Feldspar ceramic	Vitablocs® Mark II	VIT	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	A3C/I14	07BY0803

Code	Composition			Flexural strength	Flexural modulus
	Monomer	Filler			
		Composition	mass%	MPa	GPa
BLO	UDMA, TEGDMA	Silica powder, micro fumed silica, Zirconium silicate	61	191	9.5
CER	Bis-MEPP, UDMA, DMA	Silica (20 nm),	71	231	7.5
GRA	UDMA, methacrylate copolymer	Silica, F-Al-silicate glass, prepolymerized filler	76	208	11.1
ULT	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO_2 (20 nm), ZrO_2 (4-11 nm), aggregated ZrO2/SiO2 cluster (SiO_2 = 20 nm, ZrO_2 = 4-11 nm)	80	204	12.8
ENA	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86	150-160	30
VIT	—	Feldspathic crystalline particles in glassy matrix	—	154	45

Abbr.: Bis-GMA: bisphenol A diglycidylether methacrylate; Bis-MEPP: 2,2-Bis(4-methacryloxypolyethoxyphenyl)propane; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; DMA: dimethacrylate.

Table 2 Composition and properties of CAD/CAM blocks examined as published by manufacturers[27]

Three-point bending test

Flexural properties were determined using a three-point bending test according to ISO 6872: 2008[17]. The bar-shaped specimens, 4.0 mm wide, 14.0 mm long and 1.2 mm thick, were prepared using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). All specimens were wet ground and polished with #600 and #1000 diamond pads (Maruto, Tokyo, Japan) and #1000 diamond sheet (Maruto), mounted on a metallographic lapping machine (Dia-Lap ML-150P, Maruto) to achieve the required dimensions of 4.0±0.2×14.0±0.2×1.2±0.2 mm. To minimize edge failure in the bar specimens during flexural testing, an edge chamfer, 0.15 mm wide, was prepared using the lapping machine with a #1000 diamond sheet. After polishing, all specimens were stored in a desiccator with silica gel for 7 days prior to flexural testing. Thirty specimens of each CAD/CAM block brand were randomly divided into three groups of ten each. Specimens from the first group were kept under dry conditions at room temperature (23±2°C). The second group was stored in 37°C deionized water for 7 days, while the third group was stored in 37°C deionized water for 7 days followed by 10,000 thermal cycles (5°C to 55°C, dwelling time 60 seconds) using a thermocycling machine (HA-K178, Tokyo Giken Inc., Tokyo, Japan). The width and thickness of each specimen were measured with a digital micrometer (MDC-25M, Mitsutoyo Co., Tokyo, Japan; minimum reading: 0.001 mm). A three-point bending test with a support span of 12.0 mm and a crosshead speed of 1.0 mm/min was conducted at room temperature (23±2°C) using a universal testing machine (AG-X,

Shimadzu Corp., Kyoto, Japan). The flexural strength and modulus were calculated using software (TRAPEZIUM X, Shimadzu Corp., Kyoto, Japan). The flexural modulus (*E*) was calculated using the following formula:

$$E=FL^3/4bh^3d$$

where *F* is the load at a convenient point in the straight-line portion of the load/deflection graph, *L* is the span distance (12.0 mm), *b* is the width of the specimen, *h* is the thickness of the specimen, and *d* is the deflection at the load *F.* The flexural strength (σ) was calculated using the following formula:

$$\sigma=3F_1L/2bh^2$$

where F_1 is the maximum load during the flexural test.

Vickers hardness test

Ten fractured specimens after the flexural test from each group were used for the Vickers hardness test.

The surface hardness of the specimens was measured using a micro hardness testing machine (MVK-H2, Akashi, Kawasaki, Japan). A Vickers indenter, with a load of 300 gf was applied for 15 seconds dwell time. Five indentations were made on each specimen and the hardness values were averaged.

Measurement of filler content (by ash method)

To measure the mass percentage of inorganic filler content of the CAD/CAM blocks, the standard ash method was used in accordance with ISO 1172:1996[133]. An

alumina crucible (PC2, Nikkato, Osaka, Japan) was kept in an electric furnace (Auto Furnace QF1, GC Corp., Tokyo, Japan) set at 625°C for 30 minutes. After cooling to ambient temperature in a desiccator, the weight of the crucible was measured using a precision digital balance (AUW120D, Shimadzu, Kyoto, Japan; minimum reading: 0.01 mg) until constant mass was obtained. The specimens of each CAD/CAM block (approximately 1 g, n=3) were placed in the crucible, and the weight of each specimen including the crucible was measured with the precision digital balance. The crucible with the specimen was heated in the electric furnace at 625°C for 30 minutes to burn out the organic matrix. After cooling to ambient temperature in a desiccator, the residue and crucible were reweighed until a constant mass was obtained at two subsequent measurements. The inorganic filler content (mass %) was determined using the following equation:

$$Filler\ content=(m_3-m_1)/(m_2-m_1)\times 100$$

where m_1 is the initial mass of the dry crucible (g); m_2 is the initial mass of the dry crucible plus the dried specimen (g); m_3 is the final mass of the crucible plus the residue after calcination (g).

Filler micro-morphology and element analysis

After the bending test, the broken bar-shaped specimens (4.0×7.0×1.2 mm) were observed using scanning electron microscopy (SEM) to evaluate the filler morphology and distribution. The surfaces of the specimens were wet polished using SiC paper (#600, #1500, #2000, and #4000) and diamond lapping films (3.0 μm and

0.3 μm) and subsequently ultrasonically cleaned with acetone for 5 minutes and with isopropanol for another 5 minutes. The specimens were dried, mounted on aluminum stubs, and sputter-coated with carbon.

The surface of each specimen was examined using a scanning electron microscope (SEM; S-4500, Hitachi High-technologies Corp., Tokyo, Japan) and an energy-dispersive X-ray spectroscope (EDS; EMAX-7000, Horiba, Kyoto, Japan). SEM and EDS analyses were performed on each specimen in three randomly selected areas. EDS analysis for each selected area was carried out under 15 kV acceleration voltage for 100 seconds.

Statistical analysis

The flexural strength, flexural modulus and hardness were separately analyzed with two-way analysis of variance (ANOVA) with type of CAD/CAM block and storage conditions as the main factors, followed by Tukey's post-hoc comparisons. The filler content was analyzed using one-way ANOVA and Tukey's post-hoc comparison. The significance level was set at α=0.05.

Part II *In vitro* evaluation of the wear resistance of composite resin blocks for CAD/CAM

Materials investigated

Table 3 shows the CAD/CAM blocks investigated[134] together with their compositions, as publicly available information from the respective manufacturers. Four composite resin blocks (BLO, CER, GRA and ULT) were evaluated and compared with one hybrid ceramic (ENA) and one feldspar ceramic block material (VIT). The hybrid ceramics is according to the manufacturer "the dominant fine-structure ceramic network strengthened by a polymer network".

Table 3 shows the CAD/CAM blocks investigated[134]

Material	Shade/Size	Type	Code	Manufacturer	Batch	Composition		
						Monomer	Filler	
							Composition	mass%
Block HC	A2-LT/M	Composite resin	BLO	Shofu Inc., Kyoto, Japan	111301	UDMA, TEGDMA	Silica, Silicate, Zr-silicate	61
Cerasmart	A3 LT/14	Composite resin	CER	GC Corp., Tokyo, Japan	1308261E	Bis-MEPP, UDMA, DMA	Silica (20 nm), Ba-glass (300 nm)	71
Gradia Block	A3/14	Composite resin	GRA	GC Corp., Tokyo, Japan	1308012	UDMA, methacrylate copolymer	Silica, F-Al-silicate glass, Prepolymerized filler	76
Lava™ Ultimate	A3-HT/ 14L	Composite resin	ULT	3M ESPE, St. Paul, MN, USA	N494437	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO_2 (20 nm), ZrO_2 (4-11 nm), Aggregated ZrO_2/SiO_2 cluster (SiO_2 = 20 nm, ZrO_2 = 4-11 nm)	80
Vita Enamic®	2M2T/ EM-14	Hybrid ceramic	ENA	Vita Zahnfabrik H. Rauter GmbH&Co.KG, Bad Säckingen, Germany	37740	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86
Vitablocs® Mark II	A3C/I14	Feldspar ceramic	VIT	Vita Zahnfabrik H. Rauter GmbH&Co.KG, Bad Säckingen, Germany	07BY0803	-	Feldspathic crystalline particles in glassy matrix	-

Abbr.: Bis-GMA: bisphenol A diglycidylether methacrylate; Bis-MEPP: 2,2-Bis(4-methacryloxypolyethoxyphenyl)propane; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; DMA: dimethacrylate.

