

Effect of ultrasonic excitation on the ultimate tensile strength of glass ionomer cements after different water storage times

Efeito da excitação ultrassônica na resistência máxima à tração de cimentos de ionômero de vidro, após diferentes períodos de armazenamento

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Resumo

Introdução: A aplicação de ondas ultrassônicas no cimento de ionômero de vidro acelera a velocidade da reação de presa inicial e melhora as propriedades mecânicas do material. **Objetivo:** Este estudo avaliou a resistência máxima à tração de cimentos de ionômero de vidro após excitação ultrassônica e tempos diferentes de armazenamento de água. **Material e método:** Doze corpos de prova de cada material (Fuji IX GP, Ketac Molar Easymix e Vitremer) foram preparados e seis receberam aplicação de ultrassom por 30 segundos durante a reação de presa inicial. Após armazenamento de 24 horas ou 30 dias, foram seccionados em espécimes na forma de palito e submetidos ao teste de microtração. Os valores médios de resistência à tração foram avaliados pela análise de variância e teste de Tamhane com correção de Welch. **Resultado:** O cimento Vitremer apresentou as maiores médias de resistência à tração. Foi observado que o tratamento com ultrassom aumentou a resistência do cimento Fuji IX GP com 24 horas de armazenamento e esta se manteve após 30 dias de armazenamento ($p < 0,05$). No grupo controle, Fuji IX GP com 30 dias armazenamento apresentou resistência à tração maior que o armazenamento de 24 horas ($p < 0,05$). **Conclusão:** O tratamento com ultrassom aumentou a resistência à tração do Fuji IX GP, no período inicial de sua maturação.

Descritores: Cimentos de ionômero de vidro; ultrassom; resistência à tração.

Abstract

Introduction: The application of ultrasound waves with a conventional dental ultrasonic scaler on glass ionomer cements surface accelerated initial setting reaction and improved the mechanical properties. **Objective:** This study evaluated the ultimate tensile strength of glass ionomer cements after ultrasonic excitation and different water storage times. **Material and method:** Twelve specimens of each material (Fuji IX GP, Ketac Molar Easymix and Vitremer) were prepared, and six of each received a 30-second ultrasound application during initial setting of the cements. After storage of the 24 hours or 30 days, the specimens were sectioned into stick to microtensile testing and the mean ultimate tensile strength values were submitted to Welch's ANOVA and Tamhane's test. **Result:** The results showed that the Vitremer presented the highest mean tensile strength. The chemically set Fuji IX GP presented significantly higher mean tensile strength after 30 days than after 24 hours of storage ($p < 0.05$). At 24 hours, the ultrasonically set Fuji IX GP presented significantly higher mean tensile strength than their counterparts set under standard conditions ($p < 0.05$). **Conclusion:** Treatment with ultrasound increased the tensile strength of Fuji IX GP in the early period of maturation.

Descriptors: Glass ionomer cements; ultrasonics; tensile strength.

INTRODUCTION

Glass ionomer cements (GIC) are materials with multiple applications in dentistry due to their chemical adhesion to dental substrates, biocompatibility and fluoride-releasing property. However, as all dental materials, GIC also have limitations, such as, lack the fracture strength, poor abrasion resistance,

maintenance water balance, and their characteristics should be understood in order to achieve optimal results^{1,2}.

The initial setting reaction of GIC is a slow and long-term process, which makes these materials susceptible to premature moisture contamination within the first 24 hours after mixing.

Protection of the material's surface is required to avoid excess water uptake and leaching of ions that are important for cement strengthening. Desiccation should also be avoided because the salts contained in the material should be hydrated during the continuous setting reaction process³. Furthermore, the decrease of the setting reaction time is important to reduce the chances of moisture contamination under clinical conditions⁴ and to shorten the clinical chair time, which is an advantage when dealing with pediatric patients⁵. Therefore, several modifications in the composition of GIC have been proposed to increase the clinical success, such as incorporation of metallic particles or resin monomers, control of particle size, concentration and distribution of glass particles in the powder, and alterations in polyacid formulation in the liquid component^{1,3}.

