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Effect of storage time and chlorhexidine addition on the mechanical properties of glass ionomer cements

Juliana de Carvalho Machado[1], Josânia Pitzer de Oliveira[2], Cristiane Duque[1*], Angela Scarparo[2]

[1] State University of São Paulo (Unesp), School of Dentistry, Department of Pediatric Dentistry and Public Health, Araçatuba, SP, Brazil.

[2] Federal Fluminense University (UFF), Institute of Health, Department of Dentistry, Nova Friburgo, RJ, Brazil.

Aims: To evaluate the effect of the chlorhexidine (CHX) incorporation and the storage time on the mechanical properties of glass ionomer cements (GICs). **Methods:** The following GICs were evaluated: Ketac Molar Easymix (KM), Vidrion R (VR) and Vitromolar (VM), containing or not CHX. GIC liquid was modified by adding 1.25 % CHX digluconate and then manipulated with the power and placed into the stainless steel cylindrical or bar-shaped molds. GICs specimens were stored into water for 1, 7 and 28 days. After these periods, specimens were submitted to flexural, diametral tensile and compressive strength tests, according to ISO standards. Data from mechanical tests were statistically analyzed using 2-way ANOVA and Tukey tests. **Results:** Overall, the storage time did not influence any of the mechanical properties of the GICs tested. In contrast, the inclusion of CHX reduced significantly these properties for all GICs tested. KM presented the highest values of compressive strength for all storage times. KM + 1.25% CHX had lower compressive strength results than KM, however, it showed similar results when compared to another GICs without CHX. **Conclusions:** The presence of chlorhexidine, independent of the storage time, interfered on the mechanical characteristics of GIC.

Keywords: glass ionomer cements, chlorhexidine, dental caries, water storage, mechanical properties

Corresponding author:
Cristiane Duque
Department of Pediatric Dentistry and Public Health
State University of São Paulo (Unesp)
School of Dentistry
Address: R José Bonifácio, 1193, CEP. 16015050, Araçatuba, SP, Brazil.
Tel: (+55) 1836363315.
E-mail: cristianeduque@yahoo.com.br, cduque@foa.unesp.br

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Introduction

The atraumatic restorative treatment (ART) approach consisted in the removal of infected dentin using cutting hand tools under relative isolation followed by the restoration of the cavities with conventional glass ionomer cements (GICs) as the material of choice[1-6]. This procedure is considered of low cost, easy handling and high applicability[1-3,5-7], which causes minimal discomfort to the patient by eliminating the need for local anesthesia[2,3,5]. The World Health Organization (WHO) recognized ART in 1994 as a new approach for dental treatment in regions economically less favored[8]. It has been stated in the minimum intervention philosophy for several reasons, such as the maintenance of healthy tooth structure, early intervention in the progression of caries lesions[3,7], preservation of decayed teeth without endodontic involvement, promotion of oral health education concomitant with the restorative treatment[9]. ART has contributed to the management of pediatric patients behavior mainly in the cases of non-cooperative patients and patients with special needs[3]. The use of GICs for ART approach is due to their intrinsic properties, such as adhesion to the dental structure, biocompatibility, coefficient of thermal expansion similar to tooth structure and fluoride release[1,3-5,7,10].

GICs more indicated for ART are those with a high proportion powder/liquid[11], mainly Ketac Molar and Fuji IX, due to improved mechanical properties for restorations in posterior teeth. Previous studies showed that the restorations longevity rates are higher when the ART of high viscosity cements are used, both in the primary dentition and in permanent[11,12]. However, high aspect ratio powder/liquid have resulted in decreased solubility and fluoride release[13].

Studies have shown that GICs have a variable antimicrobial activity, possibly related to different initial pH values, the amount of fluoride released as well as the chemical components present in each cement powder. The GICs indicated for ART have shown reduced antimicrobial activity[5,6,14,15], and for this reason, the incorporation of antimicrobial agents (chlorhexidine and antibiotics) to the cement has been suggested in several investigations[1,6,14-18], in order to increase the effect of these materials against residual microorganisms on cavities after partial caries removal. Chlorhexidine is the antimicrobial agent commonly used in Dentistry due its safety and broad-spectrum bactericidal effect, affecting the growth of gram-positive bacteria especially oral streptococci[19], gram-negative bacteria, fungi and yeasts, facultative aerobic and anaerobic species[20]. Conflicting results about the influence of the incorporation of chlorhexidine on the physical-mechanical properties of GICs are still present in the literature. Some studies have shown that the addition of chlorhexidine decreased the mechanical properties of GICs, such as the compressive strength[1,14-16], and others showed no negative effects on these properties, except for concentrations above 2% (8,17). However, the influence of the storage time on the mechanical properties of GICs containing chlorhexidine has been not studied yet. Thus, the aim of this study was to evaluate the effect of the chlorhexidine incorporation and the storage time on the mechanical properties of glass ionomer cements. The study's hypotheses were that 1) chlorhexidine incorporation affects mechanical properties of glass ionomer cements along the time and 2) water storage affects mechanical properties of glass ionomer cements containing or not chlorhexidine along the time.

