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Original Article

Fracture toughness of three heat pressed ceramic systems

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Aim: The aim of this study was to evaluate fracture toughness by indentation method of three dental ceramics processed by heat pressing. The ceramics evaluated were fluorapatite glass ceramic (ZIR), glass ceramic containing leucite (POM) and leucite-reinforced glass ceramic (EMP). **Materials and methods:** Ninety disks (13mm of diameter x 4mm of thickness) and nine rectangular specimens (25x4x2mm) were made to evaluate, respectively, microhardness/fracture toughness (n=30) and elastic modulus (n=3). Samples were obtained by pressing ceramic into refractory molds. After polishing, Vickers microhardness was evaluated under 4,904N load for 20s. Elastic modulus was measured by impulse excitation technique. Data from microhardness and elastic modulus were used to calculate fracture toughness, after measuring crack length under 19,6N load applied for 20s. Results were evaluated by ANOVA and Tukey´s test. **Results:** Microhardness (VHN) of POM (637.9±53.6) was statistically greater ($p<0.05$) than ZIR (593.0±14.3), followed by EMP (519.1±21.5); no significant difference ($p=0.206$) was noted for elastic modulus (GPa) (ZIR: 71.5±9.0; POM: 67.3±4.4; EMP: 61.7±2.3). Fracture toughness (MPa/m) of POM (0.873±0.066) was statistically lower ($p<0.05$) than ZIR (0.977±0.021) and EMP (0.965±0.035). **Conclusion:** The results suggest that fluorapatite glass ceramic (ZIR) and leucite-reinforced glass ceramic (EMP) processed by heat pressing presented greater fracture toughness, improving clinical prognosis of metal free restorations.

Keywords: Dental porcelain. Hardness tests. Elastic modulus.

INTRODUCTION

The use of dental ceramics has increased because of aesthetics, biocompatibility, chemical stability, compression strength and color stability[1-2]. Nevertheless, ceramics are brittle materials that are unable to absorb elastic energy and resists to crack propagation, fracturing. Then, several all-ceramic materials and processing techniques have been introduced in the past decade[3-4]. Heat pressing, slip-casting and computer aided design-computer aided machining (CAD-CAM) are the processing techniques developed to offer greater mechanical performance associated with higher crystalline content of new ceramic systems, extending the indications to posterior restorations and fixed partial dentures[5].

Common defects, inherent in the processing, such as internal microcracks, pores, inclusions and second-phase clusters, can possibly propagate under tensile stresses until achieving a critical size, fracturing[6].

The methods used in the processing of materials may lead to different mechanical properties[7]. Heat pressing involves the use of heat and pressure to inject molten ceramic in an investment mold[8]. Several studies have evaluated the effect of heat pressing in the properties of different ceramic systems[8-12]. There is not a consensus about the effect of heat pressing at the mechanical properties and microstructure of ceramic. A previous study reported that repeated heat pressing increased the size of crystals and porosities and decreased density, hardness, flexural strength and fracture toughness of lithium disilicate[12]. Other authors argued that mechanical properties of lithium disilicate were not significantly affected by repressing, although qualitative differences in the microstructure[11]. Another study that evaluated lithium disilicate and leucite based ceramics reported a decrease in porosities and a better crystal distribution after heat pressing, increasing flexural strength although the similar fracture toughness[9]. Veneering ceramics obtained by heat pressing and layering for metal and zirconia copings were also compared and authors reported that veneering ceramics to zirconia are affected by processing technique while the veneering ceramic for metal alloys, not[8].

Fracture toughness is defined as the resistance of brittle materials to rapid crack propagation[13]. Then, this property represents serviceability in the oral cavity[14]. There are several methods to evaluate fracture toughness: indentation fracture, single edge "v" notch beam, single edge precracked beam, surface crack in flexure and chevron notch beam, but the indentation method is popular because it is simple, non-destructive and permits evaluation at small samples[15]. Fracture toughness is related to elastic modulus, crack length and microhardness[16].

Thermal mismatch, chemical stress and mechanical process such as cyclic loading can lead to crack initiation at surface, and these microscopic cracks propagate trough bulk of the restorations, leading to catastrophic failures[17-18]. Then, fracture toughness permits to predict the clinical performance (fracture and wear) of ceramic materials describing the ability of the material to withstand crack propagation[7,19].