Several studies *in vitro* have shown that the application of ultrasound waves with a conventional dental ultrasonic scaler on GIC surface accelerated initial setting reaction and improved the mechanical properties⁴⁻¹⁴. Ultrasonic excitation has been shown to increase surface hardness⁵, compressive strength^{4,6,8} and tensile strength¹⁰⁻¹³, minimize the void formation within the material^{4,5,9,14}, promote a more intimate contact between the glass particles and the polyacid, and break up particle clustering, thus offering a greater surface area for reaction⁴, without modifying the chemical composition of the GIC^{6,8}.

Ultrasonic excitation of these restorative materials can be applied easily in clinical practice because it does not require any special equipment and can be performed with conventional ultrasonic scalers, which is available in most dental clinics¹⁵. However, the literature still lacks of studies evaluating the effect of ultrasonic excitation on the GIC ultimate tensile strength (UTS). The purpose of this study was to evaluate the UTS of three GIC after ultrasonic excitation and different water storage times, using the microtensile technique.

MATERIAL AND METHOD

Two high-viscosity GIC Fuji IX GP (GC Corporation, Tokyo, Japan, batch 0508091) and Ketac Molar Easymix (ESPE Dental AG, Seefeld, Germany, batch 233717), and one resin-modified GIC (RMGIC) Vitremer (3M/ESPE, St. Paul, MN, USA, batch 0715100073) were used. The materials were mixed using the powder/liquid ratios specified in the manufacturers' instructions at room temperature of 23 ± 1 °C and relative humidity of $50 \pm 5\%$, in conformance with ISO 9917-1, 2003 specifications.

Twelve specimens of each material were fabricated using a cylindrical polyester matrix (6 mm height and 8 mm diameter), which was filled in two increments using a Centrix injector (Centrix Inc., Shelton, CT, USA). Six specimens of each material received ultrasound application during the initial setting reaction of the cement (test specimens), while the other 6 specimens set under standard conditions (control specimens).

After insertion of each increment into the polyester matrix, the test specimens received a 15-second (total of 30 s) application of ultrasound waves, using a conventional dental ultrasonic scaler (Profi III Bios; Dabi Atlante; Ribeirão Preto, SP, Brazil)

with vibration frequency of 28 KHz and 80% of total power. A flat-shaped ultrasonic tip indicated for removal of calculus in periodontics was used holding in the laterals walls of the polyester matrix. After second increment, the ultrasonic tip was applied in the laterals and upper walls of the GIC specimen. No water-cooling was used to avoid any interference in the material properties and setting reaction⁷. For the Vitremer specimens, ultrasonic excitation of each increment was followed by a 40-second light-activation with a halogen light-curing unit (Ultralux, Dabi Atlante, Ribeirão Preto, SP, Brazil), which was calibrated with 450 mW/cm^2 power density. After insertion of the second increment and before ultrasonic excitation, the material surface was covered with a polyester strip and finger pressure was applied for 2 seconds to provide complete accommodation of the material. The polyester strip was removed after 20 minutes of cement mixing and then, all specimens were coated with one layer of colorless nail polish and stored in distilled water at 37 °C for either 24 hours or 30 days. Three specimens of each (test and control specimens) were subjected to microtensile strength testing after each of the storage times.

For the microtensile strength test, the specimens were taken to a precision cutting machine (Isomet 1000; Buehler Ltd., Lake Bluff, IL, USA) with a water-cooled 0.5-mm-thick diamond saw (Diamond Wafering Blade, Buehler Ltd., Lake Bluff, IL, USA), rotating at 250 rpm under 200 gf load and serial 1.0- μm -thick sections were cut vertically. Then, the specimens were rotated 90° and a new series of 1.0- μm -thick sections were done producing sticks with a cross-sectional area of approximately 1.0 mm².

