

## Influence of ultrasonic setting on microhardness of glass-ionomer cements

Carvalho C A<sup>1</sup>, Fagundes T C<sup>2</sup>, Barata T J<sup>3</sup>,  
Navarro M F<sup>4</sup>

### Abstract

The aim of this study was to evaluate the influence of ultrasonic treatment on the microhardness of glass-ionomer cements. Nine commercially available brands of glass-ionomer cements were evaluated: Fuji IX, Ketac-Molar, Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus, Maxxion R, Bioglass R and Vitro Molar. Ten cylindrical specimens (2mm in diameter and 2mm in thickness) were made for each material and for each evaluation period. For the experimental group, ultrasonic treatment was applied to the unset specimens for 15s, using an EMS FT-081DN Mini PIEZON device, and they were subsequently covered with celluloid strips. The samples were exposed to 150g at 23°C for 15min and stored for durations of 15min, 1h, 12h and 24h. The Knoop Hardness Number was determined on indentations, made by applying a 50g load for 5s. Statistically significant effects of ultrasonic treatment in microhardness could be demonstrated for all the materials evaluated, except for Bioglass R and Vitro Molar after 24h of storage. At 15min, most of the samples were still too soft to be tested. With the exception of the Riva Self Cure, the earliest measurable Knoop Hardness Number values (with ultrasonic application)

were higher or similar to those values obtained (without ultrasonic application) for subsequent storage times. Conclusion: Ultrasonic command setting improved the microhardness of the glass-ionomer cements. Clinical Significance: The ultrasonic treatment accelerated surface hardening, which might reduce the early weakness of the glass-ionomer restorations. *First published in Int Dent S Afric 2007; 9: 24-32.*

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<sup>1</sup> Department of Operative Dentistry, Bauru School of Dentistry, University of São Paulo, Brazil  
Department of Dental Materials, University of Siena, Policlinico Le Scotte, Siena, Italy

<sup>2</sup> Department of Operative Dentistry, Bauru School of Dentistry, University of São Paulo, Brazil

<sup>3</sup> Department of Operative Dentistry, Bauru School of Dentistry, University of São Paulo, Brazil

<sup>4</sup> Bauru School of Dentistry, University of São Paulo, Brazil

### Address of first author:

Julia Miranda 18, Zip Code: 36.400-000  
Conselheiro Lafaiete – Minas Gerais- Brazil  
Phone: 55-31-3763-3077  
E-mail: carcarvalho@usp.br

## Introduction

The setting reaction of glass-ionomer cements occurs in two phases: an initial set with the formation of mainly calcium polyacrylate and a subsequent hardening process with the formation of aluminum polyacrylate<sup>1</sup>. During the initial setting stages, the loosely bound water causes some integrity problems<sup>1,2</sup>. During the first reaction, the material is very susceptible to water uptake and to dehydration in the second phase<sup>1,2</sup>. If the material is exposed to water during the first 15min of setting, a superficial surface softening, probably caused by an inhibition of the reaction in the superficial layer of the glass-ionomer cements is observed<sup>3</sup>. One method of reducing the dependence of early water uptake was the development of the "set-on-command" glass-ionomer materials, based on admixing light-curable hydrophilic resins<sup>1,4</sup>.

Despite the advantage of easier handling, the resin-modified glass-ionomer cements also have some disadvantages associated with the presence of resin; such as swelling in aqueous media<sup>5</sup>, toxicological problems related to monomer release, and poor long-term mechanical properties compared to those of conventional glass-ionomer cements<sup>6</sup>.

Although ultrasonic treatment was used initially to decrease the number of air bubbles in the glass-ionomer material<sup>7</sup>, recent studies were conducted with the aim of accelerating the setting reaction and improving the mechanical properties<sup>8,9,11-14</sup>. Since an effect of ultrasonic treatment on glass-ionomer cements is an increase in temperature by approximately 13°C, the chemical reaction is substantially intensified thus enabling a typology of 'command' set of conventional glass-ionomer cements<sup>9</sup>. It was reported by Fagundes et al.<sup>10</sup> that ultrasonic treatment after 24 h increased the tensile bond strength of high-viscosity, conventional, and resin-modified glass ionomers to dentin<sup>10</sup>. Moreover the rise

in temperature might account for some water evaporation and thus increase of the powder/liquid ratio. It has also been observed that ultrasonic treatment of conventional glass-ionomer cements improved their mechanical properties<sup>8,9,11-14</sup>. The aim of this study was to assess the influence of ultrasonic treatment on the microhardness both of conventional and of highly viscous glass-ionomer cements during different storage periods.

