## UNIVERSIDADE DE UBERABA ÂNGELA BARROS CECÍLIO

EFEITO DO PERÓXIDO DE HIDROGÊNIO À 38% NA RESISTÊNCIA FLEXURAL E PROPAGAÇÃO DE TRINCAS DE UMA CERÂMICA ODONTOLÓGICA

# ÂNGELA BARROS CECÍLIO

# EFEITO DO PERÓXIDO DE HIDROGÊNIO À 38% NA RESISTÊNCIA FLEXURAL E PROPAGAÇÃO DE TRINCAS DE UMA CERÂMICA ODONTOLÓGICA

Dissertação apresentada ao Programa de Mestrado em Odontologia da Universidade de Uberaba, para obtenção do Título de Mestre em Odontologia, área de concentração em Biomateriais.

Orientador: Prof. Dr. Gilberto Antonio Borges

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#### UNIVERSIDADE DE UBERABA

## Pró-Reitoria de Pesquisa, Pós-Graduação e Extensão Programa de Mestrado em Odontologia



Ata da Sessão Pública de defesa de dissertação para obtenção do título de Mestre em Odontologia, área de concentração em Biomateriais, a que se submeteu a aluna Ângela Barros Cecílio – matrícula 6101948/1, orientada pelo Prof. Dr. Gilberto Antonio Borges Aos vinte e sete dias do mês de fevereiro do ano de dois mil e doze, às 8 horas, na sala 2C06 da Universidade de Uberaba, reuniu-se a Comissão Julgadora da defesa em epígrafe indicada pelo o Colegiado do Programa de Mestrado em Odontologia da Universidade de Uberaba, composta pelos Professores Doutores: Gilberto Antonio Boges - Presidente, Marcos Massao Shimano e Thiago Assunção Valentino, para julgar o trabalho da candidata Ângela Barros Cecílio, apresentado sob o título: "Efeito do Peróxido de Hidrogênio à 38% na ResistênciaFlexural e Propagação de Trincas de uma Cerâmica Odontologica". O Presidente declarou abertos os trabalhos e agradeceu a presença de todos os Membros da Comissão Julgadora. A seguir o candidato dissertou sobre o seu trabalho e foi argüido pela Comissão Julgadora, tendo a todos respondido às respectivas argüições. Terminada a exposição, a Comissão reuniu-se e deliberou pelo sequinte resultado:

XAPROVADO

REPROVADO (anexar parecer circunstanciado elaborado pela Comissão Julgadora).

Para fazer jus ao título de MESTRE EM ODONTOLOGIA ÁREA DE CONCENTRAÇÃO BIOMATERIAIS, a versão final da tese, considerada Aprovada devidamente conferida pela Secretaria do Mestrado em Odontologia, deverá ser entregue à Secretaria dentro do prazo de 30 dias, a partir da data da defesa. O aluno Aprovado que não atender a esse prazo será considerado Reprovado. Após a entrega do exemplar definitivo, o resultado será homologado pela Universidade de Uberaba, conferindo título de validade nacional aos aprovados. Nada mais havendo a tratar, O Senhor Presidente declara a sessão encerrada, cujos trabalhos são objeto desta ata, lavrada por mim, que segue assinada pelos Senhores Membros da Comissão Julgadora, pelo Coordenador do Programa de Mestrado em Odontologia da UNIUBE, com ciência da aluna. Uberaba, aos 27 dias do mês de fevereiro de dois mil e doze.

Prof. Dr. Gilberto Antonio Borges Reception	
Prof. Dr. Marcos Massao Shimano	
Prof. Dr. Thiago Assunção Valentino	
Prof. Dr. José Bento Alves	
Coordenador do Programa de Mestrado em Odontologia da UNIUBE	
Poliana Gomes da Silva Alves + chana Alve!	
Secretária do Programa de Mestrado em Odontologia da UNIUBE	
Ciência do Aluno:	hashandarian of our