Material and Methods

Dental materials

Three glass ionomer cements (GICs) were evaluated: Ketac Molar Easymix (3M/ESPE, St. Paul, USA), Vidrion R (SS White Dental Products Ltd., Rio de Janeiro – RJ, Brazil) and Vitromolar (DFL, Rio de Janeiro – RJ, Brazil). The composition of each GIC is presented in Table 1 and the distribution of the groups according to the factors studied (GIC, presence or not of CHX, storage time and mechanical test) and the number of samples per group (n) is shown in Table 2. GIC liquid was modified by adding 1.25 % chlorhexidine digluconate (CHX – C9394 Sigma–Aldrich, Steinheim, Germany), as proposed by Türkün et al.[15] and then manipulated with the power, according to each GIC manufacturer's instructions without altering liquid/powder ratio. The scoop of powder and the droop of liquid were previously weighted in an analytical balance in order to standardize the recommend powder/liquid ratio for each GIC (BIOPRECISA, São Paulo – SP, Brazil).

Each GIC containing or not CHX were mixed and placed into the stainless steel cylindrical or bar-shaped molds using a restorative dispenser (and tip, slightly overfilled and compressed with polyethylene sheets and glass slabs. After 5 minutes of the initial setting of the materials, specimens were removed from the mold and excess were removed manually using a 600-grit SiC paper. The dimensions of specimens were checked using a digital caliper (Digimatic caliper, Mitutoyo Corp., Tokyo, Japan). A colorless glaze protection (nail enamel, Revlon Inc, NY, USA) was applied on all surfaces of GIC samples, in order to inhibit the syneresis and imbibition processes. The specimens were then stored inside plates containing gauze soaked with water (relative humidity) at 37°C for 1 h. After this period, GIC samples were immersed in 10mL of deionized water in individual plastic containers and stored for 1, 7 and 28 days. Control group comprises specimens without CHX incorporation. After the storage times, samples were submitted to mechanical tests described below.

Table 1. Composition of the conventional glass ionomer cements used in this study

Cement	Code	Composition		Manufacturer
		Liquid	Powder	
Vidrion R	VR	Sulphate Na-Ca-Ba-Al-fluorosilicate, acrylic acid	Tartaric acid and water	SS White Dental Products Ltda, Rio de Janeiro, RJ, Brazil
Vitromolar	VM	Ba-Al silicate, polyacrylic acid and dehydrated zinc oxide	Polyacrylic acid, tartaric acid and distilled water	DFL. Rio de Janeiro, RJ, Brazil
Ketac Molar Easymix	KM	Polialquenoic acid, tartaric acid and water	Fluorosilicate glass Al-Ca-La copolymer (5% acrylic acid and maleic acid	3M/ESPE, St. Paul, USA

Mechanical Tests

Flexural strength

The flexural strength (FS) was measured according to the ISO Standard ISO9917-2 using 25mm length x 2mm width x 2mm height bar-shaped specimens (n=10). After storage times, the specimens were submitted to a 3-point bending test (the distance between the two supports is 20 mm) on a Universal Testing Machine (Instron no. 4442, Instron Corp, Canton, MA, USA) at a crosshead speed of 1mm/min until the fracture. The FS was calculated with the following formula: $FS=3R/2wh^2$, where R is the load required to fracture in MPa; L is the distance between the supports (20.0 mm); w is the specimen width and h is the specimen height.

Diametral tensile strength

The specimens (n=10) for diametral tensile strength (DTS) were made using cylindrical metal molds with 4mm diameter X 6mm thickness, according to the specifications of ISO Standard ISO9917-1. After the storage periods, were subjected to the DTS test on a Universal Testing Machine (Instron), at a speed of 0.5 mm/min in horizontal position until the fracture. The DTS was calculated with the following formula: DTS =2F /pdT, where F is the load required to fracture; p is 3.1416; d is the diameter and T the thickness).

