ORIGINAL ARTICLE

Initial Mechanical Stabilization of Conventional Glass Ionomer Cements with Different Active Principles

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ABSTRACT

Objective: To determine the initial mechanical stabilization of conventional glass ionomer cements (GICs) indicated for the atraumatic restorative treatment (ART) in different storage periods. **Material and Methods:** Specimens were divided according to the GIC (n=12): IZ - Ion-Z, KM - Ketac Molar Easymix, RS - Riva Self Cure, and GL - Gold Label 9. They were prepared and stored in distilled water. Superficial microhardness (SMH) was evaluated (KHN) in three phases: (A) after 1, (B) 3, and (C) 7 days of storage. Data were submitted to 2-way ANOVA and Tukey tests (α = 5%). **Results:** The average KHN values for phases A, B, and C were, respectively, 33.05 ± 9.74; 33.21 ± 10.31 and 52.07 ± 11.75 (IZ); 50.35 ± 11.39; 66.05 ± 10.48 and 67.77 ± 13.80 (KM); 89.63 ± 15.59; 71.31 ± 23.86 and 57.70 ± 16.89 (RS); 42.18 ± 9.03; 68.54 ± 6.83 and 57.95 ± 8.24 (GL). Significant differences were observed: GIC, time, and interaction of both (p<0.05). KHN values differed between the groups, except in the GIC parameter for KM and GL. The time parameter values of phase A were lower than those of B and C, except for IZ and RS. **Conclusion:** The initial mechanical stabilization differed between the types of GIC tested and the storage time, and after the final period, all had similar SMH.

Keywords: Dental Cements; Glass Ionomer Cements; Hardness Tests; Zinc.

Introduction

Atraumatic restorative treatment (ART) is a minimally invasive technique, allowing for the maintenance of a sound dental structure through the selective removal of caries (softened dentine) with manual instruments. It also allows for the immediate restoration of cavity preparation, involving glass ionomer cement (GIC) of high viscosity, which takes advantage of its physical-chemical characteristics, manual handling, adhesive capacity, and fluoride release power that acts as an antibacterial agent [1,2]. This treatment modality reduces the number of pulp exposures, which reduces the number of endodontics and extractions, leading to less stress and anxiety for the patient since it rarely causes pain. Additionally, it does not require the use of anesthesia, rotatory instruments, or rubber dam isolation [3]. Thus, it is an economical and effective method to prevent and control caries disease in vulnerable populations [4,5].

The biggest problem with GIC is related to its maturation time due to the fact that the last phase of the material setting process is very slow, lasting more than 24 hours, as well as the extremely slow release of aluminum ions from the glass powder. This maturation is clinically related to the hardening of the material, and because it is not fully hardened, the first 24 hours after the application of this material becomes critical [3]. The GIC is susceptible to hygroscopic changes in the environment and may suffer syneresis and imbibition processes, which are the loss and gain of water to the external environment, respectively. Thus, there is a high potential for solubility with its mechanical properties and not reaching a satisfactory standard [6]. Thus, a seal of this cement is indicated right after its insertion in the oral environment, which is extremely humid, which can be carried out with cavity varnish, dental adhesives, Vaseline, or even colorless nail polish [7].

ART has been performed by professionals who are unaware of the technique or have used it in contraindicated cavities, or even with inadequate GICs [8]. GICs adhere well to the tooth structure, but fail in the mechanical properties of the material itself [9], such as compressive strength (CS), flexural strength (FS), diametrical tensile strength (DTS), fracture toughness (FT), Knoop microhardness (KHN) or Vickers (VHN), wear rate (WR), fatigue, and creep [6].

Although GIC is not a resistant material under areas of great masticatory effort [10], many attempts to improve its mechanical properties are reported in the literature. These aim to improve these materials' physical properties to use them in the posterior region with the ART technique [11]. Some of these studies attempted including zirconia hydroxyapatite inserts/particles [12], carbon nanotubes [13,14], titanium dioxide [15], and casein phosphopeptide-amorphous calcium phosphate [16]. They also tried using auxiliary methods of accelerating the setting reaction (ultrasound, heating, halogen light, and LED) [17,18] and adding zinc oxide (ZnO) nanoparticles [19]. These new materials are expensive in the Brazilian market, and national companies have been launching materials to meet these new needs.