## Material and methods

### *Sample preparations*

The restorative glass-ionomer cement products used in this study are listed in Table 1. All specimens were prepared at a room temperature of 23±1°C and a relative humidity of 50±5% in conformance with ISO 9917-1:2003 specifications<sup>15</sup>. Mixing was performed according to the respective manufacturer's instructions. Where mechanical mixing was required, an Ultramat 2 (SDI, Bayswater, Australia) was used. For each glass-ionomer formulation, ten cylindrical specimens (2mm in diameter and 2mm in thickness) were made for each evaluation period. Freshly mixed pastes of each sample were packed into the poly-tetra-fluoroethylene mold using a Centrix syringe (Centrix Incorporated, Shelton, USA), and slightly overfilling the matrix. Immediately after filling the mold was filled, ultrasonic energy was applied to the specimens in the experimental group for 15s with a frequency of 25-30 kHz using an EMS FT-081DN Mini PIEZON (EMS Nyon, Geneva, Switzerland) with an instrument B tip (Figure 1 and 2). This instrument has a flat shape with 10mm of length and 2mm of width. It is used in clinic for gross supragingival deposit removal

**Table 1.** Materials used in this study

Materials	Manufacturer	Batch no.	Color	Expiry Date	P:L ratio
Fuji IX	GC Dental Corp., Tokyo, Japan	0309051	A3	09/2006	3.6:1
Ketac Molar	3M-ESPE, Seefeld, Germany	158458	A3	01/2006	2.9:1
Bioglass R	Biodinâmica, Ibiporã, Brazil	157/04	Uni- versal	03/2006	0.18:0.06g
Vitro Molar	DFL, Rio de Janeiro, Brazil	0306548	Uni- versal	04/2006	3:1
Riva Self Cure	SDI Limited, Bayswater, Australia	88422/7	A1	11/2005	Capsule
Riva Self Cure	SDI Limited Bayswater,Australia	88142/1	A3	09/2005	3.36:1
Ionofil Plus AC	VOCO, Cuxhaven,Germany	441674	A3	10/2005	Capsule
Ionofil Plus	VOCO, Cuxhaven, Germany	421034	A1	09/2007	2.4:0.5
Maxxion R	FGM-Produtos Joinville, Brazil	280104	A1	09/2007	0.17:0.06g

and it can also be used for the removal of orthodontic cements. Owing to its shape, it was possible to apply the ultrasonic tip above the restorative material.

After the ultrasonic treatment in the experimental group and immediately after the material was packed in the control group, the samples were covered with celluloid strips. The material was allowed to set for 15min under a load of 150g at  $23^{\circ} \pm 1^{\circ}C^{16}$  and protected with petroleum jelly.