The sticks were carefully examined with a light microscope (Carl Zeiss, Jena, Germany) at $\times 30$ magnification, it were excluded those with defects or air bubbles were discarded. The cross-sectional area of each selected stick was measured with a digital caliper to the nearest 0.01 mm (Model 500-144B, Mitutoyo Sul Americana Ltda., São Paulo, SP, Brazil) for further calculation of the mean UTS values. A mean cross-sectional area of $0.91 \text{ mm} \pm 0.1$ was obtained. Each stick was individually fixed to a custom-made microtensile testing jig with cyanoacrylate ester adhesive (Super Bonder Gel e Ativador 7456; Henkel Loctile Ltda, São Paulo, SP, Brazil) and tested in tension in a mechanical testing machine (MTS 810; MTS System Corporation, Eden Prairie, MN, USA) set with a load cell with maximum capacity of 1 kN and running at a crosshead speed of 0.5 mm/min until failure. The mean UTS values were calculated by dividing the maximum load at failure by the cross-sectional area of the specimen and expressed in MPa.

The effect of ultrasonic excitation on the mean UTS values of the GIC after different storage times was analyzed by three-way ANOVA with Welch's correction (*material* \times *treatment* \times *storage time*). The Tamhane's test was used for pair wise comparisons of the means. A significance level of 5% was set for all analyses.

RESULT

The analysis of data in Table 1 shows that the chemically set Fuji IX GP specimens presented significantly higher mean UTS values after 30 days (control group 30d = 16.59 ± 5.35 MPa) than

Table 1. Ultimate tensile strength for the three glass ionomer cements with (US) or without ultrasonic excitation treatment (control) and after 24 hour or 30 day water storage*

GIC	Treatment	Storage time			
		24 hours		30 days	
Fuji IX GP	Control	n	33		26
		Mean (SD)	2.82 (0.77)	a*	16.59 (5.35)
	US	n	38		30
		Mean (SD)	15.44 (4.85)	bc	17.90 (5.11)
Ketac Molar	Control	n	26		31
		Mean (SD)	12.59 (3.40)	b	14.26 (1.91)
	US	n	38		31
		Mean (SD)	15.34 (4.22)	bc	13.88 (2.90)
Vitremer	Control	n	26		33
		Mean (SD)	22.65 (6.06)	d	23.11 (6.18)
	US	n	26		25
		Mean (SD)	19.82 (6.64)	cd	24.81 (6.00)

*The values are presented in as means and standard deviation (MPa). Same letters in the columns indicate no statistically significant difference among the materials within the same period. The asterisks indicate statistically significant difference between the periods.

after 24 hours (control group 24 hours = 2.82 ± 0.77 MPa) of water storage ($p \leq 0.001$). At 24 hours, the ultrasonically set Fuji IX GP specimens (ultrasonic group 24 hours = 15.44 ± 4.85 MPa) presented significantly higher mean UTS values than their counterparts set under standard conditions (control group 24 hours = 2.82 ± 0.77 MPa) ($p \leq 0.001$). However, no statistically significant difference was found at 30 days (ultrasonic group 30 days = 17.90 ± 5.11 MPa) ($p = 0.960$). For the other GIC, no statistically significant increase in the mean UTS values was observed after ultrasonic excitation at either of the experimental periods, and neither higher mean UTS values were observed as a function of time ($p > 0.05$). Regarding the nature of the materials, the RMGIC (Vitremer) presented the highest mean UTS values regardless of the ultrasonic excitation and storage period (control group 24 hours = 22.65 ± 6.06 MPa; ultrasonic group 24 hours = 19.82 ± 6.64 MPa; control group 30 days = 23.11 ± 6.18 MPa; ultrasonic group 30 days = 24.81 ± 6.00 MPa) ($p \leq 0.001$).

DISCUSSION

The microtensile bond strength test was introduced by Sano et al.¹⁶ in the 1990's and has been used to evaluate not only the bond strength of adhesive materials to the dental substrates, but also the UTS of dental materials and tooth structures¹⁷⁻¹⁹. It uses small specimens, to minimize presents failure and bubble at the tooth or restoration. It is also, indicated for materials with relatively low bond strengths, such as GIC¹⁷.

High-viscosity GIC, such as Fuji IX GP and Ketac Molar Easymix, presents small-sized glass particles and high powder/liquid

ratio, while the RMGIC, such as Vitremer has approximately 5% of resin monomers in its composition¹ and a homogenous distribution of smaller and larger glass particles¹⁹. The incorporation of light-activated resin monomers, the increase of powder/liquid mixing ratio and the reduction of glass particle size in these materials are strategies to accelerate the setting reaction and to improve their mechanical properties^{20,21}. Fast setting of conventional GIC can also be achieved by the application of an external energy source, such as ultrasound waves from a dental ultrasonic scaler^{5,6}.