ZnO is a compound that has been used in the form of nanoparticles in dental materials, in addition to being biocompatible and non-toxic in small concentrations [20,21]. It is believed that ZnO nanoparticles inhibit the growth of various microorganisms [22] due to the ability to change the components of the bacterial cell membrane, causing the loss of intracellular material and, consequently, cell death [23,24]. In previous studies, experimental GICs that were ZnO-based showed great potential for biomedical application. However, the mechanical properties were lower than conventional GICs that are based on aluminum silicate [25-27]. Although ZnO nanoparticles have antibacterial potential at low concentrations (0.02% to 5%) without affecting biocompatibility and physical and mechanical properties [21,28], studies that associate ZnO nanoparticles with GICs have been explored in the scientific literature [29-31].

In the Brazilian market, there is a GIC that is reinforced with self-curing zinc (Ion-Z, FGM, Joinville, SC, Brazil) with active bactericidal and dentinogenic ingredients, aiming at the control of caries disease. According to the manufacturer, the main characteristics involve high bactericidal capacity, release of fluoride ions, high mechanical resistance, high chemical adhesion to enamel and dentin, fast setting reaction, excellent viscosity and biocompatibility, less curing contraction, and high smoothness. The manufacturer recommends its use for temporary restorations in permanent teeth, as a base for permanent restorations, in non-carious cervical lesions, restorations in primary teeth, and the ART technique. It is worth mentioning that, to date, the literature evaluating the properties of this new material with bioactive properties is scarce [18,32,33].

Even though GICs have improved properties in the long-term, it is essential to understand how long it takes for these materials to stabilize mechanically since maturation does not happen immediately after handling [2,8]. This knowledge is important to support possible restorations failures (dissolution and fractures) and / or adhesives that occur with restorations made with these materials. Thus, this study is necessary to verify whether these improvements of the mechanical properties of GICs using zinc particles are indicated for ART.

The aim of this study was to determine the initial mechanical stabilization of different GICs used in the ART technique, determined by analyzing their surface hardness in different periods of storage in distilled water. The null hypotheses were that the surface microhardness of the studied GICs (including the zinc modified GIC) do not differ between them and that the analyzed times do not interfere in this property either.

Material and Methods

Pilot Study

To determine the ideal powder-liquid proportion for each type of GIC tested in the study, 10 repetitions of the powder and liquid were performed as indicated by the manufacturer and the respective weighing on an analytical precision balance (Shimadzu, Japan). Following this, the mean and standard deviation values for the powder and liquid were obtained. To provide a standardized measurement, the powder-liquid proportions shown in Table 1 were followed.

Table 1. Proportion of powder and liquid for the different types of GIC.

GIC	Powder-Liquid
Ion-Z (FGM)	1.45:1.00
Ketac Molar Easymix (3M/ESPE)	3.88:1.00
Riva Self Cure (SDI)	2.87:1.00
Gold Label 9 (GC)	3.67:1.00

Six cylindrical specimens (5 mm in diameter and 4-mm thick) of each type of GIC were also made to carry out the pilot study after 24 hours (1 day) and 72 hours (3 days) of storage in distilled water. The data from the pilot test are shown in Table 2. The data from this pilot study were used for the sample calculations of the study in question.

Table 2. Surface microhardness mean values for different GIC and storage periods – pilot test. Values in KHN.

GIC	After 1 Day	After 3 Days
Ion-Z (FGM)	39.05 ± 13.85	184.62 ± 71.38
Ketac Molar Easymix (3M/ESPE)	116.59 ± 36.29	119.08 ± 35.33
Riva Self Cure (SDI)	68.48 ± 18.06	94.47 ± 21.87
Gold Label 9 (GC)	75.21 ± 20.70	108.16 ± 23.30

Materials and Samples

Four types of GICs indicated for the ART treatment were tested: Ion-Z (FGM, Joinville, SC, Brazil); Ketac Molar Easymix (3M / ESPE, St. Paul, Minnesota, USA); Riva Self Cure (SDI Limited, Victoria, Australia) and Gold Label 9 (GC Dental Corporation, Tokyo, Japan). From the data obtained with the pilot test (mean and standard deviation), the sample calculation was performed (G * Power 3 for Mac). The following parameters were used: test power at 80%, number of groups to be tested equal to four, probability error at 0.05 and effect size (f) equal to 1.255815 (determined using pilot data), and the sample calculations of 12 specimens per group.

Specimen Preparation

For the preparation of the specimens, the powder and liquid mass of the GIC was initially determined using an analytical precision balance (Shimadzu Corp., Kyoto, Japan) to standardize the relative proportions of the GIC components [14].