#### **AGRADECIMENTOS**

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#### **RESUMO**

Tratamento clareador é rotineiramente utilizado no consultório odontológico, e por vezes este material é aplicado sobre restaurações cerâmicas existentes. O objetivo deste estudo foi avaliar a influência do peróxido de hidrogênio à 38% na resistência flexural e propagação de trincas de uma cerâmica odontológica enriquecida com leucita (Vitablock III, Vita Zanfabrik, Alemanha). Vinte barras de cerâmica (11 mm X 1.5 mm X 0.7 mm) foram obtidas de blocos cerâmicos e uma superfície (1.5 mm) foi polida com lixa de carbeto de silício em ordem decrescente de granulação até a lixa 1200 e divididas em dois grupos experimentais: 1) Grupo controle - Ponta penetradora Vickers foi aplicada na superfície polida (500 gr por 15 s), 2) - Procedimento de clareamento foi conduzido com peróxido de hidrogêncio 38% seguindo as recomendações do fabricante e após, a ponta Vickers foi penetrada da mesma maneira que no grupo controle. Comprimento das trincas geradas pelo penetrador foi medido para todos os corpos-deprova imediatamente e após sete dias (grupo controle: armazenado em água destilada a 37° C e grupo clareado recebeu os procedimentos de clareamento conforme protocolo do fabricante). Após, todos os corpos-de-prova foram submetidos ao teste de flexão de três pontos até a fratura. Teste de normalidade e teste t forma aplicados em nível de significância de 5%. Não houve diferença estatística entre os grupos avaliados (p=0. 3547). Dentro das limitações do estudo pode concluir que o tratamento clareador com peróxido de hidrogênio a 38% não afetou a resistência flexural e propagação de trinca da cerâmica enriquecida com leucita estudada.

#### **ABSTRACT**

Bleaching treatment is a popular esthetic dental treatment in many clinical situations. Sometimes it is not possible to avoid the contact between the bleaching material and the esthetic restorative materials. The aim of this study was to evaluate the influence of 38% hydrogen peroxide treatments on the flexural strength and crack propagation of ceramic enriched with leucite feldspathic. Twenty ceramic bars (11 mm X 1.5 mm X 0.7 mm) were obtained from ceramic blocks and one surface (1.5 mm) was polished up to 1200 carbide paper disc and divided into two experimental groups: 1) control group - a Vickers indenter was applied on the polished surface, bleached group (500 gr for 15 s),2) – the bleaching procedure was carried with 38% hydrogen peroxide following the manufacturer's instructions after Vickers indentation on the polished surface. Crack lengths were measured for all specimens immediately after indentation and seven days after bleaching or storage. The control group was stored in distilled water at 37° C for seven days, and the bleached group was subjected to the bleaching protocol. After, all specimens were tested by three-point bending until fracture. The normality test and t test were applied with 5% of significance. There was no statistical difference between the control and bleached groups (p=0. 3547). Within the limitations of this study, it can be concluded that the bleaching treatment used does not affect the flexural strength and crack propagation of the leucite enriched dental ceramic evaluated.

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#### 2-Title

Effect of 38% hydrogen peroxide on flexural strength and crack propagation of a dental ceramic.

Running title: Effect of hydrogen peroxide on dental ceramic mechanical properties.

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Effect of 38 % hydrogen peroxide on flexural strength and crack propagation of a

dental ceramic

Short title: Effect of hydrogen peroxide on dental ceramic mechanical properties

Clinical relevance: The results of this study suggest that, in a mechanical point of view, the

application of 38% hydrogen peroxide on ceramic restoration will not cause deleterious effect.

ABSTRACT

**Statement of problem.** Bleaching treatment is a popular esthetic dental treatment in many

clinical situations. Sometimes it is not possible to avoid the contact between the bleaching

material and the esthetic restorative materials.

Purpose. The aim of this study was to evaluate the influence of 38% hydrogen peroxide

treatments on the flexural strength and crack propagation of ceramic enriched with leucite

feldspathic.

Material and methods. Twenty ceramic bars (11 mm X 1.5 mm X 0.7 mm) were obtained

from ceramic blocks and one surface (1.5 mm) was polished up to 1200 carbide paper disc

and divided into two experimental groups: 1) control group – a Vickers indenter was applied

on the polished surface, bleached group (500 gr for 15 s),2) – the bleaching procedure was

carried with 38% hydrogen peroxide following the manufacturer's instructions after Vickers

indentation on the polished surface. Crack lengths were measured for all specimens

immediately after indentation and seven days after bleaching or storage. The control group

was stored in distilled water at 37° C for seven days, and the bleached group was subjected to

8

the bleaching protocol. After, all specimens were tested by three-point bending until fracture.