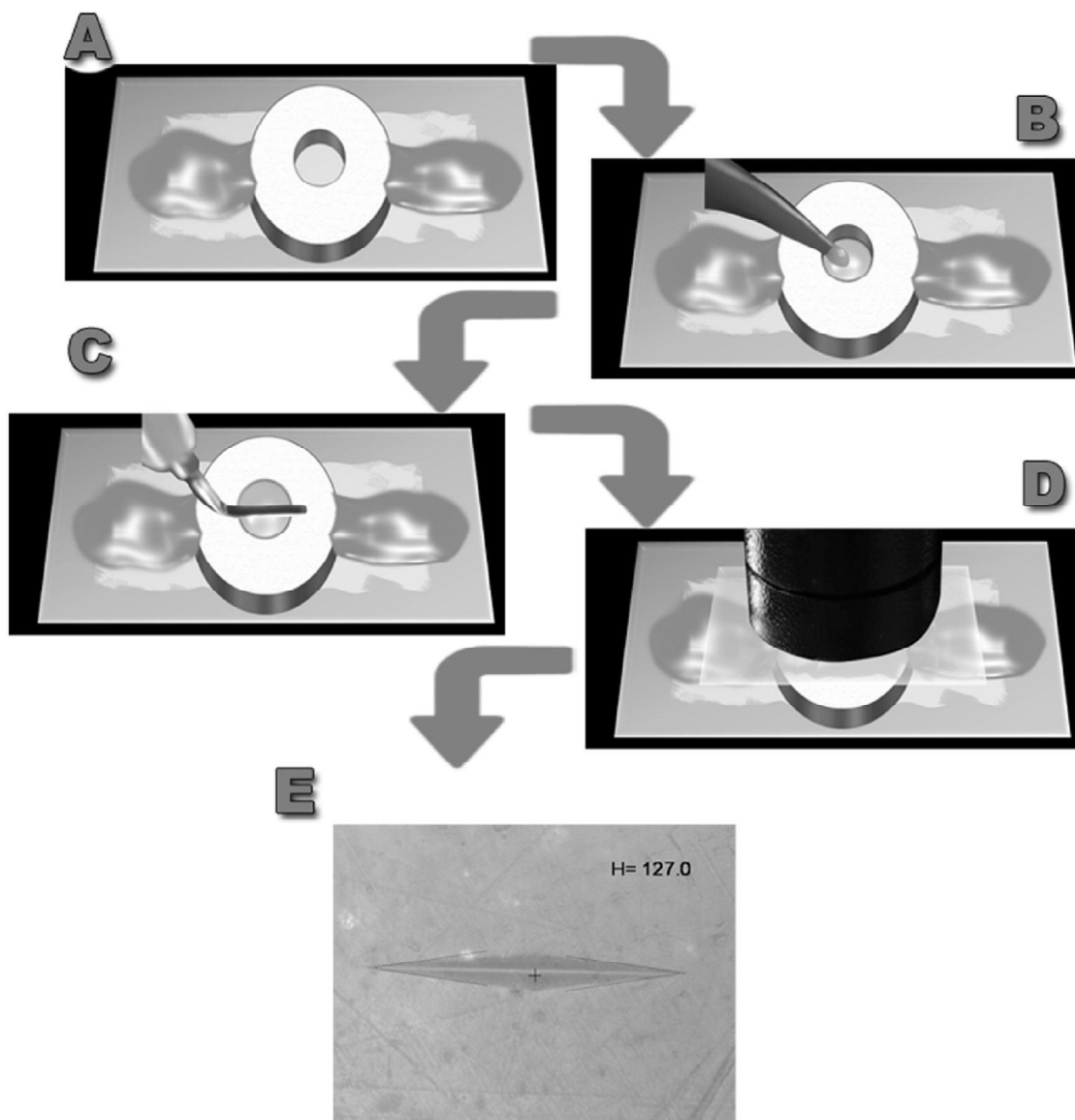
The samples with storage periods of 15min and 1h were kept at  $23^{\circ}C$  and a relative humidity of  $50 \pm 5\%$ . The samples to be measured after 12h and 24h were stored at  $37^{\circ}C$  at 100% relative humidity.

#### *Microhardness measurements*

The microhardness was determined on the basis of Knoop Hardness Number (KHN) using a Shimadzu (HMV-2, Shimadzu Corporation, Kyoto, Japan). Five indentations were made on the flat surface of each specimen and a 50g load was applied for 5s at  $23^{\circ}C$ . The hardness indentation was measured through a video control connected to a light microscope and readings were automatically converted to KHN.

#### *Statistical analysis*

The KHN data obtained were analyzed using SPSS statistical software package 14.0 (SPSS Inc., Chicago, IL, USA). T-test was used to identify statistical difference between the ultrasonic treated and non-treated groups with the significance level set at  $p < 0.01$ .



