Influence of ultrasonic setting on microhardness of glass-ionomer cements

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Abstract

Objective: The aim of this study was to evaluate the influence of ultrasonic treatment on the microhardness of glass-ionomer cements. **Methods and Materials:** Nine commercially available brands of glass-ionomer cements were evaluated: Fuji IX (GC Dental Corp., Tokyo, Japan), Ketac-Molar (3M-ESPE, Seefeld, Germany) , Riva Self Cure (capsule and hand-mix) (SDI Limited, Bayswater, Australia), Ionofil Plus AC, Ionofil Plus (VOCO, Cuxhaven,Germany), Maxxion R (FGM–Produtos, Joinville, Brazil), Bioglass R (Biodinâmica, Ibiporã, Brazil) and Vitro Molar (DFL, Rio de Janeiro, Brazil). 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 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. **Results:** Statistically significant effects of ultrasonic treatment in microhardness could be demonstrated for all the materials evaluated after 24h of storage, with the exception of Bioglass R and Vitro Molar. **Discussion:** At 15min, most of the samples were still too soft to be tested. Chemical and physical effects obtained with ultrasonic treatment confer a characteristic "command" setting to glass ionomer cements. These effects may promote benefits that could enhance the setting time. **Conclusion**: Ultrasonic command setting improved the microhardness of the glass-ionomer cements. **Clinical Significance:** Ultrasonic treatment accelerated surface hardening which might reduce early weakness of glass-ionomer restorations.

Keywords: ultrasonic, glass-ionomer cements, surface hardness, physical properties, sonication

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.[1] During the initial setting stages, the loosely bound water causes certain integrity problems.[2,3] The material is very susceptible to water uptake during the first reaction, and to dehydration in the second phase[2,3]. Should the material be 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, can be observed.[4] One method of reducing the dependence of early water uptake was the development of "set-on-command" glass-ionomer materials, based on admixing light-curable hydrophilic resins.[2,5]

Despite the advantage of easier handling, resin-modified glass-ionomer cements also have certain disadvantages associated with the presence of resin, such as swelling in aqueous media[6], toxicological problems related to monomer release, and poor long-term mechanical properties compared to conventional glass-ionomer cements.[7]

Although ultrasonic treatment was used initially to decrease the number of air bubbles in glass-ionomer material[8], recent studies were conducted with the aim of accelerating the setting reaction and improving the mechanical

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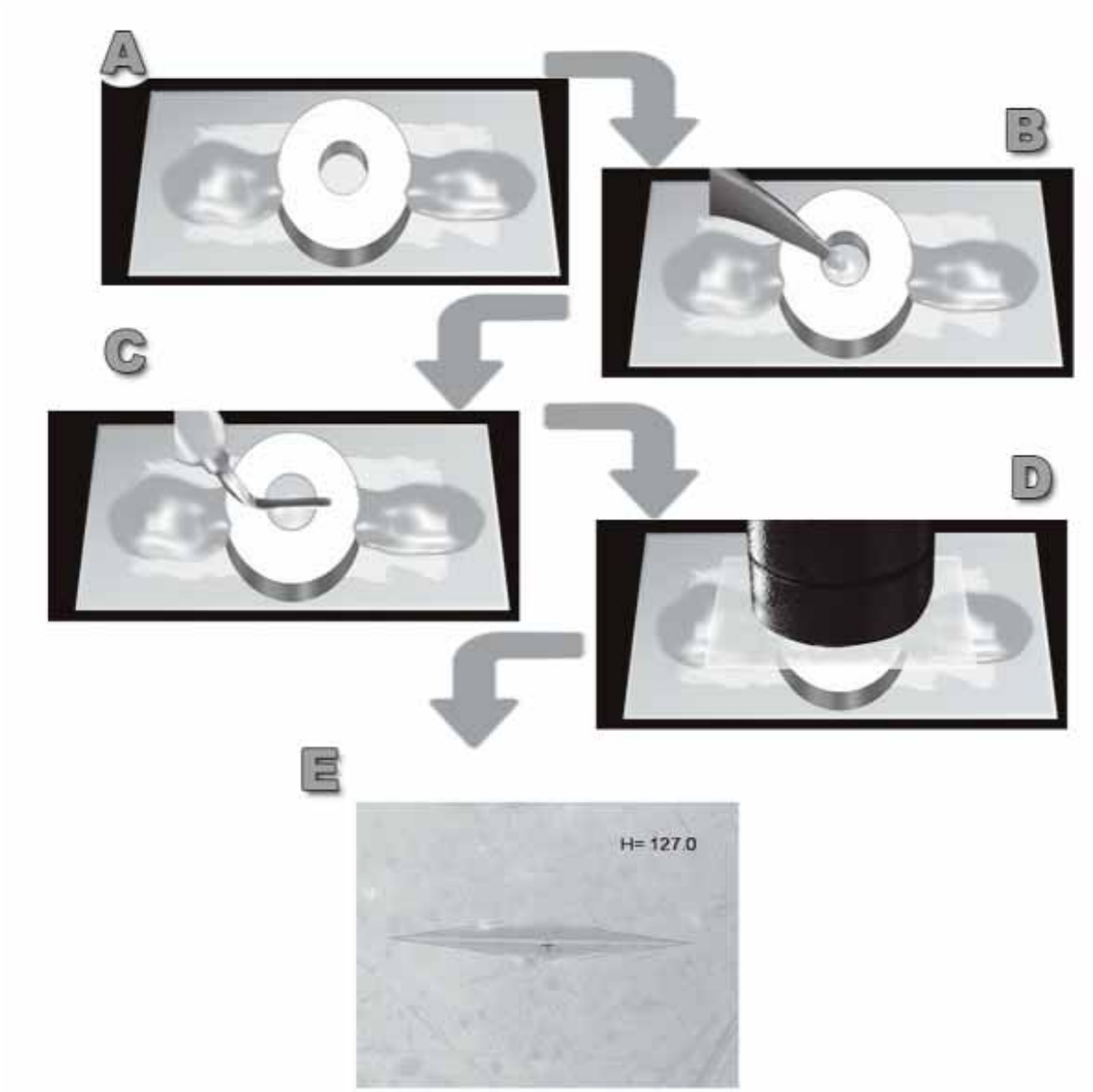