The GICs were manipulated according to the manufacturer's recommendations on a pad of paper and using a plastic spatula. The manipulated GIC was inserted with the aid of a Centrix syringe in plastic molds to make specimens with equal proportions, i.e., 4 mm in thickness and 5 mm in diameter. The pressure was then applied with a polyester matrix and glass slide onto the specimen surfaces to promote straight and smooth surfaces. The exposed surfaces were then protected with Vaseline [14].

After the initial setting reaction (10 to 15 minutes), the specimens that were protected with Vaseline and kept in containers containing distilled water at 37ºC for 1, 3, and 7 days were removed from the plastic mold. After this period, the specimens were fixed with sticky wax on acrylic resin blocks and their surfaces were polished using a circular polishing machine (EcoMet / AutoMet 250, Buehler Ltd. Lake Bluff, IL, USA) with P4000 sandpaper (Extec Corp, Enfield, CT, USA) [14].

Surface Microhardness

The specimens were subjected to a superficial hardness analysis using a Knoop indenter in a digital microdurometer (HMV-2, Shimadzu Corp., Kyoto, Japan) using a force of 25 g and an indentation time of 30 seconds. Five measurements were made on the surface of each specimen, spaced 100 μm apart, and the means of these were used for the results [14]. The data obtained were recorded in a KHN unit in three phases: (A) after 1, (B) 3, and (C) 7 days of storage in distilled water.

Data Analysis

The data were tabulated and analyzed using MedCalc for Windows software (version 17.2). In this study, a two-way repeated measures ANOVA test was used, with the type of GIC used (four groups) and the storage time in distilled water (3 phases) as the variables. The Tukey test was used to detect differences between the groups. A p-value <0.05 was considered statistically significant.

Results

Table 3 and Figure 1 show the mean values and standard deviation of the microhardness determined in the different types of GICs after 1 day, 3 days, and 7 days of storage in distilled water. The ANOVA test detected statistically significant differences for the following parameters: type of GIC (p<0.001), storage time in water (p=0.0141) and the interaction between both of these (p<0.001). The Tukey post-test found that the

KHN values differed between the groups, except for in the Ketac Molar and Gold Label 9 GICs. The microhardness values after 1 day of storage were lower than after 3 and 7 days of storage in terms of time parameters, except for the Ion-Z group (1 day = 3 days <7 days) and Riva Self-Cure GICs (1 day > 3 days = 7 days) (p <0.05).

Table 3. Mean and standard deviation of superficial microhardness values (KHN) considering the factors: types of GIC and the storage time in distilled water for 1, 3, and 7 days.

GIC	After 1 Day	After 3 Days	After 7 Days
Ion-Z (FGM)	33.05 ± 9.74 [A,a]	33.21 ± 10.31 [A,a]	52.07 ± 11.75 [A,b]
Ketac Molar Easymix (3M/ESPE)	50.35 ± 11.39 [A,a]	66.05 ± 10.48 [B,b]	67.77 ± 13.80 [A,b]
Riva Self Cure (SDI)	89.63 ± 15.59 [B,a]	71.31 ± 23.86 [B,b]	57.70 ± 16.89 [A,b]
Gold Label 9 (GC)	42.18 ± 9.03 [A,a]	68.54 ± 6.83 [B,b]	57.95 ± 8.24 [A,b]

ANOVA test & Tukey post-test; Capital letters represent statistical differences in the columns (within each time); Lower case letters represent statistical differences in the lines (within each GIC).
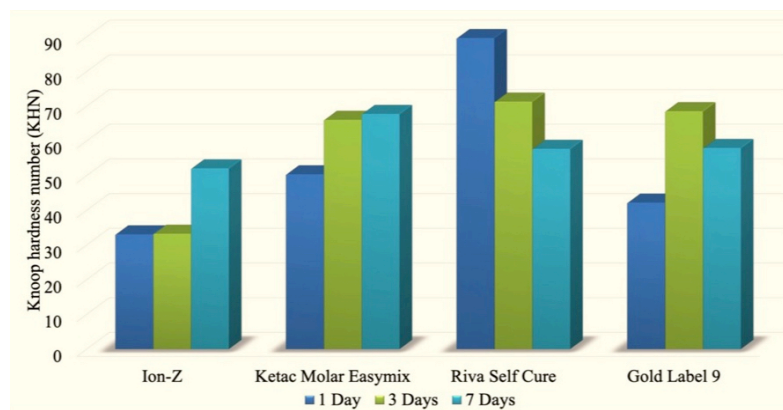
Figure 1. Mean values of surface microhardness (KHN) considering the factors: types of GIC and the storage time in distilled water for 1, 3, and 7 days.