The normality test and t test were applied with 5% of significance.

**Results.** There was no statistical difference between the control and bleached groups (p=0. 3547).

Conclusions. Within the limitations of this study, it can be concluded that the bleaching treatment used does not affect the flexural strength and crack propagation of the leucite enriched dental ceramic evaluated.

Key words: dental ceramic; bleaching; mechanical property.

#### INTRODUCTION

Dental ceramics have characteristics such as chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to the tooth structure. These properties make them one of the preferred materials in restorative dentistry. In addition, they have esthetic appearance that are similar to natural dentition. However, if not well planned, their clinical use can jeopardize the longevity of the restoration because of the susceptibility to fracture, which is a result of material characteristics such as surface cracks and bulk defects. The use of ceramic restoration has increased considerably, because of esthetic and materials durability, which is attractive to both patients and clinicians. Materials Science has evaluated different ceramics with various crystalline phases and their processing techniques to achieve greater strength and toughness in order to avoid the use of metal core and improve esthetic.

Today, in a world of esthetics, patients desire a beautiful smile with whiter teeth being the key component. In many clinical situations, there are pre-existing restorations which are subjected to repeated bleaching processes.

There are studies about the surface roughness and color alterations of ceramic materials after bleaching treatments. 9,10 The literature shows controversy among the studies. However, limited data is available evaluating the effect of bleaching treatment on flexural strength and crack propagation of dental ceramics. Several methods have been used to evaluate different mechanical properties of ceramic materials. One of the most important properties is fracture toughness, which is a property that is a measure of a material resistance to brittle fracture when a crack is present<sup>4</sup>. There are different methods to make the crack on the ceramic surface, and one of the most commonly used is called Vickers test indenter, which is also called diamond pyramid test. The test consists of forcing a very small pyramid diamond indenter into the material surface for some seconds. After taking the indenter out, the impression is observed under a microscope. Cracks propagate in each end of the impression mark. By measuring these crack lengths, it is possible to see if the material will be more prone to fracture or not. Furthermore, using this indenter and knowing the cracks, it is possible to calculate the fracture toughness, thus confirming the capability of a brittle material to resist a crack growing. 11

As there is a lack of studies evaluating the effect of bleaching treatment on mechanical properties of dental ceramics, the aim of this study was to evaluate the effect of an in-office bleaching material on the fracture toughness and crack propagation of a dental ceramic. The null hypothesis was that the bleaching treatment would not affect flexural strength and crack propagation of Vitablock II feldsphatic ceramic.

#### **MATERIALS E METHODS**

#### Specimens fabrication

Blocks of Vitablock II Feldsphatic ceramic (Vita Zahnfabrik, Bad Säckingen, Germany) were sectioned using a low speed saw machine Isomet<sup>®</sup> 1000 (Buehler, Lake Bluff,

IL, United States) with a diamond blade of 100 mm diameter and 0.3 mm thickness. The saw speed was 300 rpm. Bars of 11.0 mm length, 1.5 mm wide and 0.7 mm thickness were obtained.

Only one wide surface (1.5 mm thickness) of the bars was polished starting with 600 silicon carbide paper disc to achieve a thickness of 0.67 mm, following by 800 to achieve 0.63 thickness, and finally 1200 to get 0.60 mm thickness (Figure 2). All polishing procedures were carried out on a bench polishing machine (APL-4, Arotec, São Paulo, Brasil) with 80 rpm speed. The thickness was checked with a digital caliper (Mitutoyo Sul Americana Ltda., São Paulo, Brasil), with 0.01 mm precision.

After polishing, the dimension of the bars was checked under a stereomicroscope Ken-Vision 4424, to assure that all specimens were free from voids and irregularities. Any specimen that showed any defect was eliminated. Twenty bars were divided in two groups (n=10):

Control group – The Vickers diamond indenter was applied on the polished surface (500 gr for 15 s), and the specimens were stored in distilled water at 37° C for seven days (same period of time that the bleaching procedure took).