Specimen preparation for wear testing

From each of the 6 block materials 12 cylindrical specimens were prepared (8 mm in diameter and 2 mm in thickness) using a lathe and a low-speed diamond saw (Isomet Buehler, Lake Bluff, IL, USA). The discs were mounted with adhesive resin (Panavia SA Cement, Kuraray, Okayama, Japan) in cylindrical aluminum molds, immersed in deionized water at 37°C for 7 days, and wet-ground on SiC papers, #600, #1,500 and #4,000. Two reference points were created using a diamond round point (#40, Shofu, Kyoto, Japan) on the peripheral area covered by an aluminum container for slurry[106]. Then specimens were ultrasonically cleaned in water for 5 minutes. Six specimens of each material were randomly selected and allocated to testing under two- and three-body wear conditions, respectively.

Wear testing device

The testing machine used was a custom made ball-on-disc sliding machine. The sliding antagonist was a zirconia ball, 4 mm in diameter (YTZ ball, Nikkato Corp., Osaka, Japan) serving as the antagonist "cusp" and loading the mounted CAD/CAM block specimens at 15° angulation along a 3.7 mm sliding path[106]. At the end of each sliding cycle the ball was automatically lifted and returned to the zero position for the following sliding cycle (50 N, 1.2 Hz). A schematic diagram of the designed pathway of the antagonist movement is illustrated in Fig. 1. During testing, the specimens were either immersed in water for simulation of two-body wear or in aqueous slurry of 33 mass% finely pre-ground poppy seed for simulation of three-

body wear. With both testing modes, specimens were subjected to a total of 50,000 sliding cycles. The two reference points above-mentioned were covered during testing. Following each 10,000 cycles, the water and the abrasive poppy seed slurry were renewed, and the zirconia ball surface was microscopically examined for intactness.

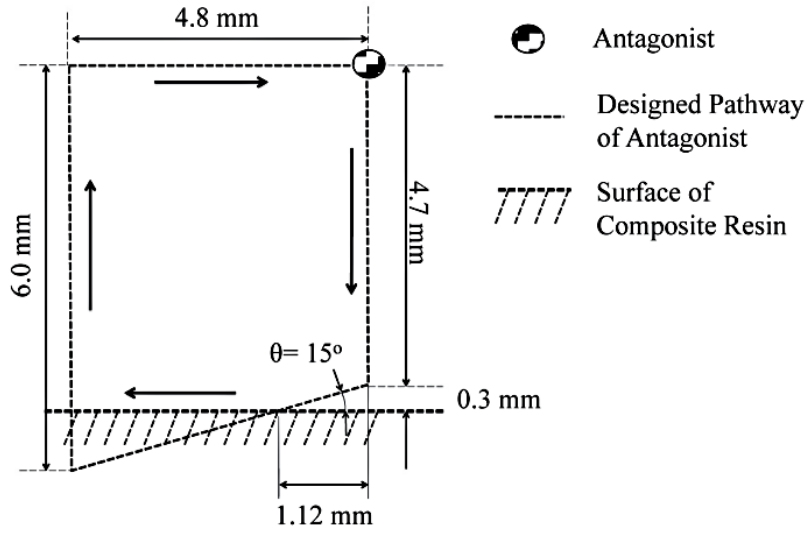

Fig. 1 Schematic diagram of designed pathway of antagonist movement[134]

Determination of wear

After each 10,000 sliding cycles, the specimens were removed from the stage of the wear testing device, rinsed with water and air-dried for inspection of the wear traces produced. The traces were scanned at 5 μm vertical intervals with a digital CCD microscope (VHX-1000 fitted with VH-Z R lens, KEYENCE Corp., Osaka, Japan) at 100-fold magnification. The specimens were carefully horizontally adjusted using three points neighbouring the engraved reference points. Volume loss in mm^3 of the wear trace after manually aligning the reference plane was calculated using software

attached to the CCD microscope. The wear data was statistically analyzed using one-way analysis of variance (ANOVA) and Tukey's HSD *post-hoc* comparisons at a pre-set significance level of α=0.05 (SPSS Statistics, version 21 for Mac, IBM Corp., Cary, USA). Linear regression analyzes for the relationships between volume losses and numbers of sliding cycles were calculated for each block material and each wear mode.

Scanning electron microscopy (SEM)

From each block material and each of the two wear modes one representative specimen with volume loss close to the group average after 50,000 sliding cycles was selected, sputter-coated with gold and inspected by SEM (VE-8800, Keyence Corp., Osaka, Japan) at 10 kV and 3,000-fold magnification. Photographs were taken in the centers of the initial and terminal thirds of the wear traces.

The polished surfaces covered by the aluminum container of each block specimen were also observed using the SEM to evaluate the filler micro-morphology and distribution.

Part III Color differences of CAD/CAM composite resin blocks after immersion in coffee

Materials used

Eight CAD/CAM blocks (five composite resin blocks: BLO, CER, GRA, KZR, ULT, one hybrid ceramic block: ENA, one cross-linked PMMA block: TEL, and one feldspar ceramic block: VIT) and four conventional restorative composite resins (one hybrid: APX, one microfilled: DUR, and two nanofilled: ESQ, FSU) were examined in this study. The materials and their compositions, published by the respective manufacturers are shown in Table 4.

Table 4 Materials investigated

Type	Brand	Code	Manufacturer	Shade/Size	Batch	Composition		
						Monomer	Filler	
							Composition	Mass% (Vol%)
Composite resin block	Block HC	BLO	Shofu Inc., Kyoto, Japan	A3-HT/M	021401	UDMA, TEGDMA	Silica powder, micro fumed silica, zirconium silicate	61.0
	Cerasmart	CER	GC Corporation, Tokyo, Japan	A3-LT/14	1308261E	Bis-MEPP, UDMA, DMA	Silica (20 nm), barium glass (300 nm)	71.0
	Gradia Block	GRA	GC Corporation, Tokyo, Japan	A3/14	1308012	UDMA, methacrylate copolymer	Silica, Al-silicate glass, prepolymerized filler	76.0
	KZR-CAD Hybrid Resin Block	KZR	Yamamoto Precious Metal Co., Osaka, Japan	A3	01021410	UDMA,TEGDMA	SiO_2 (20 nm), aggregated SiO_2-Al_2O_3-ZrO_2 (200-600 nm) cluster (1-6 µm)	74.0
	Lava™ Ultimate	ULT	3M ESPE, St. Paul, MN, USA	A3-HT/ 14L	N494437	Bis-GMA, UDMA, Bis-EMA,TEGDMA	SiO_2 (20 nm), ZrO_2 (4-11 nm), aggregated ZrO_2/SiO_2 cluster (SiO_2 = 20 nm, ZrO_2 = 4-11 nm)	80.0
Hybrid ceramic block	Vita Enamic®	ENA	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	3M2-HT/ EM-14	47630	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86.0 (75.0)
PMMA block	Telio CAD	TEL	Ivoclar Vivadent Inc., Schaan, Liechtenstein	A3	T09738	MMA	-	-
Feldspar ceramic block	Vitablocs® Mark II	VIT	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	A3C/I14	07BY0803	-	Feldspathic crystalline particles in glassy matrix	-
Conventional restorative composite resin	Clearfil AP-X	APX	Kurarey, Okayama, Japan	A3	01447A	Bis-GMA, TEGDMA	Silanated barium glass, Silanated colloidal silica, Silanated silica (0.1-15 µm)	86.0 (70.0)
	Durafill® VS	DUR	Heraeus Kulzer, Hanau, Germany	A3	10218	Bis-GMA, UDMA, TEGDMA	SiO_2 (20-70 nm), prepolymer < 20 µm, SiO_2 in prepolymer: 32 wt%	75.3 (66.0)
	Estelite Sigma Quick	ESQ	Tokuyama Dental Corporation, Tokyo, Japan	A3	151064P	Bis-GMA, TEGDMA	Silica-zirconia filler, composite filler (0.1-0.3 µm)	80.0 (71.0)
	Filtek Supreme Ultra	FSU	3M ESPE, St. Paul, MN, USA	A3	N433373	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO_2 (20 nm), ZrO_2 (4-11 nm), aggregated ZrO_2/SiO_2 cluster (SiO_2 = 20 nm, ZrO_2 = 4-11 nm)	78.5 (63.3)

