



Effect of thermocycling aging on the flexural strength of feldspathic ceramic

Efeito da termociclagem na resistência flexural de uma cerâmica feldspática

Ronaldo Luís Almeida de CARVALHO¹, Estevão Tomomitsu KIMPARA¹, Eduardo BRESCIANI², Márcia Carneiro VALERA², Renata Marques de MELO¹

1 - São Paulo State University (Unesp) – Institute of Science and Technology – Department of Dental Materials and Prosthodontics – São José dos Campos – SP – Brazil.

2 - São Paulo State University (Unesp) – Institute of Science and Technology – Department of Restorative Dentistry – São José dos Campos – SP – Brazil.

ABSTRACT

Objective: The aim of the study was to evaluate the effect of aging through different thermocycling protocols on the flexural strength of a feldspathic ceramic. **Material and Methods:** Fifty ceramic bars, Vitablock Mark II (VITA), with dimensions of 18X4X2mm were prepared. The bars were randomly distributed to the groups (n = 10), which were defined according to the number of thermal cycles (TCy): G0 - no TCy; G500 - 500 cycles of TCy; G6000 - 6000 TCy; G10000 - 10000 TCy; G15000 - 15000 TCy. After aging, the specimens were subjected to the three point bending test in a universal testing machine (EMIC DL 1000), under 0.5mm/min speed and loading of 50kgf, until catastrophic failure. A sample of each group was evaluated for topographic morphology under Scanning Electron Microscopy. **Results:** For mechanical testing, the following mean values and standard deviation (MPa) were obtained: G0 (99.78 ± 5.07); G500 (101.64 ± 5.59); G6000 (98.13 ± 4.95); G10000 (91.77 ± 9.68); G15000 (101.51 ± 4.22). An analysis of variance by regression equation (p = 0.387) was performed, demonstrating a weak and non-significant correlation between flexural strength and number of thermal cycles. **Conclusion:** It can be concluded that aging in water solely by different numbers of temperature cycles did not influence on the flexural strength of a feldspathic ceramic.

KEYWORDS

Material resistance; Aging; Ceramics.

RESUMO

Objetivo: O objetivo do estudo foi avaliar o efeito do envelhecimento por meio de diferentes protocolos de ciclos térmicos na resistência à flexão de uma cerâmica feldspática. **Material e Métodos:** Cinquenta barras de cerâmica, Vitablock Mark II (VITA), com dimensões de 18X4X2mm foram preparadas. As barras foram distribuídas aleatoriamente pelos grupos (n = 10), os quais foram definidos de acordo com o número de ciclos térmicos (CT): G0 – nenhum; G500 - 500 CT; G6000 - 6000 CT; G10000 – 10000 CT; G15000 - 15000 CT. Após envelhecimento, as amostras foram submetidas ao ensaio de flexão de três pontos em uma máquina de ensaios universal (EMIC DL 1000), sob a velocidade de 0,5 mm / min e célula de carga de 50kgf, até uma falha catastrófica. Uma amostra de cada grupo foi avaliada quanto à morfologia topográfica sob microscopia eletrônica de varredura. **Resultados:** Para os testes mecânicos, foram obtidos os seguintes valores médios e desvio padrão (MPa): G0 (99,78 ± 5,07); G500 (101,64 ± 5,59); G6000 (98,13 ± 4,95); G10000 (91,77 ± 9,68); G15000 (101,51 ± 4,22). Uma análise de variância pela equação de regressão (p = 0,387) foi realizada, o que demonstra uma correlação fraca e não significativa entre a resistência à flexão e do número de ciclos térmicos. **Conclusão:** Pode-se concluir que o envelhecimento somente em água por diferentes números de ciclos térmicos não influenciou na resistência à flexão de uma cerâmica feldspática.

PALAVRAS-CHAVE

Resistência de materiais; Envelhecimento; Cerâmicas.