Compressive strength

The compressive strength (CS) was measured under the same conditions (storage conditions, testing machine and number of specimens) with specimens with 4mm diameter and 6 mm thickness (n=10), at a speed of 1mm/min in a vertical position until the fracture, according to the specifications of ISO9917-1. The values obtained were converted into MPa using the following formula: $CS =F/1/4pd^2$, where F is the load at fracture, p is 3.1416 and d^2 the diameter in mm^2.

Statistical Analysis

Two-way ANOVA and Tukey post hoc tests ($p = 0.05$) were used to test the influence of CHX and storage time on the mechanical properties of the GICs, considering $p<0.05$. The software used was SPSS version 17.1 (SPSS Inc., Chicago, USA).

Results

The mean and standard deviation values (MPa) for flexural, diametral tensile and compressive strength are summarized in Tables 2, 3 and 4, respectively. Overall, the storage time did not influence any of the mechanical properties of the GIC tested. In contrast, the inclusion of CHX reduced significantly the mechanical properties of all GICs tested. GIC without CHX did not differ from each other for flexural and diametral tensile strength tests (Tables 2 and 3), independent on the storage time evaluated. The same was observed among GICs containing CHX. KM presented the highest values of compressive strength for all storage times. KM + 1.25% CHX had lower compressive strength results than KM, however, it showed similar results when compared to another GICs without CHX (Table 4).

Table 2. Comparison of means (SD - standard deviations) in MPa of flexural strength for the glass ionomer cements containing or not 1.25% CHX at different storage times.

Material	1 day	7 days	28 days
VR	17.08(5.62)^{Aa}	19.54(3.67)^{Aa}	19.31 (3.77)^{Aa}
VR + 1.25% CHX	5.00(2.61)^{Ba}	4.56(2.70)^{Ba}	3.95(1.85)$^{Ba.b}$
VM	20.01 (6.78)^{Aa}	16.88(10.01)^{Aa}	18.21 (7.85)^{Aa}
VM+ 1.25% CHX	6.35 (2.09)^{Ba}	6.07(2.30)^{Ba}	6.13 (2.92)^{Ba}
KM	20.79(5.81)^{Aa}	16.82(9.97)^{Aa}	19.63 (6.18)^{Aa}
KM + 1.25% CHX	5.18(2.50)^{Ba}	5.67(1.80)^{Ba}	4.49 (2.40)^{Ba}

^{A} Different upper case letters showed statistical difference between GICs containing or not CHX, according to ANOVA and Tukey tests.

^{a} Different lower case letters showed statistical difference comparing the storage time for the same GIC, according to ANOVA and Tukey tests.

Table 3. Comparison of means (standard deviations) in MPa of diametral tensile strength for the glass ionomer cements containing or not 1.25% CHX at different storage times.

Material	1 day	7 days	28 days
VR	7.60 (2.80)^{Aa}	7.42 (0.74)^{Aa}	7.11 (0.97)^{Aa}
VR + 1.25% CHX	3.31 (1.86)^{Ba}	3.50 (1.19)^{Ba}	3.82 (1.93)^{Ba}
VM	7.09 (2.68)^{Aa}	6.83(3.66)^{Aa}	6.64 (2.72)^{Aa}
VM+ 1.25% CHX	4.12 (2.08)^{Ba}	3.94(2.49)^{Ba}	4.08 (2.56)^{Ba}
KM	8.25 (3.99)^{Aa}	8.31(2.37)^{Aa}	7.54 (1.90)^{Aa}
KM + 1.25% CHX	4.48 (1.03)^{Ba}	4.39(0.63)^{Ba}	4.37 (2.09)^{Ba}

^{A} Different upper case letters showed statistical difference between GICs and GICs containing CHX, according to ANOVA and Tukey tests.

^{a} Different lower case letters showed statistical difference comparing the storage time for the same GIC, according to ANOVA and Tukey tests.

Table 4. Comparison of means (standard deviations) in MPa of compressive strength for the glass ionomer cements containing or not 1.25% CHX at different storage times.

Material	1 day	7 days	28 days
VR	10.60 (3.00)^{Aa}	10.98(3.79)^{Aa}	11.16 (2.64)^{Aa}
VR + 1.25% CHX	6.46 (2.22)^{Ba}	6.61(2.86)^{Ba}	6.71(2.21)^{Ba}
VM	10.10(2.26)^{Aa}	13.96(5.09)^{A,Cb}	11.55 (3.18)^{Aa}
VM+ 1.25% CHX	8.15(3.40)^{Ba}	8.28(3.20)^{Ba}	8.36 (3.35)^{Ba}
KM	16.76 (5.04)^{Ca}	16.36(2.96)^{Ca}	16.62 (2.68)^{Ca}
KM + 1.25% CHX	10.71 (2.48)^{Aa}	10.63(3.36)^{Aa}	10.33 (3.56)^{Aa}

^{A} Different upper case letters showed statistical difference between GICs and GICs containing CHX, according to ANOVA and Tukey tests.