Although fracture toughness of some ceramic systems is sometimes provided by manufacturers, studies have reported that heat pressing has affected differently mechanical properties of ceramic systems. Thus, the aim of the present study was to evaluate fracture toughness of three different ceramic systems processed by heat pressing.

MATERIAL AND METHODS

For fracture toughness evaluation by indentation technique, microhardness and elastic modulus were also evaluated.

Specimen preparation

In the present study, three ceramic systems were evaluated (Table 1). For this, thirty disks (13mm diameter x 4mm thickness) and three rectangular (25mm length x 4mm width x 2mm thickness) specimens of each ceramic were obtained dripping molten wax in teflon matrixes. Then, wax patterns were invested (Bellavest SH, BEGO Bremer Goldschlägerei, Bremen, Germany), submitted to the heating cycle (Table 2) of the investment. After, ceramic ingots and plunger were positioned in the investment block and ceramic was heat pressed (Table 3) in the ceramic furnace (Alumini Sinter Press, EDG Equipamentos, São Carlos, SP, Brazil).

After pressing, samples were divested using airborne-particle abrasion with 100µm glass beads (Renfert, Hilzingen, Germany), and polished with silicon carbide papers (320-2000, Norton, Saint-Gobain Abrasivos Ltda., Igarassu, Pernambuco, Brazil).

Microhardness

Vickers microhardness was evaluated (n=30) at disks with a load of 4,904 N applied for 20 seconds (HMV-2, Shimadzu Corp., Kyoto, Japan)[20]. Five randomly chosen sites per sample were evaluated and a mean value (VHN) was obtained.

Table 1. Ceramic systems, indications and commercial names.

Materials	Indication	Commercial name (Manufacturer)
Fluorapatite glass ceramic (ZIR)	Pressing onto zirconia copings and frameworks	IPS e.max ZirPress (Ivoclar Vivadent)
Leucite-reinforced glass ceramic (EMP)	All-ceramic single tooth restorations	IPS Empress Esthetic (Ivoclar Vivadent)
Glass ceramic containing leucite (POM)	Pressing onto metallic copings and frameworks	IPS Inline POM (Ivoclar Vivadent)

Table 2. Heating cycle of investment blocks.

Initial temperature	Temperature increase rate 1	Stage 1	Temperature increase rate 2	Stage 2	Pressing
Room temperature	20°C/min	300°C (30 min)	25°C/min	800°C/850°C* (60min)	800°C/850°C*

* Investment blocks were heated until 850°C only for E pressing.

Table 3. Firing parameters of each ingot pressing for all ceramic systems tested.

Material	Stand-by temperature	Temperature increase rate	Firing temperature	Holding time
IPS e.max ZirPress (ZIR)	700°C	60°C/min	900°C	15 min
IPS Empress Esthetic (EMP)	700°C	60°C/min	1075°C	20 min
IPS Inline POM (POM)	700°C	60°C/min	940°C	20 min

Elastic modulus

Elastic modulus was evaluated (n=3) at rectangular specimens by impulse excitation technique (Sonelastic, ATCP Physical Engineering, Ribeirão Preto, SP, Brazil)[21]. Specimens were positioned and submitted to an impact by a pulsator. The acoustic response was captured by a transducer and translated into electric sign to read the resonance frequencies. Elastic modulus was determined by bending excitation.

Fracture toughness

Fracture toughness was evaluated (n=30) by the indentation method proposed by Anstis[22], using a microhardness tester (HMV-2, Shimadzu Corp., Kyoto, Japan)[4,11,18,20]. A load of 19,6N was applied for 20 seconds. Three randomly sites per sample were evaluated. Crack length was measured from the center of indent and a mean value for cracks was used to calculate fracture toughness using the formula[20], where K_{IC} is fracture toughness (MPa/m), E is elastic modulus (GPa), P is the load applied (N), H is Vickers microhardness (GPa) and c is crack length(m):

$$K_{IC} = 0{,}016 \left(\frac{E}{H}\right)^{0{,}5} \left(\frac{P}{C^{1{,}5}}\right)$$

The data of microhardness, elastic modulus and fracture toughness were analyzed by one-way ANOVA and post-hoc Tukey´s test using statistical software (SPSS 17.0, IBM SPSS software, IBM Corporation). A significance level of 5% was used.