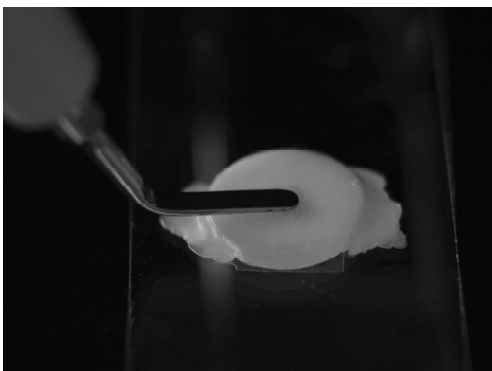
**Figure 1.** Sample preparations: A: matrix for building the specimen; B: insertion of glass-ionomer; C: application of ultrasonic device; D: application of load (150g); E: glass-ionomer specimen after microhardness indentation.

## Results

The mean values and corresponding standard deviations of the KHN measurements as a function of the pre-treatment of the samples are summarized in Table 2. At 15 min, most of the materials were still too soft to be tested, with the exception of Fuji IX and Ketac Molar.

At all storage times, there were statistically significant differences in KHN values for Fuji IX and Ketac Molar with the application of the ultrasonic treatment, ( $p < 0.01$ ). After 1h, 12h and 24h of storage, there were statistically significant Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus and effects of ultrasonic treatment for Maxxion R ( $p < 0.01$ ).

There were statistically significant differences in KHN values for Bioglass R and Vitro Molar after 1h and 12h ( $p < 0.01$ ), but no statistically significant effects of ultrasonic treatment could be demonstrated after 24h of storage ( $p \geq 0.01$ ).



**Figure 2.** Ultrasonic tip being applied to a specimen

## Discussion

Owing to the slow setting process of Bioglass R, Vitro Molar, Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus and Maxxion R, it was not possible to determine microhardness during the first 15min after mixing. Only the highly filled and consequently highly viscous Fuji IX and Ketac Molar demonstrated sufficient resistance to allow permanent indentations.

All experiments were performed at a power position 8 on scale of 1 to 10. When ultrasonic application exceeded 15s, or the power was higher than 8 on the scale, cracks invariably occurred on the material surface. On the other hand, with ultrasonic application for a time span shorter than 15s and at a power lower than 8, no positive effects could be observed.

The ultrasonic wave propagation depends on the transmission properties of the medium<sup>17</sup>. The size and shape of the mold were designed to simulate a clinical restoration according to Towler et al.<sup>12</sup> the mold was built

with poly-tetra-fluor-ethylene to simulate dentin. A study into ultrasonic wave propagation in a phantom tooth revealed the following velocities (m/s): enamel – 3100, dentin – 1900, pulp – 1570, gold – 3240 and amalgam – 2260<sup>18</sup>. In addition, the ultrasonic wave propagation in a poly-tetra-fluor-ethylene material is 1.518m/s with variation of 0.3%<sup>19</sup>. In future studies more concern could be given to the mold material in order to optimally simulate the tooth structure.

Factors such as the integrity of the interface between the glass particles and the matrix, as well as particle size play an important role in the mechanical properties of glass-ionomer cements. Increasing the powder to liquid ratio<sup>20</sup>, the poly-acid concentration<sup>20</sup> or the molecular weight of the poly-acid<sup>21</sup> are established methods for improving the physical properties of glass-ionomer cements<sup>22</sup>. Increased viscosity can be obtained by mixing the poly-acid in a dried form with the glass powder. The maximum strength was obtained when the poly-acid content in the powder was in the range of 7-9%<sup>22</sup>.

The early measurable KNH values indicate that sonication confers a characteristic “command” setting to glass-ionomer cements. This effect is stronger with highly viscous materials as they have demonstrated sufficient resistance to being permanently indented during the first 15 min of the setting process. This may be due to the different sizes and shapes of glass particles dispersed in the matrix, allowing more efficient packing and thereby resulting in a denser material and the highly integrated glass particle–polyacid matrix resulting in higher hardness. Several factors such as chemical composition and the size of the glass particles could have influenced the impossibility of obtaining results