INTRODUCTION

Indirect restorations are exposed to adverse conditions after cementation. Artificial *in vitro* aging can be performed to simulate *in vivo* conditions, namely temperature alterations and loading of the oral environment. Mechanical and thermal cycling are among the aging methods that can be used are. These procedures have been used to determine lifetime of restorative materials [1], in which surface cracks or wear contribute to faster deterioration and reduce the fracture resistance [2].

Thermocycling consists of water baths at different temperatures during several cycles. According to Fisher et al. [3], thermal cycles are responsible for aging, reproducing a period of clinical service on restorative materials [3].

The effects of thermal cycling on materials are repeated stresses of contraction and expansion, resulting in propagation of cracks and fractures, loss of retention, formation of cracks at interfaces, and degradation of the restorations [4-9]. In addition to that, water absorption and hydrolytic degradation during the process exert deleterious effect on materials. As an example, Amaral et al. [10] and De Munck et al. [5] showed the importance of water in the degradation of ceramics when the Si-O bonds of the tip of a crack are broken by hydrogen ions leading to the growth of defects. However, these effects are all dependent on aging protocols, such as number of cycles, resting time in each bath and time interval between baths. The consequences of these protocol variations are often contradictory results [10]. It is also difficult to find a consensus with regard to the number of cycles that represents a certain period of clinical service. There are studies indicating 10,000 thermal cycles are equivalent to one year [6] or five years [11-13] of clinical use, or even that 6000 cycles are equivalent to 5 years of use *in vivo* [3]. Thus, the number of cycles used herein was established based on these previous studies.

In spite of the method constraints, thermocycling is still widely used due to its ability to simulate some of the conditions that occur *in vivo*. Its use is mainly for the ageing of bonded interfaces, but there is a need to conduct thermocycling in one material alone so that we

can better understand its behavior at varying temperatures and moisture. In the present study we chose a feldspathic ceramic and several thermocycling duration times to determine the flexural strength. The null hypothesis was that there were no differences in the flexural strength depending on the thermocycling protocol.

MATERIAL & METHODS

Information about the material used in this study is presented in Table 1.

Table 1 - Manufacturer and composition of the material used

Material	Manufacturer	Classification	Composition
Vitablock Mark II	VITA Zahnfabrik (Bad Säckingen, Baden-Württemberg, Germany)	Feldspathic Ceramic	Mixture of crystalline feldspathic particles embedded in glass matrix. SiO ₂ (56-64%); Al ₂ O ₃ (20-23%); Na ₂ O (6-9%); K ₂ O (6-8%), CaO (0.3-0.6%)

For the fabrication of ceramic Vitablock Mark II bars, the blocks were sectioned into slices with diamond discs in a cutting machine (ISOMET 1000 Buehler Ltd; Lake Bluff, Illinois, USA). A second cut was performed perpendicularly to the slices in order to obtain the bars. At this point the bars were still larger than the desired final dimension.

To standardize the specimens' dimension, bars were polished with 320, 400, 600, and 1200 grit sandpaper under water irrigation. The margins were beveled as recommended by the ISO standard for flexure test. Dimension of the final bars was 18X4X2mm, measured with a digital caliper (Mitutoyo; Suzano, São Paulo, Brasil).

Thermocycling consisted of baths in water at 5 °C and 55 °C for 30 seconds, with 2 seconds time interval between baths.

Flexural strength was determined for the 50 experimental units, randomly distributed among different groups (Table 2) (n = 10).

Table 2 - Group names and description according to the number of thermal cycles

Groups	Description
G0	Vitablock Mark II without thermocycling
G500	Vitablock Mark II + 500 Cycles of thermocycling
G6000	Vitablock Mark II + 6,000 Cycles of thermocycling
G10000	Vitablock Mark II + 10,000 Cycles of thermocycling
G15000	Vitablock Mark II + 15,000 Cycles of thermocycling

For the three point bending test, bar was placed in a metal device, being supported on two cylinders (2 mm in diameter) with a span distance of 16mm. The load was applied by cylindrical rod (2 mm in diameter) attached to a universal testing machine (EMIC DL 1000; São José dos Pinhais; Paraná, Brazil), subjecting the specimen to bending (0.5 mm/min speed, 50 kgf load cell) until catastrophic failure [14,15].