Discoloration tendency

Twelve disk-shaped specimens, 10 mm in diameter and 2.1 mm in thickness, were produced from each material. CAD/CAM block specimens were prepared using a stone abrasive wheel (Vitrified Dia, Shofu Inc., Kyoto, Japan) attached to a lathe (YS-550V, Towa seiki Co., Ltd., Aichi, Japan) to make cylindrical specimens from which disk-shaped specimens were cut with a low-speed diamond saw (Isomet, Buehler, Illinois, USA). The conventional composite resin specimens were prepared in cylindrical acrylic molds (10 mm in diameter and 2.1 mm in thickness) and activated for 40 seconds on each side using a light-emitting diode (LED) curing unit (Elipar™ S10, 3M ESPE, Seefeld, Germany) with a light intensity of 600 mW/cm^2. The specimens were ground sequentially on wet SiC paper (grit #600, #1,000 and #1,500). Grinding and polishing were performed on one side of the samples to produce 2.0±0.1 mm thick specimens. The thickness was controlled with a digital micrometer (MCM-25M, Mitsutoyo, Tokyo, Japan), with a reading accuracy of ±0.001 mm. The specimens were ultrasonically cleaned in deionized water for 10 minutes, shortly dried with compressed air, immersed in deionized water in a brown glass vial and kept in an incubator (TVN480DA, Toyo Seisakusyo Kaisha, Co., Ltd, Chiba, Japan) at 37±0.2°C for 24 hours. Subsequently, the specimens were removed from the vials, washed with deionized water, gently wiped with filter paper, and dried by shaking in air for 30 seconds prior to the colorimetric measurements.

The CIE *L*a*b** values of the polished surfaces of each specimen were measured and placed on a black background (L^* = 1.50, a^* = −2.37, and b^* = −8.41) using a spectrophotometer (Crystaleye, Olympus Corp., Tokyo, Japan). The light source illumination corresponded to average daylight (D65).

Deionized water and coffee were used as immersion media. The coffee was prepared by dissolving 0.51 g of instant coffee powder (Nescafe Gold Blend, Nestle Japan, Kobe, Japan) in 50 ml of deionized water[37, 43]. Six specimens of each CAD/CAM block were immersed in each of the immersion solutions placed in a shaking incubator (37±0.2°C, 1 Hz frequency). The solutions were changed every day. Color measurements were done after one day, one week and one month of immersion.

The color difference (ΔE) was calculated based on the *L**, *a** and *b** on black background between the specimen values before and after immersion in one of the solutions using the following equation:

$$\Delta E = [(L^*_1 - L^*_{2)}{}^2 + (a^*_1 - a^*_{2)}{}^2 + (b^*_1 - b^*_{2)}{}^2]^{1/2}$$

The subscripts 1 and 2 refer to the color coordinates before and after immersion, respectively. A high ΔE value indicates a large color difference. Three measurements were made on each specimen, and the average value was recorded.

After 6 months storage in a desiccator all specimens subjected to 1-month immersion in coffee were polished with a prophylaxis paste (Proxyt fluoride-free, RDA 36; Ivoclar Vivadent, Schaan, Liechtenstein) applied with a soft rubber cup at 5,000

rpm for each 20 seconds on the plane surfaces and on the peripheral surface, and the color was measured again.

Statistical analyses

The ΔE was analyzed by two-way analysis of variance (ANOVA) with immersion media and immersion periods as main factors, followed by Tukey's post-hoc comparisons. The differences between ΔE of 1-month coffee and after polishing were analyzed by paired t test. The significance level was set at α=0.05.

CHAPTER IV

RESULTS

Part I Mechanical properties of composite resin blocks for CAD/CAM

Results of flexural strength, flexural modulus, Vickers hardness, and inorganic filler content are summarized in Table 5.

	Condition	BLO	CER	GRA	ULT	ENA	VIT	sig
Flexural strength	Dry	170.5 (10.5)	242.0 (11.6)	204.0 (20.2)	170.5 (28.7)	140.7 (8.5)	126.6 (8.1)	
(MPa)		c,d	f	e	c,d	b	a,b	
	Water	121.5 (14.6)	197.3 (10.8)	188.4 (9.3)	141.9 (14.5)	133.0 (10.3)	121.1 (15.2)	
		a,b	e	d,e	b	a,b	a,b	
	Water/TC	117.6 (13.9)	194.3 (14.9)	165.1 (12.0)	120.1 (15.6)	134.6 (6.7)	129.0 (5.7)	
		a	e	c	a,b	a,b	a,b	
Flexural modulus	Dry	9.6 (0.4)	10.0 (0.2)	14.7 (0.3)	14.5 (0.3)	28.5 (1.1)	51.5 (3.1)	
(GPa)		b,c	c	e	e	f	g	
	Water	7.8 (0.3)	9.0 (0.2)	13.5 (0.4)	12.8 (0.1)	28.3 (0.8)	52.8 (4.1)	
		a,b	a,b,c	d,e	d,e	f	g,h	
	Water/TC	7.2 (0.6)	8.7 (0.3)	13.2 (0.4)	12.2 (0.3)	28.6 (0.8)	54.9 (1.0)	
		a	a,b,c	d,e	d	f	h	
Vickers hardness*	Dry	65.3 (2.4)	64.1 (1.7)	97.3 (1.9)	97.9 (1.7)	189.8 (7.8)	454.8 (21.4)	C
	Water	58.9 (1.6)	59.0 (1.2)	87.5 (2.7)	86.7 (1.5)	184.0 (5.9)	453.8 (7.7)	B
	Water/TC	57.4 (1.5)	58.0 (0.7)	80.3 (3.1)	83.0 (1.0)	177.1 (5.2)	448.5 (19.4)	A
	sig	a	a	b	b	c	d	
Filler content		61.9 (0.1)	65.0 (0.1)	70.5 (0.1)	73.1 (0.2)	86.4 (0.0)	99.8 (0.0)	
(mass%)	sig	a	b	c	d	e	f	

Mean values (n=10) and standard deviations in parentheses.

Dry: Dry storage; Water: 7-day 37°C deionized water storage; Water/TC: 7-day 37°C deionized water storage before thermocycling.

For each mechanical property, values denoted with same letters are not significantly different ($p>0.05$).

*Only Vickers hardness did not show significant interaction, but two main factors were significant. Therefore Vickers hardness, pooled data of all conditions and all materials; same letters are not significantly different ($p>0.05$).

Table 5 Summary of results[27]

The flexural strength determined after dry storage ranged from 126.6 to 242.0 MPa. Flexural strength was lower after 7-day water storage and thermocycling. Two-way ANOVA suggested that the two main factors (type of CAD/CAM block and storage conditions) and their interaction were significant. The flexural strength of BLO, CER and ULT was significantly decreased after 7-day water storage when compared with dry storage, but not after water storage and thermocycling. The flexural strength of GRA did not change significantly after 7-day water storage but was significantly lower after thermocycling. ENA and VIT specimens showed no significant change in flexural strength after each of the storage modes tested.

The flexural modulus under dry conditions ranged from 9.6 to 51.5 GPa. These values changed slightly after 7-day water storage and thermocycling. Two-way ANOVA suggested that the two main factors and their interaction were significant. The flexural modulus of BLO and ULT did not change after 7-day water storage but significantly decreased after thermocycling, whereas the flexural modulus of GRA, CER, and ENA did not change. The flexural modulus of VIT was significantly greater after thermocycling than after dry storage.