**Table 2.** Microhardness (KHN) of glass ionomer cements, as a function of treatment and time

Materials	Time	Traditional (n=25)		Ultrasonic (n=25)		P value
		Mean	SD	Mean	SD	
Ketac Molar	15 min	34.77 <sup>A</sup>	4.99	42.11 <sup>B</sup>	7.04	P<0.01*
Molar	1 h	39.44 <sup>A</sup>	3.64	52.52 <sup>B</sup>	8.00	P<0.01*
	12 h	81.55 <sup>A</sup>	6.94	99.19 <sup>B</sup>	2.39	P<0.01*
	24 h	89.27 <sup>A</sup>	11.06	120.77 <sup>B</sup>	14.49	P<0.01*
	15 min	34.80 <sup>A</sup>	4.28	40.44 <sup>B</sup>	3.48	P<0.01*
Fuji IX	1 h	40.02 <sup>A</sup>	6.09	48.15 <sup>B</sup>	5.15	P<0.01*
	12 h	70.03 <sup>A</sup>	5.69	104.51 <sup>B</sup>	8.03	P<0.01*
	24 h	83.66 <sup>A</sup>	10.27	102.98 <sup>B</sup>	13.9	P<0.01*
	15 min	-	-	-	-	-
Bioglass R	1 h	32.98 <sup>A</sup>	1.92	40.22 <sup>B</sup>	5.69	P<0.01*
	12 h	33.50 <sup>A</sup>	2.46	37.32 <sup>B</sup>	3.22	P<0.01*
	24 h	36.69 <sup>A</sup>	4.62	39.43 <sup>B</sup>	5.95	P=0.07
	15 min	-	-	-	-	-
Vitro Molar	1 h	31.87 <sup>A</sup>	1.14	35.16 <sup>B</sup>	3.20	P<0.01*
	12 h	34.41 <sup>A</sup>	2.43	46.07 <sup>B</sup>	6.80	P<0.01*
	24 h	47.41 <sup>A</sup>	6.67	53.22 <sup>B</sup>	8.66	P=0.01
	15 min	-	-	-	-	-
Ionofil Plus	1 h	36.90 <sup>A</sup>	2.00	44.74 <sup>B</sup>	4.19	P<0.01*
	12 h	44.35 <sup>A</sup>	2.70	59.82 <sup>B</sup>	4.89	P<0.01*
	24 h	62.34 <sup>A</sup>	8.47	82.99 <sup>B</sup>	9.24	P<0.01*
	15 min	-	-	-	-	-
Ionofil Plus AC	1 h	36.68 <sup>A</sup>	2.66	48.67 <sup>B</sup>	5.74	P<0.01*
	12 h	45.88 <sup>A</sup>	2.68	55.32 <sup>B</sup>	2.57	P<0.01*
	24 h	74.30 <sup>A</sup>	8.07	101.34 <sup>B</sup>	14.33	P<0.01*
	15 min	-	-	-	-	-
Rifa Self Cure (capsule)	1 h	39.51 <sup>A</sup>	2.99	60.06 <sup>B</sup>	6.74	P<0.01*
	12 h	75.46 <sup>A</sup>	9.06	97.84 <sup>B</sup>	10.52	P<0.01*
	24 h	90.12 <sup>A</sup>	8.03	107.73 <sup>B</sup>	7.23	P<0.01*
	15 min	-	-	-	-	-
Rifa Self Cure (hand-mix)	1 h	39.65 <sup>A</sup>	4.00	55.17 <sup>B</sup>	5.18	P<0.01*
	12 h	70.32 <sup>A</sup>	6.05	93.29 <sup>B</sup>	8.82	P<0.01*
	24 h	86.67 <sup>A</sup>	6.55	109.40 <sup>B</sup>	6.19	P<0.01*
	15 min	-	-	-	-	-
Maxxion R	1 h	70.20 <sup>A</sup>	3.05	99.08 <sup>B</sup>	2.72	P<0.01*
	12 h	85.64 <sup>A</sup>	9.37	108.96 <sup>B</sup>	12.11	P<0.01*
	24 h	42.61 <sup>A</sup>	5.21	61.89 <sup>B</sup>	3.69	P<0.01*
	15 min	-	-	-	-	-

Subscript letters show differences within the same rom (p<0.05)  
Asteriks (\*) indicate statistically significant difference

for Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus, Vitro Molar and Maxxion R after 15 min. It is certain that the differences in composition, viscosity and the incorporation of porosity have been proven to influence the microhardness of glass-ionomer cements<sup>24</sup>.