Indented group – The Vickers diamond indenter was applied as in the control group, and, after that, bleaching procedure was carried on the polished surface.

#### Microhardness test

The penetration was carried out using an indenter model HMV - 2 (Shimadzu, Tokyo, Japan) with a Vickers diamond tip (Figure 3). The Vickers tip was applied with a 500 gram force for 15 s. The equipment conducted the test automatically, and a microscope connected to it allowed verifying the impression as well as cracks. Before the test, all dimensions of the specimens were checked again. After, a mark was done in the center of the polished surface of the bar. This procedure was made to make sure that the flexural load tip would be carried at the center of the bar.

Following the penetration, the crack length was measured. The control group was stored in distilled water at 37° C for seven days (same period of the bleaching protocol) and the bleached group was submitted to the bleaching protocol.

#### Bleaching procedure

All surfaces out of that polished of all specimens were insulated with a hydrophobic adhesive (Adper<sup>®</sup> Scotchbond multiporpose – 3M ESPE, Brasil).

The hydrogen peroxide at 38% (Opalescence® Xtra® Boost™ – Ultradent) was mixed with the activator following the manufacturer's instructions. Three bleaching sessions with three days interval between them were conducted. Three applications of 15 min were carried out. After each session, the specimens were washed and dried. At the end of each session the specimens were stored in distilled water at 37° until the next session. After the last session, the specimens were kept in distilled water for 24 hours.

#### Three point bending test

The crack length of all specimens was measured again in the same equipment HMV – 2 (Shimadzu, Tokyo, Japan), before the flexural test.

The three point bend flexural test was carried in a universal testing machine (DL3000 (EMIC – São José dos Pinhais, Brazil) using a cross speed of 0.5 mm/min. The specimens were placed on the testing machine using a specific three point device. The polished surface of the specimens was always placed in a facing down position, and this resulted in a tensile strain on that surface. The distance between the supports was 7.0 mm. A brass device with the same design of the supporters was used to apply the load on the superior surface right on the middle of the specimens, which position coincided to the penetration that was done on the inferior and polished surface. The diameter of the cylinders of the supporters and device load was 2.5 mm (The environmental temperature was kept between 23°C and 25°C).

#### Crack propagation

The crack length was measured immediately after indentation and seven days after, that corresponded the period of the bleaching treatment. The measuring was conducted as shown in the figure 1.

The measuring and all data collection were performed by the same operator. The mean of the four cracks of each specimen was used to calculate the crack mean of both groups, control and bleached.

#### **RESULTS**

The t test was applied to the control and bleached group immediately after indentation and showed no statistical difference (p=0.4072). The same way, the t test was applied to the control and bleached group seven days after indentation and showed no statistical difference (p=0.2299).

The t paired test showed significantly statistical difference between time periods (immediately and seven days or bleached) after crack propagation (p<0.0001). However, no significant differences were found between control and bleached groups (p=0.1190).

The t Student test, 5% significance was applied to analyze the flexural strength data. No statistical difference was detected between the groups (p=0.3547). All results are summarized in graphs 1 and 2 and table 1.

#### DISCUSSION

The null hypothesis that the bleaching treatment would not affect the flexural strength and crack propagation of the Vitablock II feldsphatic ceramic was accepted. The data and statistical analysis showed that no statistical difference was found between the control and bleached groups (graphs 1 and 2).

In literature, no study was found analyzing the effect of bleaching treatment on flexural strength and crack propagation of dental ceramics. However, studies have been published showing no adverse effect to micro hardness, surface roughness, or bond strength between ceramic and resin cement when subjected to carbide peroxide.<sup>18</sup> The findings of the

previous studies are in conjunction with the present. However, further research is needed to evaluate the effect of different concentrations of the bleaching agents on different restorative materials.