The hardness of specimens kept under dry condition ranged from 64.1 to 454.8. These values decreased after 7-day water storage and thermocycling. Two-way ANOVA suggested that the two main factors were significant, but not their interaction. The hardness of VIT was significantly greater than any of the other materials tested. The ranking of hardness figures was as follows: VIT>ENA>ULT≥GRA>BLO≥CER.

Hardness after the three storage conditions was significantly different as follows: dry condition>7-day water storage>thermocycling.

The inorganic filler content was significantly different among all materials tested: VIT>ENA>ULT>GRA>CER>BLO.

SEM images of the CAD/CAM blocks are shown in Fig. 2.

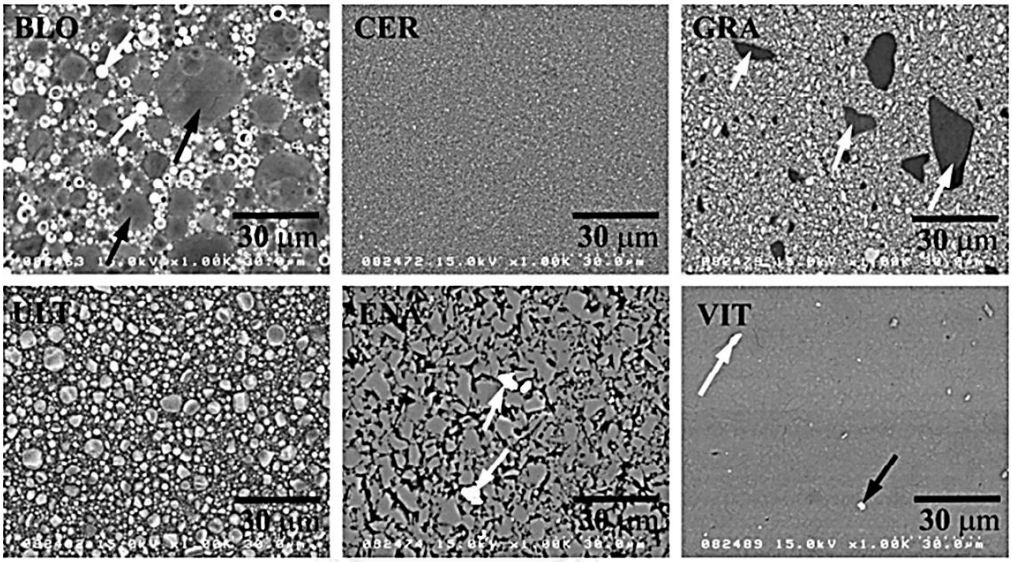

Fig. 2 SEM images of CAD/CAM blocks (1k original magnification). BLO: small white spherical particles (white arrow) of zirconium silicate and large spherical particles (black arrow) of silica; CER: uniform small particles of alumina-barium-silicate; GRA: large irregular shaped particles (white arrow) of silica and small irregularly shaped particles of potassium-alumina-silicate; ULT: wide size range of zirconia-silicate fillers; ENA: dense network structure of potassium-alumina-silicate, small white particles of yttrium-silicate (white arrow); VIT: network-like structure of potassium-alumina-silicate, small

white particles of yttrium-silicate (white arrow) and zirconia-silicate (black arrow)[27].

BLO exhibited two types of spherical particles, composed of silica and zirconium silicate. CER contained relatively small and uniformly distributed particles of alumina-barium-silicate. GRA contained two distinct particle types: relatively large irregularly shaped particles consisting mainly of silica, and relatively small irregularly shaped fillers consisting of potassium-alumina-silicate. ULT exhibited a wide range of zirconia-silicate particle sizes. ENA was characterized by a dense network structure mainly consisting of potassium-alumina-silicate and small particles of yttrium-silicate. VIT exhibited a dense network-like structure mainly consisting of potassium-alumina-silicate, small particles of yttrium-silicate and a small component of zirconia-silicate and yttrium-silicate particles probably representing coloring agents.

Part II *In vitro* evaluation of the wear resistance of composite resin blocks for CAD/CAM

Figure 3 displays the average volume losses and related 95% confidence intervals of the tested materials by wear modes after 50,000 sliding cycles. The same letters for block materials and wear modes denote groups that are not significantly different ($p \geq 0.05$). When tested under water, the three composite resin blocks BLO, CER and GRA showed low volume losses, whereas the composite resin ULT and the two reference materials VIT and ENA in this order were significantly worn more. When exposed to the abrasive poppy seed slurry, all six CAD/CAM blocks revealed similar small volume loss that was not significantly different from the results obtained with BLO, CER and GRA under two-body wear condition.

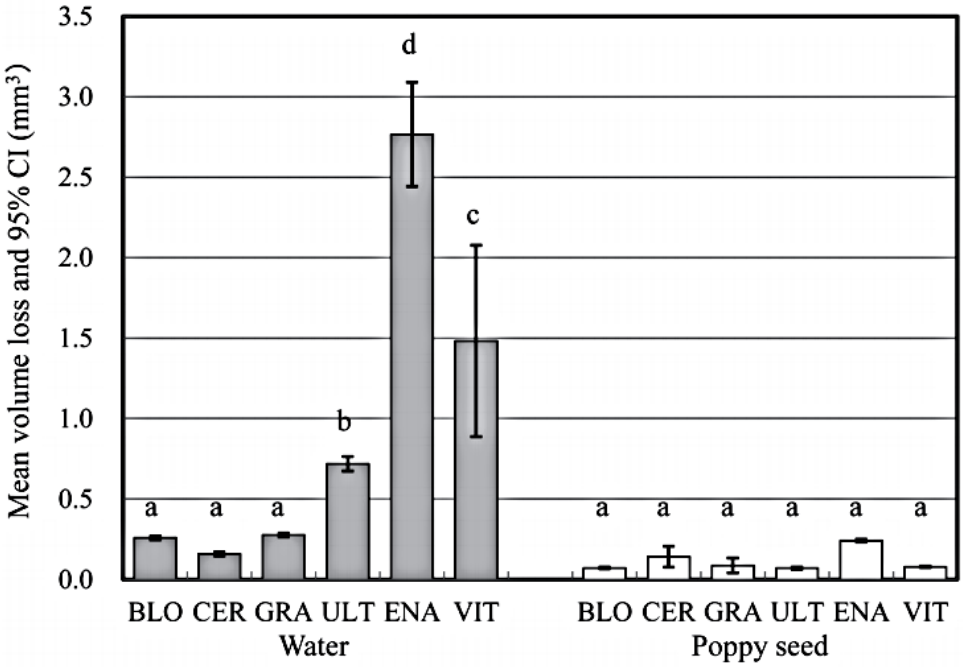

Fig. 3 Mean wear in terms of volume loss after 50 k sliding cycles in water (shaded bars) and in poppy seed (open bars). Whiskers represent the 95% intervals of confidence. One-way ANOVA: $p<0.001$; Tukey HSD: $p<0.05$. Same lower case letters denote groups that are not significantly different[134].

Table 6 summarizes the results of linear regression analyzes by slopes of the regression lines for the relationships between volume losses and numbers of sliding cycles by material and wear mode. The regression lines were forced through zero and all relationships were significant ($p<0.01$). The coefficients of determination (R^2) were equal or larger than 0.87 in 11 of the 12 cases, only the R^2 for the hybrid ceramic block material ENA after two-body wearing was lower.

Materials	Water		Poppy seed	
	slope	R^2	slope	R^2
BLO	0.0541	0.953	0.0144	0.982
CER	0.0295	0.975	0.0238	0.871
GRA	0.0588	0.970	0.0159	0.957
ULT	0.1496	0.976	0.0145	0.959
ENA	0.6167	0.711	0.0495	0.955
VIT	0.3086	0.871	0.0159	0.967

Coefficients of determination (R^2). All relationships were statistically significant ($p<0.01$).

Table 6 Slopes of linear regression lines forced through zero for relationships between volume loss in water and poppy seed slurry, respectively, and number of sliding cycles[134].