The mechanical testing procedures were performed immersed in distilled water at 37 °C. For this purpose, a glass container that has received an electric resistance and a thermometer, allowing the device to mechanical testing stay located inside was used.

After mechanical test, a sample from each group was evaluated for topographic morphology in Scanning Electron Microscope (Fei Company Model Inspect-S50; Hillsboro, Oregon, USA), with acceleration speed of 25 kV and an increase of approximately 1,000X.

To determine the relation between the number of cycles and strength, an analysis of variance was performed using a regression equation.

RESULTS

Data from the mechanical testing, initially collected in kgf, were converted to MPa values and averaged. The descriptive statistics is presented in graphical form (Figure 1).

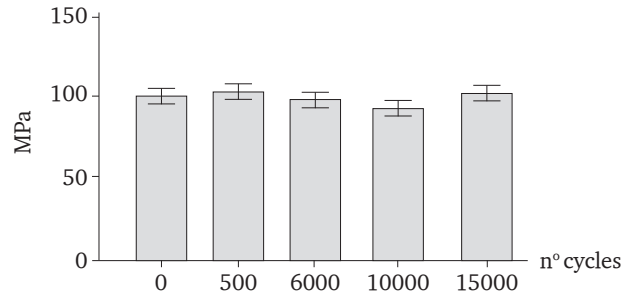


Figure 1 - Chart presenting mean strength values (MPa) and SD according to the number of cycles.

The analysis of variance using a regression equation was performed (p = 0.387), demonstrating a weak and negative (reverse) correlation (Figure 2) between strength and number of thermal cycles.

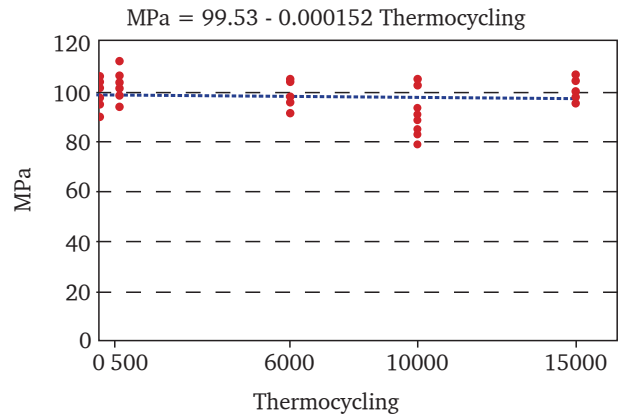


Figure 2 - Graph of regression equation.

To illustrate the topographic surface of ceramic bars after different thermocycling protocols, the surface analysis in Scanning Electron Microscope, with acceleration speed of 25 kV and magnification of 1000X was performed.

The photomicrographs of feldspathic ceramic (Figure 3) revealed similar topographic patterns for all groups, consisting of a flat surface with the presence of pores and polishing marks (indicated by arrows in Figure 3).