Regarding the flexural strength and crack propagation analysis, many techniques have been used. In the present study, Vickers micro hardness test was performed. 19,20 Additionally, the specimen dimensions can be relatively small, and the parameter to fracture growing is determined by cracks that are similar in size compared to clinical evaluation.<sup>21</sup> Moreover, the specimen thickness was standardized in 0.6 mm to try to simulate as closely as possible the thickness of a laminate ceramic that vary between 0.5 to 0.7 mm. 22,23 It could be inferred that the behavior of the ceramic material to propagate a crack would be different if the specimen was bonded to a dental substrate. In this point of view, the present results cannot be disregarded, because they evaluate an intrinsic property of the material. As bleaching procedure is largely used and the trend is to increase the use, the present results could be thought to advice the clinician that this treatment would not cause deleterious effect on ceramic restoration present in a mouth that needs bleaching. In a clinical situation that laminate ceramic restoration is present; no need to protect the restoration from the bleaching material would be needed. Next step in this subject maybe should be carried in ceramic restoration bonded to the tooth structure, even though this would be also related to the cementing agent.

#### **CONCLUSION**

Within the limitations of this study, it is possible to conclude that 38% hydrogen peroxide did not affect the flexural strength and crack propagation of enriched leucite feldsphatic ceramic.

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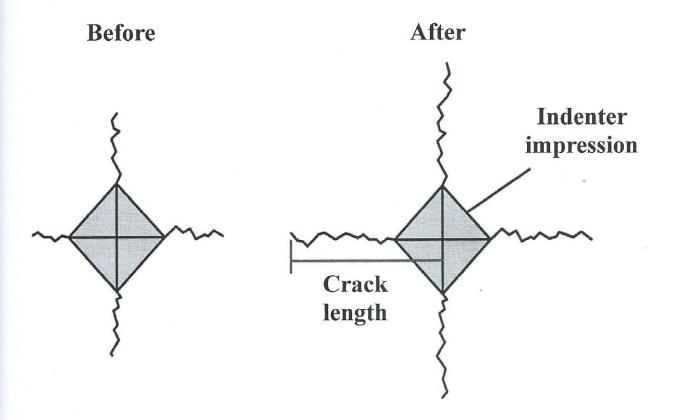
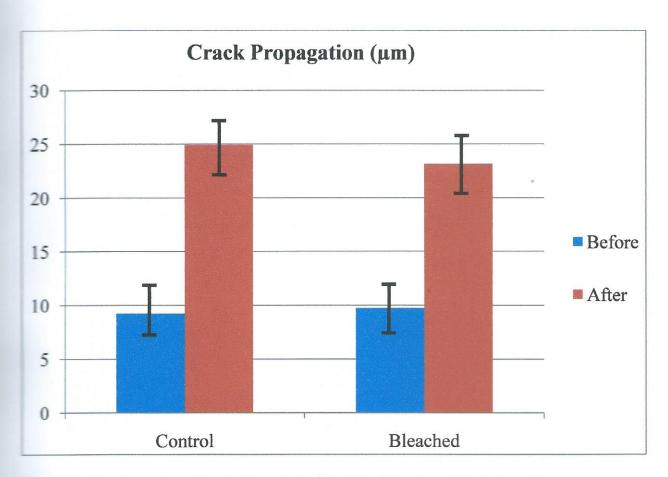


Figure 1. Schematic diagramo of the crack length before and after bleaching period.

Tabela I. Crack length (Mean and standard deviation (SD) in ( $\mu m$ ).

Time	Contr	ol		Ble	ached	
	N	Mean	SD	N	Mean	SD
Immediate	10	9.23 <sup>a</sup> A	(1.36)	10	$9,74^{a}A$	(1.52)
Seven days	10	24,92 <sup>a</sup> B	(3.42)	10	23.14 <sup>a</sup> B	(2.96)

Mean followed for distinct lower letters in row, and capital letter in column do not differ statistically (p>0,05).