The contours of the wear traces were characteristically different for the two wear modes evaluated. In case of two-body wearing the traces were pear-shaped, i.e. starting with a tapered form in sliding direction and ending in spherical shape, or ellipsoid (Fig. 4a). Under three-body wearing the traces produced were half dumbbell-shaped, i.e. starting with a circular shape in sliding direction and continuing in bar shape, or parallel bar-shaped (Fig. 4b).

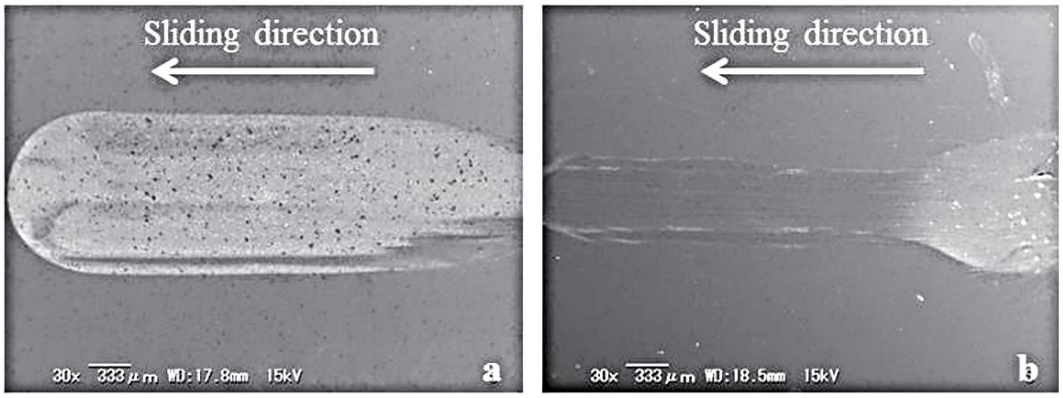

Fig. 4 Representative SEM images (30× magnification) of two- and three- body wear traces. a: GRA after wear under water, b: CER after wear under poppy seed slurry[134].

The SEM images at 3,000-fold magnification of the polished surface and after wear testing of the specimen are illustrated in Fig. 5. The sliding directions were from right to left. BLO showed two types of spherical fillers namely, zirconium silicate and silica, respectively[6)] (Fig. 5a). CER contained small and uniformly distributed fillers (Fig. 5f). GRA contained two distinct filler types: relatively large irregularly shaped fillers and relatively small irregular shaped fillers (Fig. 5k). ULT showed a wide range of filler particles (Fig. 5p). ENA was characterized by dense network structure (flat bright areas in Fig. 5u) and small filler particles. VIT exhibited a dense network-like structure and small particles (Fig. 5z).

Under two-body wear (attrition), worn BLO surfaces both at terminal and initial thirds were similar to the polished surface before wear testing (Figs. 5a, b, c). The large circular silica particles were abraded to the same level as the surrounding smaller diameter fillers in both the initial and the terminal trace thirds. However, in

the terminal sector gaps and small fractured areas were displayed around some of the circular elements. In contrast, when worn with poppy seed slurry under three-body wear (abrasion) in the initial part of the trace shallow grinding grooves were seen in direction of the sliding movement and some of the minor spherical fillers were partly fractured or exfoliated (Fig. 5e). The terminal third of the wear trace was characterized by loosened filler particles and concave holes after debonding of smaller spherical particles (Fig. 5d).

The composite resin CER showed under attrition both in the initial and terminal third of the wear trace alternating dark and bright areas. The dark zones were apparently compressed zones, whereas the bright ones revealed the structural morphology of the composite after surface delamination (Figs. 5g, h). In the abrasion mode, the CER surface appeared rather grainy with many small protruding particles, others were dug out from the surface (Figs. 5i, j). In the terminal sector uniformly worn areas dominated with many small pinholes and some larger seemingly delaminated areas (Fig. 5i).

The morphological features of GRA were very different according to the wear modes. When worn in the attrition mode rather large irregularly shaped, smoothly worn particles with defects left after quarrying out of minor parts were seen both in the initial and the terminal third of the track (Figs. 5l, m). Areas with smaller irregular-shaped fillers surrounded the large particles. Some of these smaller particles slightly protruded from the surface, others were plucked out from the underlying bulk of the

material. When abraded in three-body wear mode, in the initial third almost parallel patterns with compressed composite material were discernable from deeper grooves with degraded material (Fig. 5o). The terminal third of the wear trace featured mainly compressed dark areas next to numerous surface defects (Fig. 5n).

Under attrition the composite resin block material ULT revealed in the initial third of the trace almost circular filler particles of varying size that protruded from the surface reminding of a faint relief polish (Fig. 5r). Only occasionally small craters, left after exfoliated fillers, were detected. The appearance of the trace in the terminal third was strikingly different (Fig. 5q). Filler elements protruded from the adjacencies, some were loosely bonded to the matrix, others were removed leaving surface defects behind. In contrast, when worn under the third-body slurry at both inspected sites of the trace the morphology of the surface was predominantly smooth with rarely seen defects apart from occasional minute gaps at the interfaces between fillers and matrix (Figs. 5s, t). In the initial part grooves in sliding direction were readily discernable.

When subjected to attrition wear the hybrid ceramic material ENA exhibited the structure-sintered ceramic matrix with the polymer filled interstices as heavily destructed surface (Figs. 5v, w). The degraded ceramic matrix was the dominating appearance, both in the initial and the terminal third of the wear trace. Although a similar destruction pattern was recognizable after wearing with the abrasive slurry (Figs. 5x, y), the morphology of the deteriorated surface was less pronounced. In the

terminal section of the trace, a long crack typical for brittle materials was visible in sliding direction (Fig. 5x).

SEM investigation of the feldspar ceramic VIT showed similar surface destruction as ENA, both after two- and three-body wearing (Figs. 5α, β, γ, δ). The glassy matrix was heavily disrupted. Fractured parts of the ceramic framework and craters left after breaking off were distinguishable on the surface.