Microhardness could be attributed to the powder to liquid ratios of the respective cements. However, this assumption is not supported by the results of this study, as Ketac Molar with a powder to liquid ratio of 2.9:1 allows microhardness determinations at 15 min, while Vitro Molar with a powder to liquid ratio of 3:1 was too weak to be measured for microhardness at 15min. This observation is in agreement with the findings of van Duinen et al.<sup>24</sup>.

The chemical effects obtained with ultrasonic treatment were primarily due to acoustic cavitation, which causes bubble collapse in liquids and results in an enormous concentration of energy from the conversion of the kinetic energy of the liquid motion into heating of the bubble contents<sup>25</sup>. The high local temperatures and pressures combined with extraordinarily rapid cooling provide a unique means of driving chemical reactions under extreme conditions.

A diverse set of ultrasonic applications has been explored for the purpose of enhancing chemical reactivity, with important uses in synthetic materials chemistry. Ultrasonic cavitation in liquid-solid systems also produces high-energy phenomena.

The physical effects primarily responsible for such enhancements include the improvement of mass transport from turbulent mixing, the generation of surface shock waves and micro jets and the generation of high-velocity antiparticle collisions

having a de-clustering effect on the particles. Consequently, the particles are often clogged to each other, and the fragmentation of friable solids increases the surface area<sup>26</sup>. The addition of kinetic energy from sonication to the material could improve the rate of reaction due to the temperature increase. Since the temperature is high, the powder to liquid ratio could increase, due to the liquid evaporation that usually results in high strength of materials<sup>8,9</sup>.

Additionally, the high frequency vibration of the material could decrease the volume and number of voids intrinsically present in the cements, allowing better and more efficient packing, resulting in a more dense material<sup>8,9,11-14</sup>. The characteristics of glass-ionomer cements include the development of voids during mixing. Porosity studies report that the surface area of air ranges from 6 to 9%<sup>27</sup> and the volume ranges from 1.3% to 2%<sup>7</sup>. Consequently, the decrease in porosity increases the contact between the glass-ionomer cements and dentin. This provides another explanation for the results reported in this study.

In conclusion, the chemical and physical effects mentioned above promote benefits that could enhance the setting time of glass-ionomer cements. A comprehensive scientific understanding of the relationships between glass composition, ultrasonic treatment and physical properties of glass-ionomer cements is necessary. In addition, the knowledge of the clinical effects of ultrasonic treatment on the glass-ionomer cements and the surrounding tooth structures is essential for effective clinical application.

## **Conclusion**

From this study, it can be concluded that ultrasonic command setting of

conventional glass-ionomer cements substantially increases microhardness. From a clinical viewpoint, accelerated surface hardening might reduce the early weakness of the glass-ionomer restorations. The results of this study underscore the need to explore the application of this technique *in vivo*.

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### 摘要

本研究的目的在于评估超声波治疗对于玻璃离子粘合剂的微硬度的影响。9种可以在市场上买到的玻璃离子粘合剂接受了评估: Fuji IX、Ketac-Molar、Riva Self Cure (成品胶囊和手工混合)、Ionofil Plus AC、Ionofil Plus、Maxxion R、Bioglass R 和 Vitro Molar。为每种材料和每个评估阶段都制作了10个圆柱形的样品(直径2mm, 厚度2mm)。对于试验组, 使用一台EMS FT-081DN Mini PIEZON装置对未镶嵌的样品应用超声波处理15秒, 然后以赛璐珞条带覆盖。样品在23°C暴露于150g下15分钟, 并置放15分钟、1小时、12小时及24小时。Knoop硬度数值在施加50g压力5秒钟形成的凹痕上测取。除了Bioglass R 和 Vitro Molar在置放24小时之后的情形外, 所有参与评估的材料都显示了超声波处理对于微硬度的显著效果。在15分钟阶段, 多数样品仍然过软难以测试。除 Riva Self Cure 之外, 最早的可测读的Knoop硬度数值(采用超声波处理)比置放时间较其更长的读数(无超声波处理)要高或相似。结论: 超声波控制镶嵌改进了玻璃离子粘合剂的微硬度。临床意义: 超声波处理加速了表面硬化, 这有可能降低玻璃离子修补物的早期软弱程度。首次发表于 *Int Dent S Afric* 2007; 9: 24-32.