Discussion

Dental caries still has a large impact on children's health; ART is considered a strategy based on health promotion that promotes an improvement in quality of life, using GIC materials that allow adherence to dental structures and the release of fluorides. Although classic GIC is the material of choice in several procedures in dentistry, some of its properties are still not entirely satisfactory [34].

In this context, it is important to understand the maturation time that these materials require to stabilize mechanically since this does not happen immediately after manipulation [8] and may still be related to early restoration failures. It is known that materials with good microhardness values indicate ease of finishing, polishing, good resistance to use, with less formation of scratches or grooves, which makes it difficult to form dental biofilm, resulting in less risk of compromising its resistance to fatigue and esthetic failures [9].

Based on the literature's information, this study was necessary to verify which relative improvements in the mechanical properties of different GICs were indicated for ART, including GIC reinforced by zinc particles. It is known that the incorporation of several types of nanoparticles in GICs increases the physical-mechanical and antimicrobial properties of these materials [11]. However, in regards to GIC Ion-Z properties, the literature is still scarce with regard to its antimicrobial properties, anti-caries effect, external sources in the maturation process, and the handling/proportion of powder and liquid and its effects on surface roughness [18,32,33], which makes it difficult to compare this GIC type with previous studies regarding surface microhardness data.

In the present study, the two null hypotheses were rejected because when comparing the type of GIC factors, storage time in distilled water, and the interaction between them, statistically significant differences were observed ($p < 0.05$), corroborating the hypothesis that some materials increased their initial mechanical stabilization after their relative storage times in distilled water (Ion-Z, Ketac Molar Easymix and Gold Label 9). However, the Riva Self Cure's surface microhardness decreased over time. It should be noted that at the end of 7 days, all types of GICs showed similar surface microhardness values. We suggest that some factors, such as the GIC composition and the powder/liquid proportion [35], may have influenced these results.

The ART technique aims to reduce costs, so the dentist must relate the costs and benefits to decide which material should be used. As already demonstrated in previous studies, and as the gold standard of treatment, with proven effectiveness [12,14], the Ketac Molar Easymix has a high cost in the Brazilian market. This makes the implementation of the ART technique using this material in the public service unfeasible.

Ion-Z, more recently introduced in the Brazilian market, has the addition of zinc particles in its composition; however, it did not present a significant microhardness in its first 72 hours of maturation. However, at the end of 7 days, its microhardness increased significantly, becoming an option for ART, as long as the synerisis and imbibition phase of the material can be controlled. A study that was carried out to test the addition of 5% of ZnO nanoparticles to the conventional restorative GIC observed a decrease of about 10% in its superficial microhardness, with no changes in its flexural strength compared to the GIC without this addition [31].

In a previous study, the Ion-Z GIC showed less roughness when the proportion of powder and liquid was 1:1 [33]. In the present study, methodological care was taken to standardize the proportion of powder and liquid for the manipulation of the different GICs. It is known that the concentration of zinc nanoparticles present in the GIC can increase its mechanical resistance and chemical adhesion, improving hypomineralization [30].

According to the analyzed data, noted that Gold Label 9 was the one that came closest to the microhardness values presented by the gold standard Ketac Molar Easymix in all analyzed periods. Riva Self Cure showed the highest initial surface microhardness value after 1 day of storage and suffered a drop in its surface microhardness over the other follow-up days. However, after 7 days, it remained similar to the others. In a previous study, low Knoop surface microhardness values were observed for Riva Self Cure compared to Ketac Molar Easymix after 1 day of paraffin storage [36].

In the present study, the lack of simulated aging through thermocycling might not be considered a limitation because the influence of thermocycling on tooth-colored materials and GICs hardening is still controversial [37,38]. Moreover, laboratorial studies are limited to draw real clinical considerations.

According to all the items analyzed (time, material and interaction between them), it is suggested that the best material to be used in the ART technique is: Ketac Molar Easymix> Gold Label 9> Riva Self Cure> Ion-Z. Thus, new long-term follow-up clinical studies are necessary to elucidate the different glass ionomer cements' maturation.

Conclusion

The initial mechanical stabilization differs between the different types of GICs tested in different storage periods in distilled water. The surface microhardness of GICs reinforced with zinc particles is similar to that of standard GICs for ART after 7 days of storage.

Authors' Contributions

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All authors declare that they contributed to critical review of intellectual content and approval of the final version to be published.

Financial Support

Conflict of Interest

The authors declare no conflicts of interest.

Data Availability

The data used to support the findings of this study can be made available upon request to the corresponding author.

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