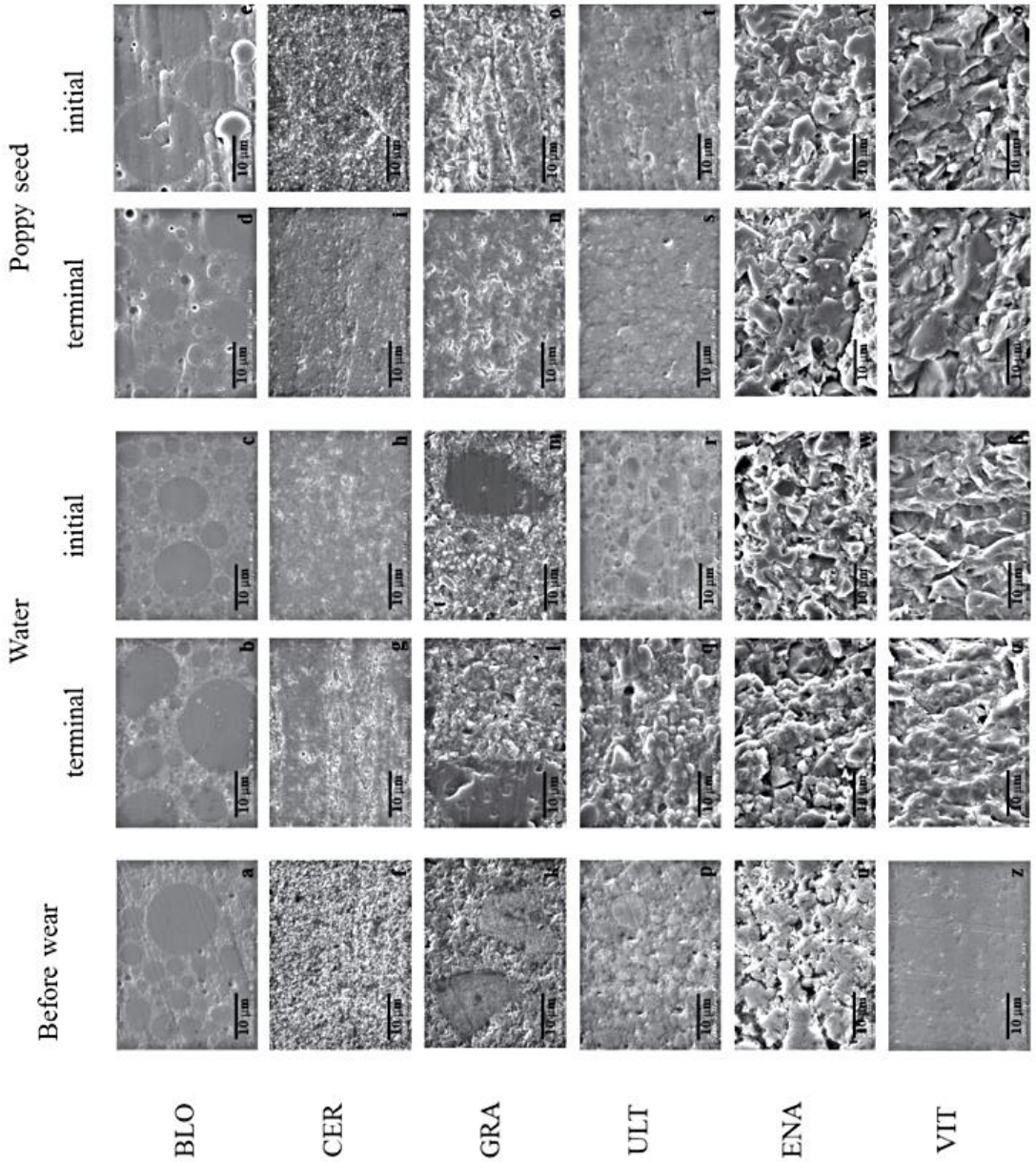

Fig. 5 Representative SEM images (3k magnification) of the six materials investigated before wear testing and after 50 k sliding cycles in water (attrition) and poppy seed slurry (abrasion). The ball sliding direction was from right to left. Right columns show the morphologies from the central part of the initial third, left columns from the central part of the terminal third of the wear traces[134].

Part III Color stability of CAD/CAM composite resin blocks after immersion in coffee

After immersion in coffee the specimens generally became darker in color as the immersion period increased. Color changes of the specimens after water immersion were not clearly detected.

Table 7 shows the ΔE values and their standard deviations after immersion in the two different media and after polishing with prophylaxis paste. Color changes measured on black and white backgrounds demonstrated a similar trend (data on white background are not shown). Therefore, only ΔE values measured on the black background are presented.

Table 7 ΔE after immersion in deionized water and coffee solution for 1 day, 1 week, and 1 month and after polishing 1-month coffee immersion

Material type	Code		Water			Coffee			
			1 day	1 week	1 month	1 day	1 week	1 month	after polishing
Composite resin block	BLO	ΔE	0.2 (0.1) ^{a}	0.3 (0.2) ^{a}	0.3 (0.1) ^{a}	1.3 (0.5) ^{b}	2.2 (0.5) ^{c}	3.7 (0.7) ^{d}	1.8 (0.5)
	CER	ΔE	0.3 (0.3) ^{d}	0.4 (0.2) ^{e}	0.7 (0.2) ^{f}	1.2 (0.4) $^{f, g}$	1.9 (0.7) ^{g}	2.7 (0.6) ^{h}	0.9 (0.4)
	GRA	ΔE	0.3 (0.2) ^{i}	0.3 (0.2) ^{i}	0.4 (0.2) ^{i}	0.5 (0.3) $^{i, j}$	0.9 (0.2) ^{j}	1.6 (0.4) ^{k}	**1.6 (0.5)**
	KZR	ΔE	0.2 (0.1) ^{l}	0.3 (0.2) ^{l}	0.3 (0.1) ^{l}	1.0 (0.3) ^{m}	1.6 (0.3) ^{n}	2.9 (0.3) ^{o}	0.9 (0.2)
	ULT	ΔE	0.7 (0.4) ^{p}	0.6 (0.3) ^{p}	0.5 (0.3) ^{p}	1.7 (0.3) ^{q}	2.7 (0.4) ^{r}	3.6 (0.6) ^{s}	1.2 (0.4)
Hybrid ceramic block	ENA	$\Delta E^{\#}$	0.3 (0.2)	0.4 (0.2)	0.5 (0.4)	0.5 (0.3)	0.8 (0.4)	1.4 (0.4)	0.5 (0.2)
Cross-linked PMMA	TEL	$\Delta E^{\$}$	0.9 (0.6)	0.9 (0.8)	1.0 (0.8)	0.9 (0.7)	1.6 (1.2)	2.0 (1.1)	1.2 (0.9)
Feldspar ceramic block	VIT	ΔE	0.3 (0.2) ^{t}	0.3 (0.1) ^{t}	0.3 (0.2) ^{t}	0.4 (0.2) ^{t}	1.1 (0.2) ^{u}	1.8 (0.3) ^{v}	0.3 (0.1)
Conventional	APX	ΔE	0.9 (0.4) ^{w}	0.8 (0.4) ^{w}	0.8 (0.7) ^{w}	1.4 (0.2) $^{w, x}$	2.1 (0.3) ^{y}	2.1 (0.2) ^{z}	0.5 (0.2)
restorative	DUR	ΔE	0.5 (0.2) ^{A}	0.7 (0.2) ^{A}	0.9 (0.3) ^{A}	1.6 (0.6) ^{A}	4.1 (1.2) ^{B}	7.9 (1.5) ^{C}	5.7 (1.4)
composite resin	ESQ	ΔE	0.3 (0.2) $^{D, E}$	0.2 (0.1) $^{E, F}$	0.9 (0.1) ^{D}	0.8 (0.3) ^{G}	1.6 (0.4) ^{H}	2.4 (0.5) ^{I}	0.7 (0.3)
	FSU	ΔE	0.3 (0.1) ^{J}	0.6 (0.2) ^{J}	1.3 (0.1) $^{J, K}$	2.3 (0.7) ^{K}	3.7 (1.0) ^{L}	6.6 (1.0) ^{M}	4.4 (0.9)

Standard deviations are in parentheses and the same superscript letter denotes homogenous subsets in each material (p>0.05)

$^{\#}$ Two main factors were significant; 1 day = 1 week < 1 month, water < coffee

$^{\$}$ Not significantly different

After immersion in coffee, most changes occurred with a decrease in *L** values and increase in *a** and *b** values, therefore the specimens become more toward red and yellow and darker in color, but such an increase in *a** and *b** values was minimal. Two-way ANOVA of ΔE values revealed that the two main factors, immersion medium and immersion period, and their interaction were significant, except for ENA and TEL. Generally, the ΔE values after immersion in water did not change significantly, whereas those after immersion in coffee were significantly increased with increase of immersion time. For ENA, the two main factors were significant, but the interaction was not. The ΔE values after immersion in coffee were significantly larger than those after immersion in water, ΔE values after one month were significantly larger than those after one day and one week. Regarding TEL, two-way ANOVA suggested no significant factor.

After polishing with the prophylaxis paste for removal of the discoloration almost all products tested showed significantly reduced ΔE values. Only, the resin composites DUR, FSU presented perceptibly high discoloration (ΔE 4.2 – 5.7) after polishing.

CHAPTER V

DISCUSSION

Part I Mechanical properties of composite resin blocks for CAD/CAM

Four composite resin blocks and one composite ceramic block were selected and compared with a feldspar ceramic block.

According to the SEM observations, the inorganic filler content and the EDS analysis, three types of structure were observed: resin matrix structure with filler (BLO, CER, GRA, and ULT), ceramic network structure with resin matrix (ENA), and ceramic structure (VIT).

Several mechanical properties may be considered for restorative material evaluation. The flexural test used in this study followed the well-established and widely accepted test method described in the ISO standard[17, 135]. Three-point bending and Vickers hardness were selected as relatively simple and reliable test procedures.

The geometry of the three-point bending test specimens for restorative composite resins (ISO 4049:2009)[135] and ceramics (ISO 6872:2008)[17] is not identical. Because of the limitation of the CAD/CAM block size, the geometry for ceramics was selected in this study. The flexural properties obtained from the two standard test methods were reportedly not identical but provided similar results[136].