RESULTS

The results of Vickers microhardness are shown in Figure 1. Microhardness of ceramic POM (621.9±78.1) was statistically greater than ZIR (593.0±14.3) and EMP (519.1±21.5), and ZIR was statistically greater than EMP (p<0.05).

The results of elastic modulus are presented in Figure 2. The elastic modulus of the ceramics ZIR (71.54±9.02), EMP (61.67±2.26) and POM (67.27±4.42) was statistically similar (p=0.206).

Fracture toughness of ZIR (0.977±0.021) and EMP (0.965±0.035) was statistically similar, and both ZIR and EMP presented greater fracture toughness than POM (0.873±0.066) (p<0.05). The results are presented in Figure 3.

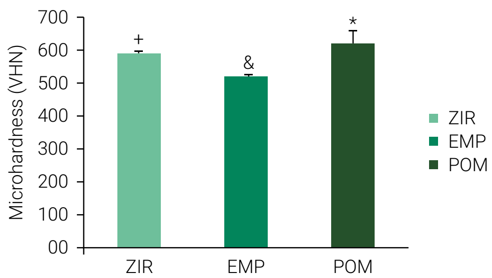

Figure 1. Results of Vickers microhardness (VHN) of the ceramics evaluated.Different symbols represent statistically significant difference (α=5%).

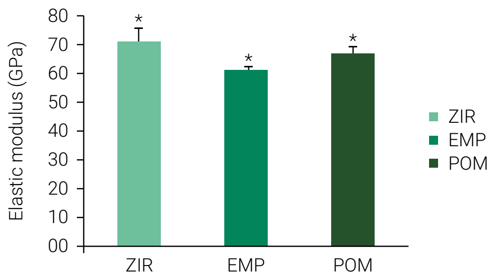

Figure 2. Results of elastic modulus (GPa) of the ceramics evaluated.Similar symbols show that elastic modulus of the evaluated ceramics is statistically similar (α=5%).

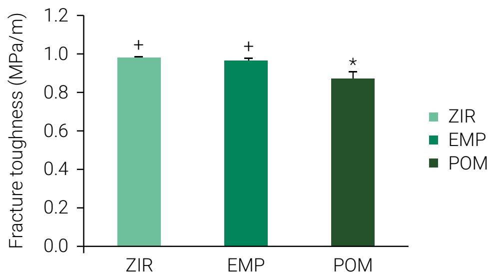

Figure 3. Results of fracture toughness (MPa/m) of the ceramics evaluated. Different symbols represent statistically significant difference (α=5%).

DISCUSSION

The ability of a material to react to a load and/or impact is described by its mechanical properties. Dental ceramics are brittle materials that fracture into two or more pieces when they are submitted to tensile forces[23]. Thus, determination of fracture toughness of these materials is important to predict the clinical performance once measures the ability of a material to withstand crack propagation[7,19].

In the present study, fracture toughness was evaluated by indentation method. This method was chosen because it is fast and permits measuring cracks in a small sample. Although there is difference in the results of fracture toughness evaluated by different methods[15,24], this study aimed to compare fracture toughness of three ceramics using the indentation method. Fracture toughness measured by indentation method is related to hardness and elastic modulus. When hardness increases, toughness and plasticity decreases, increasing fragility[25].

The ceramic EMP is a leucite-reinforced glass ceramic. According to some authors, high leucite content, particle size and homogeneous particle distribution can improve ceramic strength[6]. Then, high fracture toughness of EMP can be attributed to its leucite content (35%), once there is a positive correlation between leucite content and fracture toughness because leucite increases the resistance to crack propagation[19,26]. Additionally, fracture toughness increases with greater particle size[6], corroborating the results of the present study where greater crystals (5 to 10 micrometer) are present in EMP. A previous study reported that a homogeneous crystal distribution is achieved by heat pressing[5], possibly increasing fracture toughness because crack propagation is avoided. However, another study reported that fracture toughness of this ceramic is not affected by pressing and repressing even crystal distribution is more uniform, and pores and defects introduced during machining decrease in number and size after pressing[9].