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### Resumen

La meta de este estudio era de evaluar la influencia del tratamiento ultra-sónico sobre la micro-dureza de los cementos

vidrio-ionomer. Nueva marcas disponibles comercialmente fueran evaluadas: Fuji IX, Muela-Ketac, Auto-Cura Riva (cápsula y mezcla de mano), Ionofil Plus AC, Ionofil Plus, Maxxion R, Biovidrio R y Muela Vitro. Diez muestras cilíndricas (2mm en diámetro y 2mm en espesor) fueran hechas para cada material y para cada período de evaluación. Para el grupo experimental, se aplicó un tratamiento ultra-sónico a las muestras no colocadas durante 15s empleando un aparato de EMS FT-081DN Mini PIEZON, y cubierto posteriormente con tiras de celuloide. Las muestras estaban expuestas a unos 150g a 23°C durante 15 min. y guardadas en duraciones de 15 min., 1h, 12h y 24h. El Número Knoop de Dureza fue determinado sobre unas depresiones, hechas por aplicar una carga de 50g durante 5s. Los efectos de estadísticas significativas del tratamiento ultra-sónico en micro-dureza podía ser demostrados para todos los materiales evaluados, excepto los Bio-vidrio R y Muela Vitro después de 24h de almacenaje. En 15 min., las muestras por la mayoría estaban aún blandas demasiado a probar. A excepción de la Auto Cura Riva, los valores mensurables el más pronto del Número Knoop de Dureza (con aplicación ultra-sónica) se encontraran superiores o semejantes a esos valores obtenidos (sin aplicación ultra-sónica) para los tiempos subsiguientes de almacenaje. Conclusión: El ajuste ultra-sónico de control mejoró la micro-dureza de los cementos de vidrio-ionomer. Significativo clínico: El tratamiento ultra-sónico aceleró a endurecer la superficie el cual podría reducir la flaqueza de las restauraciones de vidrio-ionomer. Publicado primero en *Int Dent S Afric* 2007; 9: 24-32.

## Resumo

A meta deste estudo era avaliar a influência do tratamento ultra-sónico da microdureza dos cimentos de ionomer de vidro. Estiveram avaliados nove marcas de cimentos de ionomer de vidro disponíveis comercialmente: Fuji IX, Ketac-Molar, Riva Auto-Cura (cápsula e mistura de mão), Ionofil Plus AC, Ionofil Plus, Maxxion R, Biovidrio R e Esmalte de Molar. Dez espécimes cilíndricos (2mm em diâmetro e 2mm em espessura) foram feitos para cada matéria e para cada período de avaliação. Para o grupo experimental, um tratamento ultra-sónico esteve aplicado aos espécimes não fixos para 15s fazendo uso dum aparelho de EMS FT-081DN Mini PIEZON e coberto posteriormente com tiras de celulóide. As amostras estiveram espostas a 150g aos 23° C durante 15 min. E armazenadas pelas durações de 15 min., 1h, 12h e 24h. O Número Knoop de Dureza foi determinado sobre entalhes, feitos em aplicar um peso de 50g durante 5s. Uns efeitos estatisticamente significativos do tratamento ultra-sónico em micro-dureza poderian estar demonstrados por todos os materiais avaliados excepto os Biovidrio R e Esmalte de Molar depois de 24h de armazenagem. Aos 15 min, a maior parte das amostras estiveram ainda macias demais a ser postas à prova. À exceção da Riva Auto-Cura, os valores mensuráveis aos mais cedos do Número Knoop de Dureza (com a aplicação ultra-sónica) eram superiores ou parecidos com esses valores obtidos (sem aplicação ultra-sónica) para os tempos subseqüentes da armazenagem. Conclusão: A colocação do comando ultra-sónico melhorou a micro-dureza dos cimentos de vidro-ionomer.

**Significativo Clínico:** O tratamento ultra-sónico acelerou o endurecimento da superfície, o qual poderia reduzir a fraqueza cedo das restaurações do vidro-ionomer. *Publicado primeiro em Int Dent S Afric 2007; 9: 24-32.*

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