Statistical analysis of the examined properties showed significant differences among the CAD/CAM blocks tested. Therefore, the null hypothesis that there was no difference in characteristics among the CAD/CAM blocks was rejected.

The results of flexural strength and modulus after dry storage and the inorganic filler contents were similar to the values claimed by the manufacturers. The flexural strength of CAD/CAM composite resin blocks was higher than that of recently developed nanofilled composite resins[19]. The flexural modulus results of GRA and ULT were similar to that of dentin, 17.7–29.8 GPa[137]. In contrast, VIT rated a similar flexural modulus to enamel, 72.7–105.5 GPa[138, 139]. Seven-day water storage and thermocycling affected the flexural strength and flexural modulus of CAD/CAM composite resin blocks, but not those of ENA and VIT. Immersion in water caused water penetration into the resin matrix of the composite resin blocks, softening the polymer[21]. Moreover, it may be hypothesized that the absorbed water would cause hydrolysis of the interfacial silane coupling agent, especially in case of zirconia silicate that is not effectively silanized due to the high crystalline content[140]. Accordingly, the flexural strength and modulus of zirconia-containing composite resin blocks, especially BLO and ULT, decreased after 7-day water storage and thermocycling. However, ENA and VIT with their ceramic network structure did not absorb water. Future studies regarding cyclic loading and long-term storage are recommended for CAD/CAM composite resin blocks.

The surface hardness of a material is a relative measure of resistance to an external indentation force. Indentation hardness has been considered a predictor of the wear resistance of a material[88]. In several studies investigators tried to find a relationship between the hardness and wear resistance of composite resins. However, the results were ambiguous[103, 141]. Results of the two-way ANOVA of hardness values suggested that the type of material and storage conditions were significant. The changes in composite resin blocks after 7-day water storage and thermocycling could be attributed to water absorption in the matrix resin causing swelling of the network and reduction in the frictional forces between polymer chains[21]. However, ENA and VIT were only slightly affected by 7-day water storage and thermocycling due to their ceramic network structure.

The flexural properties of composite resin blocks tested in this study were comparable to those of a ceramic block, but still far inferior to the reported value of lithium disilicate glass ceramic and densely sintered yttrium-stabilized zirconia for CAD/CAM[142]. The results of this present study suggest that restorations fabricated from the CAD/CAM composite resin blocks investigated are suitable when limited to single premolar crowns, but not for fixed partial prostheses as recommended in ISO 6872:2008[17].

Part II *In vitro* evaluation of the wear resistance of composite resin blocks for CAD/CAM

The null hypotheses that there would be no difference in volume loss due to wearing and no difference in morphologic appearance of the worn block surfaces have to be rejected.

Wear is defined as a progressive loss of substance resulting from mechanical interaction between two contacting surfaces, which are in relative motion[143]. The intraoral tribosystem is highly complex and, thus, very difficult to simulate with laboratory testing devices[101]. Many different *in vitro* testing machines have been proposed in literature[144, 145], however reasonable correlations with *in vivo* wear data are seldom reported[146, 147]. Thus, most laboratory test methods are primarily useful to categorize the tested materials in terms of product rankings[146]. Wear testing of dental restorative materials should always comprise evaluation of two- and three-body wear[103, 145]. Especially, with three-body wear testing selection of a suitable third-body medium is of utmost importance. The ISO technical specification 14569-2: 2001 “Wear by two- and/or three-body contact”[148] suggests use of natural grains such as poppy seed and synthetic material polymethylmethacrylate (PMMA) as abrasive particles. When comparing laboratory wear data acquired with the Oregon Health Science University oral wear simulator, operated with a mixture of 10% PMMA and poppy seed, a good correlation with clinical data was obtained[147].

There is still no consent which kind and type of abrader is most suitable as artificial cusp used in wear machines. Instead of human enamel abraders that are difficult to standardize due to their anisotropy and brittleness, in this study a zirconia ball was used recognizing that zirconia is harder than enamel and much harder than the often used steatite balls[145]. The 50 N force on the ball was selected as it represents the mean physiological biting force[149] and the sliding frequency of 1.2 Hz simulates the average chewing frequency.

The ball-on-disc sliding machine was used both in the attrition mode, i.e. with water as intermediate substance, to simulate attrition wear (occlusal contact area, OCA), and in abrasion mode, i.e. with an abrasive third body as artificial food to simulate abrasive wear as occurring in contact free areas (CFA). Attrition is caused under opposing tooth contact, which is considered a localized process mainly related to local microfracture. On the other hand, wear in the CFA area is caused under sliding motion of the opposing cusp transmitting force through the food bolus, the third-body medium.

When comparing the volume loss under water after 50 k cycles from a previous publication where light-cured composite resins (hybrid and nanofiller-type) were investigated[107] with the present data from the composite resin block materials the volume loss of the manufacturer-produced blocks is considerably lower. The higher wear resistance is supposedly related to the high-pressure and high-temperature polymerization during the manufacturing process[5]. The volume losses of

ENA and VIT worn under water were not similar to hybrid and nanofilled composite resins[107]. In contrast, it is interesting to note that volume losses after 50 k cycles of sliding wear with poppy seed as the intermediate medium were very similar for the light-cured composite resins investigated in a previous study[107] and the composite resin blocks.

As in the previous trial, stringent linear relationships were found for the relationships between volume loss and number of sliding cycles. During clinical service, one might presumably find decreasing wear with increasing numbers of chewing cycles once the attrition and/or abrasion wear pathway is established. The explanation for this apparent discrepancy might be that in the present laboratory trial the force acting on the specimen is constantly 50 N whereas the acting force during clinical service decreases with time. Therefore, the present wear data might overemphasize the extent of wear expected from clinical trials.

Examination of the wear patterns produced under attrition and abrasion might offer valuable clues to understanding of the underlying wear mechanisms. The wear trace of initial thirds represents the impact effect followed by compression zone and tension zone ahead and behind the antagonist traveling along the trace path, respectively. The terminal thirds also clearly represent the effect of tension zone while the antagonist leaves the surface.

With BLO, containing a relatively low filler loading of 61 mass%, the antagonist ball had presumably compressed the composite structure due to support

of the load on the relatively large spherical fillers. Minor fillers were debonded, mainly in the terminal third of the sliding trace, where they had lost grip in the polymer surroundings due to loss of substance. Gaps around larger particles were mostly found at the filler sites opposite to the sliding direction and indicated that debonding had occurred due to a zone of tension behind the compression front ahead of the sliding motion. Dissipation of energy produces cracks, preferably at the weakest locations, i.e. at the interface between filler and polymer. Under three-body wear due to the scratching action of the poppy seed particles, BLO showed very shallow scratching of the fillers and local fracturing of filler particles in the initial third of the trace. The terminal third displayed holes left after filler debonding and interface cracks between fillers and matrix polymer.

The filler loading of CER is 71 mass% and the filler particles are very small and with rather narrow size distribution. After two-body wear the surface pattern was very smooth, characterized by compressed areas and delaminated zones. The delaminated zone was characterized as the loss of a very superficial deformed layer after sub-surface micro-crack formation. Delamination as an expression of fatigue related wear was apparently enhanced in the terminal third of the wear trace. Under the sliding process the antagonist ball was supported by the minute filler particles protruding from the surface. The fillers were therefore exposed to relatively large stress and were likely to be dislodged or exfoliated. The delamination areas seen were presumably caused by surface or subsurface microcracks induced during plastic

deformation of the material. Upon coalescence of such microcracks major cracks are produced under the surface that finally may result in the localized loss of the surface, i.e. delamination. The delaminated zone was the loss of very superficial deformed layer after micro-cracked formation[105, 150]. The morphology of the CER surface after three-body wear was to some extent comparable to the one after two-body wear. However, in the initial third of the trace a more grainy structure was displayed with many loosened particles. The local pressure applied through the antagonist was less pronounced due to the intermediate poppy seed slurry. The crushed particles of the seeds scratch and wear preferentially the inter-particle polymer. This phenomenon may be explained by the filler protection theory[151]. The terminal third of the trace showed rather smooth zones next to deep scratches in sliding ball direction and some delaminated areas.