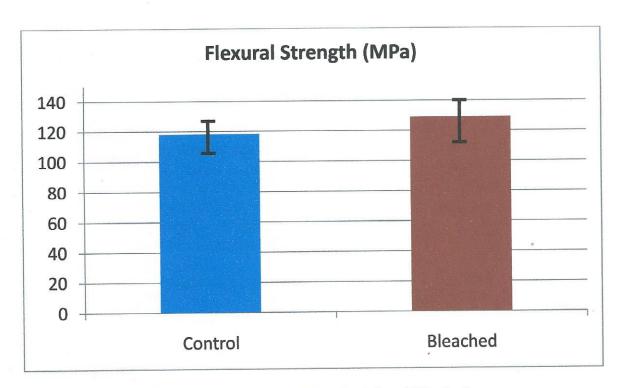


Graph 1 –Crack propagation before and after bleaching (control (sotored in distilled water) and bleached groups).

Tabel II. Flexural Strength (Mean and standard deviation (SD) in (MPa).

	Control			Bleached	
N	Mean	SD	N	Mean	SD
10	98.97 <sup>a</sup>	(8.32)	10	97,19 <sup>a</sup>	(14.32)

(p>0,05 Mean followed for distinct letters in row do not differ statistically).



Graph 2 - Flexural strength (MPa) control and bleached groups.

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Acessado no dia 07 de dezembro de 2011 às 11:40 min.

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    - Graphs
    - Tables
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- list of materials used
- potential problems
- summary of advantages and disadvantages
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#### **EXAMPLES OF REFERENCE STYLE**

- Journal article: two authors
   Evans DB & Neme AM (1999) Shear bond strength of composite resin and
   amalgam adhesive systems to dentin *American Journal of Dentistry* 12(1) 19-25.
- Journal article: multiple authors
   Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on
   adhesion to dentin Critical Review of Oral and Biological Medicine 8(3) 306 335.
- Journal article: special issue/supplement
   Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry
   Operative Dentistry (Supplement 6) 119-144.
- Abstract:
   Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003)
   Comparative study on adhesive performance of functional monomers *Journal of Dental Research* 82(Special Issue B) Abstract #0051 p B-19.
- Corporate publication:
   ISO-Standards (1997) ISO 4287 Geometrical Product Specifications Surface texture: Profile method Terms, definitions and surface texture parameters Geneve: International Organization for Standardization 1st edition 1-25.
- Book: single author Mount GJ (1990) An Atlas of Glass-ionomer Cements Martin Duntz Ltd, London.
- Book: two authors
   Nakabayashi N & Pashley DH (1998) Hybridization of Dental Hard Tissues
   Quintessence Publishing, Tokyo.
- Book: chapter
   Hilton TJ (1996) Direct posterior composite restorations In: Schwarts RS,
   Summitt JB, Robbins JW (eds) Fundamentals of Operative Dentistry
   Quintessence, Chicago 207-228.

- Website: single author Carlson L (2003) Web site evolution; Retrieved online July 23, 2003 from: http://www.d.umn.edu/~lcarlson/cms/evolution.html
- Website: corporate publication
  National Association of Social Workers (2000) NASW Practice research survey
  2000. NASW Practice Research Network, 1. 3. Retrieved online September 8,
  2003 from: <a href="http://www.socialworkers.org/naswprn/default">http://www.socialworkers.org/naswprn/default</a>

# **ANEXO** – Listade Figuras, Tabelas e Gráficos

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Figura 1. Detalhe dos blocos de cerâmica Vitablock II feldspática enriquecida com leucita (Vita Zahnfabrik, BadSäckingen, Germany).

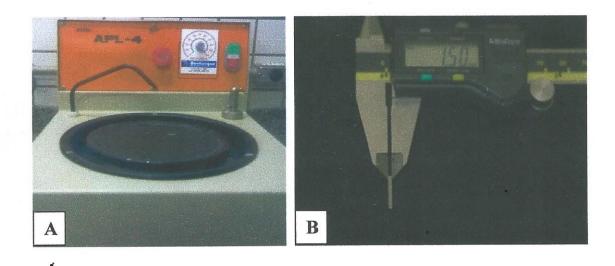


Figura 2. Polimento das barras. A - Politriz APL-4 (AROTEC, Brasil). B - Paquímetro utilizado para medir as barras de cerâmicas.

Fonte: arquivo pessoal.