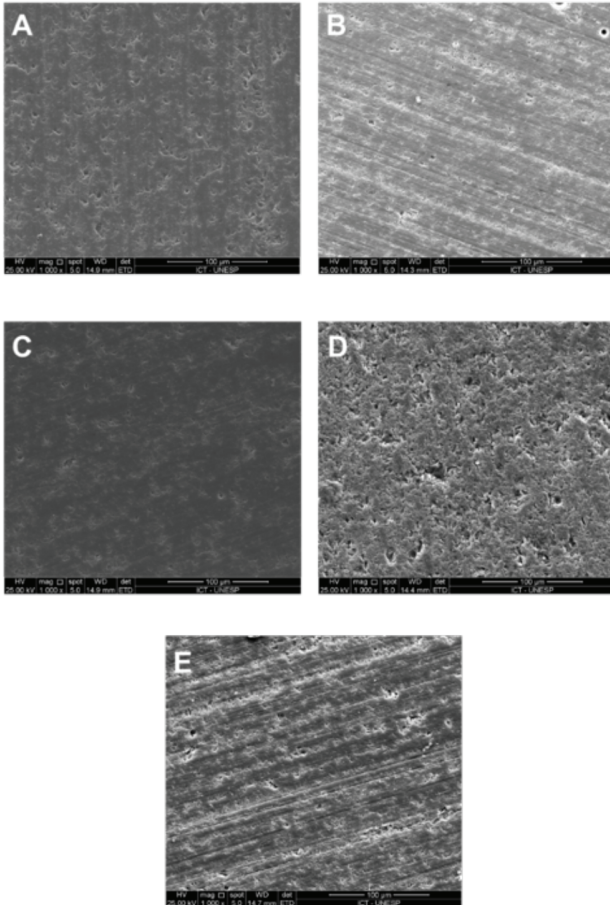


Figure 3 - Photomicrographs of the surfaces of the bars according to the numbers of cycles (A- without thermocycling; B- 500 cycles of thermocycling; C- 6,000 cycles; D- 10,000 cycles; E- 15,000 cycles).

DISCUSSION

This study showed that varying the number of thermal cycles until 15,000 did not result in statistically significant differences of the flexural strength of a feldspathic ceramic, confirming our anticipated null hypothesis.

With respect to aging protocols, few studies in the literature addressed the intrinsic resistance of ceramics over time. In this study, aging by thermocycling showed that for the feldspathic ceramic, there was not a significant decrease in flexural strength according to the number of thermal cycles performed. These results can be due to the fact that water at different temperatures was used as a single aging factor, as the crack growth is also associated with the presence of low and repetitive loads [16-20].

Moreover, other factors influence the ultimate resistance of ceramic materials including size of the specimens, the test environment, polishing procedure, area of the specimen under tension and test method [21].

From the SEM analysis, it can be observed that there was no difference between the topographical patterns of the photomicrographs for all groups. Although we used with a CAD-CAM ceramic whose homogeneity (less presence of internal and external defects) is higher than ceramics that are manually applied [22-24], the presence of surface pores certainly affected the final strength of the specimens. This may explain the greater variability of the data in the group of 10,000 cycles.

Recently, de Pinho Barcellos et al.[25] found significant differences on lithium disilicate strength depending on the type of ageing. This was probably because this a bi-phase material and the temperature variations led to different thermal contractions of the phases and a decrease in strength. Moreover, according to the authors, the ageing protocol made intra-orally caused more damage to the material than an in-vitro protocol, showing that long lasting thermocycling may not be enough to cause an important decrease in strength.

Therefore, the differences in the mechanical properties of materials due to thermocycling are often a result of thermal mismatches of different phases [26] or different materials combination such as porcelain and zirconia [27]. Also, water and temperature variations in the feldspathic ceramic used herein were not sufficient to cause topographical changes or a decrease in strength.

Thus, thermocycling aging might be an important condition only when different materials with different thermal behaviors are assessed. For the present study, this type of aging did not have an impact on ceramic strength, no matter the number of thermal cycles.

CONCLUSIONS

Based on the results of this study it can be concluded that aging in water after several number of thermal cycles had no influence on the flexural strength of a feldspathic ceramic.

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Ronaldo Luís Almeida de Carvalho (Corresponding address)

Instituto de Ciência e Tecnologia
Curso de odontologia
Av. Engenheiro Francisco José Longo, 777
12245200 São José dos Campos, SP, Brazil.
55-12-39479011
ronaldo.unesp@outlook.com

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