Under attrition wear the surface of GRA shows smoothly abraded large prepolymerized silica particles surrounded by the matrix polymer loaded with a much finer filler fraction. Many of the small particles were loosened or exfoliated, presumably due to dislodgement under the high local stress exerted by the sliding ball. In the terminal third of the wear trace, the prepolymerized particles showed multiple local fractures, probably created by loose smaller fillers which were carried ahead of the sliding ball. After three-body sliding wear the morphology of GRA was very different. Local delamination next to compressed areas was identified, especially in the initial third of the trace where grooves along the sliding pathway

were clearly visible. On the contrary, in the terminal segment of the trace compressed smooth areas interrupted by numerous small delamination zones were the dominating appearances.

ULT is a nano-ceramic material with high filler loading. Under two-body wear the initial part of the trace, where the antagonist has touched the surface at 15 degrees angulation, smoothly abraded zirconia-silica clusters of varying size were shown. The matrix between the fillers contains small clusters and according to manufacturer information discrete nano-fillers. Seemingly, several clusters were loosened from the matrix. As seen in the terminal third of the trace many cluster particles were exfoliated from the surroundings during the sliding movement of the antagonist and pushed in front of the zirconia ball along the sliding path. As a result, major surface disruption or erosion had occurred, as also demonstrated by the almost three times higher volume loss of this material, when compared with the other composite resin blocks. The appearance of ULT after three-body wearing was dramatically different. The poppy seed fragments had created fine parallel grinding scratches in direction of the antagonist's sliding path without creating major surface defects. The third part of the trace showed a very smoothly abraded surface, however many of the clusters showed debonding at the interface, which is the mechanically most vulnerable part of the composite system.

The hybrid ceramic material ENA is a polymer-infiltrated feldspar ceramic network enriched with aluminum oxide. Wear in the attrition mode had resulted in

severe surface fracturing of the ceramic network. The surface morphology showed heavy destruction both in the initial and the terminal third of the traces. In the terminal section of the wear trace, multiple fractured elements were still covered with remnants of the matrix polymer. The microscopic appearance is in good agreement with the high volume loss after 50 k sliding cycles, which is almost 10 times higher than the average volume loss recorded for the three composite resin blocks BLO, CER and GRA. The SEMs taken from ENA after 50 k sliding cycles with poppy seed slurry illustrated less pronounced surface destruction than in the two-body wear test. The ceramic network in the initial third of the wear trace was smoothly abraded, almost polished and the crushed poppy seed particles had eroded the exposed softer polymer matrix. The extent of surface damage increased along the wear trace where finally long and deep fracture lines were seen.

VIT is a feldspathic porcelain block material that also exhibited extensive surface deterioration under two-body wear, yet not as deep as the destruction found on ENA. This is also reflected in the volume loss of substance that was only half of the volume loss registered with ENA. Although the surface of VIT looks heavily eroded under three-body wear the volume loss indicates that only the very outmost layer is affected. In contrast to ENA, there was no polymer matrix in interstices that was preferentially abraded by the small scratching particles of the abrasive medium.

Part III Color differences of CAD/CAM composite resin blocks after immersion in coffee

The ΔE values obtained with the CAD/CAM composite resin block materials after immersion in coffee were significantly different. Therefore, the null hypothesis that there would be no significant difference regarding discoloration and translucency changes among the CAD/CAM composite resin blocks after immersion in coffee was rejected.

The color changes using black and white backgrounds demonstrated a similar trend. The black background was selected for determination of the discoloration in order to mimic the dark intraoral background. The ΔEs after immersion in water were not significantly different, in contrast to those after immersion in coffee that showed a significant increase, apart from TEL, a PMMA-based block material. This finding is in agreement with a previous study, reporting that upon immersion in coffee composite resin cements showed greater ΔE values compared to PMMA-based resin cements[43]. ENA, VIT and TEL demonstrated small discoloration with less than two ΔE units. This low response on the staining solution might be related to their different structure and composition when compared with the other materials investigated.

The 50:50% perceptibility threshold (PT) and 50:50% acceptability threshold (AT) of dental ceramics tested under simulated clinical setting were 1.2 and 2.7, respectively[132]. After one month of immersion in water apart from FSU the 50:50% PTs of the materials tested in this trial were less. The 50:50% ATs after one week of

immersion in coffee proved that the investigated materials, except DUR and FSU fell within the acceptability range. In contrast, the ATs of BLO, KZR, ULT, DUR, and FSU after one month of immersion in coffee exceeded the threshold.

Previous literature suggested that immersion in coffee for one week was equivalent to seven months of coffee drinking with the assumption that the coffee remained in the mouth during drinking[43]. Thus, immersion of specimens in coffee for one month for evaluation of the resulting staining effect might be an exaggeration of reality. However, in spite of this severe condition the ΔEs of all CAD/CAM block specimens after one week of immersion in coffee were under the AT. Even after the one-month challenge with coffee, apart from BLO and ULT the block materials tested went below the AT. Overall, this result indicated high resistance of CAD/CAM block materials to staining. The direct composite resins DUR and FSU showed pronounced discoloration in this test that might indicate a risk for perceptible staining during clinical service[152, 153].

After maintenance polishing with prophylaxis paste discoloration of the coffee-stained specimens was significantly reduced, apart from DUR and FSU, indicating that the discolorations noticed on most of the products investigated was extrinsic. It is assumed that regular oral hygiene measures will eliminate or reduce surface stains effectively.

In the present *in vitro* study the effects of up to one-month immersion in coffee and water of CAD/CAM composite resin blocks on discoloration were

evaluated. In the oral environment restorative materials are also subjected to numerous other liquids, to temperature and load stress, and to tooth brushing. The limitation of this *in vitro* study is that the clinical environment and its effect on the performance of CAD/CAM composite resin blocks discoloration resistance were not fully mimicked. Therefore, further investigations are recommended to evaluate the effect of additional contributing factors on the long-term discoloration resistance of CAD/CAM composite resin block materials.

CHAPTER VI

CONCLUSIONS

Within the limitations of this study, the following conclusion can be drawn:

The flexural properties of composite resin blocks tested in this study were comparable to those of a ceramic block, but still far inferior to the reported value of lithium disilicate glass ceramic and densely sintered yttrium-stabilized zirconia for CAD/CAM[142]. The results of this present study suggest that restorations fabricated from the CAD/CAM composite resin blocks investigated are suitable when limited to single premolar crowns, but not for fixed partial prostheses as recommended in ISO 6872:2008[17].

In two-body wear, all CAD/CAM composite resin block materials showed low wear compared to reference ceramic material. In three-body wear, all CAD/CAM composite resin block materials investigated showed comparable to reference ceramic material. The present quantitative *in vitro* wear data and the qualitative comparative investigation of the worn surfaces' morphologies after 50,000 sliding cycles in two- and three-body wear mode have shown that overall the wear resistance of the four composite resin blocks, the hybrid ceramic and the traditional feldspar ceramic tested was enhanced when compared with the wear produced on current light-cured direct posterior composite resin using the same test protocol[107]. Considering that wear is no longer an important concern with modern composite

resin materials, even when applied as amalgam substitutes in extended molar cavities[101, 154-156], it is justified to suppose that the present material blocks investigated are suitable for production of CAD/CAM single premolar crowns.

When selecting materials for clinical use, ΔE values of CAD/CAM composite resin blocks should be considered to ensure long-term esthetic appearance. The ΔE values after immersion in water did not significantly change, whereas those after immersion in coffee significantly increased with increasing immersion time. Maintenance polishing with prophylaxis paste reduced effectively discolorations of all CAD/CAM block materials and most of the light-cured composite resins noticed after immersion in coffee. Discoloration upon storage in coffee was predominantly extrinsic. The ΔE values after maintenance polishing of all CAD/CAM composite resin block materials were effectively reduced and still within the acceptability threshold.

To achieve clinical success of restorations, function, esthetics, longevity and biocompatibity of materials are considered important. Understanding the characteristics of new CAD/CAM materials is necessary for determining the clinical application, as well as the limitation of these materials. Our study indicated that CAD/CAM composite resin blocks is suitable to be another definite restorative material of choice in terms of adequate strength, wear resistance, and color stability.

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