POM is a glass ceramic containing leucite, but the concentration and size of leucite crystals present in this ceramic is not presented by the manufacturer. Considering that POM presented lower fracture toughness of this study, the leucite content and crystal size are probably lower than in EMP that is a leucite reinforced glass ceramic. In addition, the low fracture toughness of POM is directly affected by its high microhardness while the lowest microhardness of EMP leads to greater fracture toughness than POM. Some authors also argued that some surface flaws (microcracks, scratches and broken edges) of dental ceramics are caused by grinding or usage[6]. All samples were polished for microhardness and fracture toughness evaluation, and polishing may originate surface flaws, especially when materials present greater microhardness (POM) that make difficult polishing. Then, low fracture toughness of POM can also be attributed to surface flaws originated from polishing.

Some authors argue that fracture toughness of leucite containing ceramic is greater than non-leucite glass ceramic[26], but fracture toughness of EMP (leucite reinforced glass ceramic) and ZIR (fluorapatite glass ceramic) was statistically similar and greater than POM (glass ceramic containing leucite) in the present study. ZIR is a ceramic whose microstructure is primarily amorphous with little evidence of crystalline phase by fluorapatite crystals[26]. According to some authors, the higher content of needle-like fluorapatite crystals increases Vickers microhardness and fracture toughness[27]. Then, high fracture toughness of this ceramic is attributed to its fluorapatite crystals, that measure 100nm by 300nm and 300nm by 2 to 5 micrometer, as manufacturer provides.

The results of Vickers microhardness (600VHN) and fracture toughness (0.9±0.3MPa/m) provided in scientific documentation of POM manufacturer´s is similar to the results (POM=0.873MPa/m) found in the present study. However, the fracture toughness results are lower than results of other study that evaluated the same ceramic[8]. This difference is probably caused by the difference of the method used to evaluate fracture toughness because the previous study used single edge notched beam method[8] and there is difference in the results of fracture toughness evaluated by different methods[15,24]. The same difference was noted for fluorapatite glass ceramic (ZIR=0.977MPa/m) that presented lower fracture toughness in the present study than in the study mentioned before[8]. On the other hand, lower values of fracture toughness results were found in a study that evaluated this fluorapatite glass ceramic before and after pressing, using or not zirconia in association[26].

Differences of results in the fracture toughness values of this study in relation to the literature can be caused by residual stress after heat pressing and polishing. Some authors recommend slow cool at the last heating cycle, such as glazing, to reduce residual stress within ceramic system[26]. Additionally, defects introduced during machining also affect ceramic strength[9]. Considering that samples of this study were polished and not submitted to auto-glaze heating cycle after polishing, different fracture toughness results was probably affected by polishing that can introduce surface flaws and interfere with fracture toughness.

It is important to consider that this study evaluated intrinsic fracture toughness of ceramics ZIR and POM once this ceramics were evaluated without zirconia or metallic copings, differently from their clinical application. Some authors argued that fracture toughness of ceramic is greater than ceramic/zirconia samples because the last present residual stress in its interface because of thermal expansion coefficient mismatch[10]. Differently, other study that evaluated residual stress at ceramic-metal and ceramic-zirconia systems, correlating residual stress with fracture toughness, reported that fracture toughness is higher where compressive residual stress is greater, especially close to surface and interface while tensile stresses found in the bulk of ceramic of ceramic-metal system decreases fracture toughness[28]. Then, the compressive residual stresses created at the surface and interface of ZIR and POM associated to zirconia and metal, respectively, would increase fracture toughness of these ceramics. In addition, it is also necessary to consider the effect of the geometry of sample that can affect the stress and possibly fracture toughness[28].

Although POM presented lower fracture toughness, it is important to consider that this ceramic is indicated for metal-ceramic restorations and metallic coping would improve mechanical behavior of the restorations[19]. The ceramics ZIR and EMP presented higher fracture toughness but the association with zirconia copings can increase fracture toughness of ZIR, improving the clinical prognosis.

In the present study, microstructure of ceramics were not evaluated, but further studies are necessary to evaluate microstructure of these ceramics before and after heat pressing, correlating these information with their mechanical properties.

Based on the results of the present study, it is possible to conclude that fluorapatite glass and leucite-reinforced glass ceramics processed by heat pressing presented greater fracture toughness, improving clinical prognosis of metal free restorations.

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