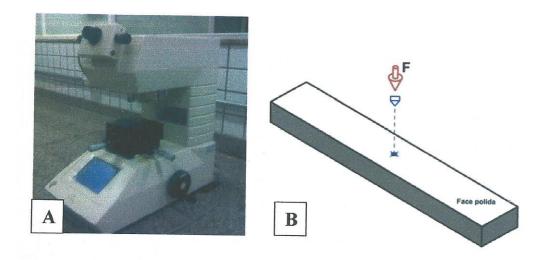


Figura 3. Teste de penetração. A - Equipamento modelo HMV-2 (Shimadzu, Tokyo, Japan) com um penetrador Vickers de diamante. B - Desenho esquemático do processo de penetração.

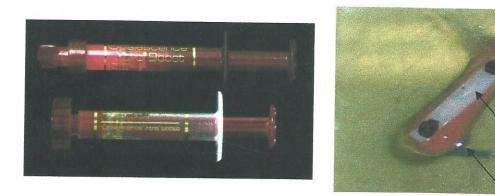


Figura 4. Clareamento. A-ClareadorOpalescence®Xtra®Boost™ da Ultradent. B-Corpo de prova submetido ao clareamento, com a face polida voltada para o agente clareador.

Fonte: arquivo pessoal.

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Corpo de prova

Clareador



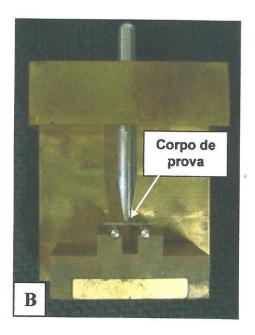


Figura 5. Ensaio mecânico de flexão em três pontos. A. Detalhes do acessório para realização dos ensaios. B-Posicionamento do corpo de prova no acessório.

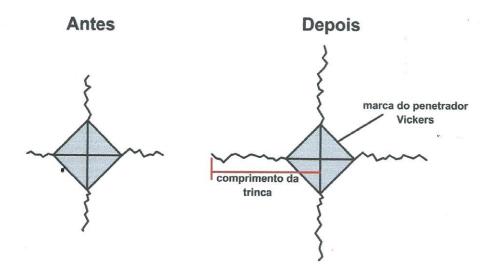


Figura 6. Desenho esquemático da obtenção do comprimento das trincas antes e após o período de clareamento.

Fonte: arquivo pessoal.

 $\mbox{Tabela $1-$M\'edia das trincas dos corpos de prova do grupo Controle antes e depois do armazenamento.}$ 

CDP	Controle			
СЫ	Antes	Depois	Diferença	
1	11,87	30,72	18,84	
2	8,73	30,30	21,57	
3	10,15	22,16	12,01	
4	8,59	22,22	13,62	
5	7,50	21,68	14,18	
6	7,51	23,28	15,77	
7	10,24	22,60	12,37	
8	9,87	24,84	14,97	
9	9,45	23,71	14,26	
10	8,37	27,74	19,37	
Média	9,23	24,92	15,69	
Desvio Padrão	1,36	3,42	3,19	

Tabela 2 - Média das trincas dos corpos de prova do grupo Clareado antes e depois do clareamento.

CDP		Clareado	
CDI	Antes	Depois	Diferença
1	9,79	22,95	13,16
<sub>e</sub> 2	10,58	18,54	7,97
3	7,85	22,59	14,74
4	10,59	19,54	8,95
5	9,21	21,58	12,38
6	7,05	23,98	16,93
7	11,03	25,42	14,39
8	8,69	22,45	13,76
9	11,85	26,14	14,30
10	10,74	28,28	17,53
Média	9,74	23,14	13,41
Desvio Padrão	1,52	2,96	3,05

Fonte: arquivo pessoal.

Tabela 3- Tensão flexural (MPa) obtida do ensaio de flexão em três pontos nos corpos de provas do Grupo Controle e Clareado.

CDP	Tensão Flexural (MPa)		
	Controle	Clareado	
1	103,15	124,11	
2	115,59	143,52	
3	127,87	196,94	
4	100,46	94,47	
5	148,96	98,98	
6	102,15	144,74	
7	103,40	. 115,46	
8	166,33	125,58	
9	109,64	121,41	
10	103,81	126,35	
Média	118,14	129,15	
Desvio Padrão	22,76	28,77	