Although its exact action mechanism remains unclear, it is suggested that ultrasonic excitation promotes a more homogenous mixture between the glass particles and the polyacid, increasing the particle rate dissolution and the ionic diffusion through the liquid, and accelerates the crosslinking of the polyalkenoic acid chains. In addition, the vibration of the material at a high frequency shortly after its insertion improves the compaction⁹. Towler et al.⁵ reported that GIC improved the mechanical properties and presented shorter initial setting time after the application of ultrasound waves. The authors verified an increase in the surface hardness after ultrasonic excitation for 10 s with 75% of total power.

In this study, it was observed that the application of ultrasound waves did not alter the UTS of the GIC, except for Fuji IX GP 24 hours after mixing. A previous study evaluating the mechanical properties of ultrasonically set GIC¹² have reported that 15-second ultrasonic excitation resulted in a significant increase in the compressive strength of all tested GIC, however had no influence on the diametral tensile strength, except for Fuji IX GP after 24-hour storage. In the present study, the Fuji IX GP specimens that were ultrasonically set and stored for

24 hours presented higher mean UTS than those of the specimens without ultrasonic excitation.

Twomey et al.⁴ observed that the GIC completed the command setting phase 45 seconds after ultrasound application and presented improvements in the compressive strength 24 hours and 7 days after mixing when compared to the chemically set materials. In addition, some studies related an increase of the compressive strength of GIC when ultrasonic excitation was applied with different application times after 24 hours and 7 days of water.^{6,8,12} According to Arcoria et al.²², the increase of bending strength after sonication was only observed with the encapsulated GIC, presenting better results after 10-second of ultrasound application and after 2 weeks of water storage. Algera et al.¹¹ reported that curing of GIC with 60-second ultrasound application at full power decreased the setting reaction rate and significantly increased the bond strength to enamel. Fagundes et al.¹⁰ reported that 15-second ultrasonic excitation of each increment increased the tensile bond to dentin. In this study the UTS increased with same ultrasonic excitation time used.

Conventional GIC and RMGIC usually require several days to reach full strength. During this period, the cements are weak and the conventional GIC, in particular, are susceptible to dissolution. A faster setting reaction with ultrasonic excitation may avoid this situation because the UTS can be reached sooner¹¹. In an investigation about the ultrasonic set of GIC, Twomey et al.⁴ reported that the compressive strengths of the ultrasonically set GIC 24 hours after mixing were close to the values obtained for the chemically set specimens after 7 days, reinforcing the theory that ultrasound accelerates the setting reaction.

In the present study, half of the test specimens and half of the control specimens were subjected to microtensile testing 24 hours after mixing and the other half of test and control specimens were tested after 30 days of storage, when the cements reached

a more advanced stage of maturation, as reported elsewhere²³. However, in a standard conditions without ultrasonic excitation, no significant differences were observed between the storage times, except for the chemically set Fuji IX GP, which showed an increase in UTS with time. In a previous study evaluating the mechanical properties of GIC affected by curing methods, Kleverlaan et al.⁶ found a significant increase in the compressive strength of ultrasonically set materials, especially at the early curing time. According to the authors, ultrasonic excitation can be used as a 'command' set method and improves the properties of GIC at early setting time. However, no significant differences were observed between the storage times (24 hours and 28 days) for the GIC set under standard conditions (no ultrasound application).

The in vitro application of ultrasound waves on GIC has shown promising results in accelerating of the setting reaction and improving the mechanical properties of these materials. Ultrasonic excitation does not alter the chemical composition of the GIC, in addition further benefits of the fast set technique are shorter clinical chair time and the use of dental scalers commonly found in dental offices, which represents no additional financial outlay by the clinician.

CONCLUSION

Although literature to present that the application of the US improves some mechanical properties of the GIC, in this study this application only increased the UTS of the Fuji IX GP cement, 24 hours. In conclusion, treatment with ultrasound increased the UTS of Fuji IX GP in the early period of maturation.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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