^{a} Different lower case letters showed statistical difference comparing the storage time for the same GIC, according to ANOVA and Tukey tests.

Discussion

High viscosity GICs for ART technique have been introduced in the market promising better mechanical properties than conventional GICs. However, The GICs indicated for ART have shown reduced antimicrobial activity[5,6,14,15], and the incorporation of chlorhexidine could be an alternative to improve their action against residual bacteria from cavities. In addition, restorations are constantly bathed with saliva, influencing the syneresis and imbibition processes of GICs. However, the effect of water storage on the mechanical properties of these high viscosity GICs containing chlorhexidine have been not studied yet.

Deionized water, artificial or human saliva have been frequently chosen as storage media simulating intraoral conditions. In this present study, GICs were immersed in water as storage media. This choice was based in the study of Mckenzie et al.[21]. These authors reported that physical-mechanical properties of conventional and resin-modified glass ionomer cements were not significantly different comparing storage in water or saliva up to 1 year. Therefore, water was considered acceptable as the storage medium for in vitro analysis of GIC properties.

In this present study, the storage time did not influence any of the mechanical properties of GICs evaluated. Our results are according with Zoergiebel and Ilie[22] that evaluated three conventional GICs in comparison with newly developed zinc-containing GIC and demonstrated low impact of storage agent (water and artificial saliva) and storage duration (7 and 30 days) on the flexural strength, modulus of elasticity and hardness properties. Other studies reported a tendency for increasing the compressive strength after 1-week storage and remaining unchanged up to 1-year storage[21,23]. The early moisture contamination could decreases mechanical properties of GICs favoring surface erosion and abrasion. In the present study, GIC specimens were protected from the influence of water by a thin colorless glaze preventing water contamination on the initial phase of the setting to endure 24h up to 2 weeks[24].

The incorporation of chlorhexidine reduced the mechanical properties of GICs studied in the current study. Our results confirmed the findings obtained by Takahashi et al.[8], Turkun et al.[15], Palmer et al.[16], Marti et al.[25], Mittal et al.[26]. The addition of CHX may alter the powder/liquid ratio and consequently the mechanical strength of the material[6,15,17]. The form of CHX salt could also influence in the GICs properties. CHX digluconate (liquid form) is solubilized faster into oral environment than the CHX diacetate (powder form), however, both forms could hamper the chemical reaction of GICs by neutralization of polyacids to release ions from glass particles or by the formation of base/polyacid complexes that block reactions between cationic ions and polyacrylic chains, consequently increasing the setting time[27-29]. Marti et al.[25] showed that the addition of CHX at concentration of 2% resulted in significant increase on setting time and decrease on the surface hardness of high viscosity GICs. In addition, the tensile bond strength of these materials also decreased significantly after adding 2% CHX. Although study of Mittal et al.[26] observed that the incorporation of 1.5% CHX did not affect 24-h compressive strength of a high viscosity GIC, the majority of the studies are in agreement that the addition of up to 1% CHX in high viscosity GICs did not change their physico-mechanical properties[25,28,30,31].

In the present study, Ketac Molar presented the best results for compressive strength. In addition, Ketac Molar with 1.25% CHX showed similar results of compressive strength compared to another GICs without CHX. Similar results were obtained by Bonifácio et al.[1] and Algera et al.[32]. The strength of GICs is influenced by their composition. High content of fluoride or zinc increases the ability of GIC form a network with acrylic acid and decreased the setting time, induced a higher compressive and flexural strength[33]. Superior flexural and compressive strengths have been reported for Ketac Molar in comparison to Vitromolar[1], similar to those found in the present study. These GICs have been exhibited high concentration of fluoride in their composition similar to resin-modified GICs and their high viscosity allows a faster setting reaction resulting on fluoride lockup within the matrix forming a reservoir to be released later. There was no relationship between the amount of fluoride in the composition of GIC and the amount of fluoride release to the oral environment. The amount of fluoride release did not affect the changes of the compressive strength or the surface hardness up to 1-year water storage[34].

In conclusion, one of the hypotheses of this present study was rejected, because neither GIC nor GIC containing CHX reduced their properties up to 30 days of water storage. However, the presence of chlorhexidine interfered on the mechanical characteristics of GIC. Among the GIC tested, Ketac Molar, containing or not 1.25% CHX, presented the highest values of compressive strength.

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