Figure 1: Sample preparations

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.

properties[9,10,12,13,14,15]. Since the 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.[10] Fagundes et al [11] reported that ultrasonic treatment after 24 h increased the tensile bond strength of high-viscosity, conventional, and resin-modified glass ionomers to dentin.[11] 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 its mechanical properties.[9,12,10,13,14,15]

The aim of this study was to assess the influence of ultrasonic treatment on the microhardness of both conventional and highly viscous glass-ionomer cements during different storage periods.

Methods and Materials

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.[16]

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

Figure 2. Ultrasonic tip being applied to a specimen.

made for each evaluation period. Freshly mixed pastes for each sample were packed into the poly-tetra-fluor-ethylene mould using a Centrix syringe (Centrix Incorporated, Shelton, USA), slightly overfilling the matrix. Immediately after filling the mould, 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 (Figures 1 and 2). This instrument has a flat shape, 10mm in length and 2mm in width. It is used clinically for gross supragingival deposit removal and can also be used for the removal of orthodontic cements. Due 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°±1°C[17] and protected with petroleum jelly. The samples with storage periods of 15min and 1h were kept at 23°C and a relative humidity of 50±5%. The samples to be measured after 12h and 24h were stored at 37°C at 100% relative humidity.

Microhardness measurements

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°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 a 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 $p<0.01$.

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 15min, 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 effects of ultrasonic treatment for Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus and 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 ($p\geq0.01$).

Discussion

Due 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 a 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. Conversely, with ultrasonic application for a time span shorter than 15s and at a power

Table I. 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	Universal	03/2006	0.18:0.06g
Vitro Molar	DFL, Rio de Janeiro, Brazil	0306548	Universal	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

lower than 8, no positive effects could be observed.

The ultrasonic wave propagation depends on the transmission properties of the medium.[18] The size and shape of the mould were designed to simulate a clinical restoration according to Towler et al[13]. The mould 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.[19] In addition, the ultrasonic wave propagation in a poly-tetra-fluor-ethylene material is 1.518m/s with variation of 0.3%.[20] In future studies more concern could be given to the mould 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,[21] the poly-acid concentration[21] or the molecular weight of the poly-acid[22] are established methods for improving the physical properties of glass-ionomer cements.[23] Increased viscosity can be obtained by mixing the poly-acid in a dried form with the glass powder. Maximum strength was obtained when the poly-acid content in the powder was in the range of 7-9%[23].

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 be permanently indented during the first 15min 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, thereby resulting in a denser material and the highly integrated glass particle–polyacid matrix resulting in higher hardness.[24]

Several factors such as chemical composition and the size of the glass particles could have influenced the failure to obtain results for Riva Self Cure (capsule and hand-mix), Ionofil Plus AC, Ionofil Plus, Vitro Molar and Maxxion R after 15min. It is evident that the differences in composition, viscosity and the incorporation of porosity have been proven to influence the microhardness of glass-ionomer cements.[25]

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 15min, 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.[25]

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

Superscript letters show differences within the same row (p<0.05)
Asterisks (*) indicate statistically significant difference

The chemical effects obtained with ultrasonic treatment are 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.[26] 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 movement from turbulent mixing, the generation of surface shock waves and micro jets, the generation of high-velocity antiparticle collisions having a de-clustering effect on the particles. Consequently, the particles are often clogged together, and the fragmentation of friable solids increases the surface area.[27] 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.[9,10]

Additionally, the high frequency vibration of the material could decrease the volume and number of voids intrinsically present in the cements, allowing a better and more efficient packing, resulting in a more dense material.[9,10,12,13,14,15] 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%[28] and the volume ranges from 1.3% to 2%[8]. 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 aforementioned chemical and physical effects 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 are necessary. In addition, 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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