Compressive and diametral tensile strength of glass ionomer cements

Bresciani E[1], Barata T[1], Fagundes T C[1], Adachi A[1], Terrin M M[1], Navarro M F[1]

Abstract

The aim of this study was to compare, in different periods of time, the compressive and diametral tensile strength of a traditional high viscous glass ionomer cement: Fuji IX (GC Corporation), with two new Brazilian GIC's: Vitro-Molar (DFL) and Bioglass R (Biodinamica), all indicated for the Atraumatic Restorative Treatment (ART) technique. Fifteen disk specimens (6.0mm diameter x 3.0mm height) for the diametral tensile strength (DTS) test and fifteen cylindrical specimens (6.0mm diameter x 12.0mm height) for the compressive strength (CS) test were made of each GIC. Specimens were stored in deionized water at 37° C and 100% of humidity in a stove until testing. Five specimens of each GIC were submitted to CS and DTS test in each period, namely 1 hour, 24 hours and 7 days. The specimens were tested in a testing machine (Emic) at a crosshead speed of 1.0mm/min for CS and 0.5mm/min for the DTS test until failure occurred. The data were submitted to two-way ANOVA and Tukey tests (α=0.05). The mean CS values ranged from 42.03 to 155.47MPa and means DTS from 5.54 to 13.72 MPa, with test periods from 1h to 7 days. The CS and DTS tests showed no statistically significant difference between Fuji IX and Vitro Molar, except for CS test at 1-hour period. Bioglass R had lowest mean value for CS of the cements tested. In DTS test Bioglass R presented no statistically significant difference when compared with all others tested GICs at 1-hour period and Bioglass R presented no difference at 24-hour and 7-day periods when compared to Vitro-Molar. Further studies to investigate other physical properties such as fracture toughness and wear resistance, as well as chemical composition and biocompatibility, are now needed to better understand the properties of these new Brazilian GIC's. ***First published in J Appl Oral Sci 2004; 12:*** **344-8.**

[1] *Department of Operative Dentistry, Bauru Dental School, University of São Paulo, Brazil*

Address of corresponding author:
Prof Maria Fidela de Lima Navarro
Faculdade de Odontologia de Bauru – USP / Diretoria / Alameda Dr. Octávio Pinheiro Brisolla 9-75 / CEP: 17012-901 – Bauru-SP, Brazil
Phone: 14 235 8295
Fax: 14 223 4679 E-mail: mflnavar@usp.br

Introduction

Conventional Glass Ionomer Cements (GICs) were introduced to the dental professional in 1971 by Wilson & Kent[1] as materials consisting of a base-usually an ion-leachable, calcium-aluminum-fluorosilicate glass powder – that is combined with polyacrylic acid or its copolymers[2]. These cements possess certain unique properties that make them useful as restorative and adhesive materials, including adhesion to moist tooth structure and base metals, anticariogenic properties due to release of fluoride, thermal compatibility with tooth enamel because of low coefficients of thermal expansion similar to those of tooth structure, biocompatibility and low cytotoxicity[2,3]. The limitations include the brittleness and poor fracture toughness of the materials[3,4].

Due to their considerable advantages and improvement, GICs have been widely indicated in the Atraumatic Restorative Treatment (ART) technique[5,6]. The ART is an approach of caries removal using only hand instruments, and restoring the cavity and sealing any associated fissures and pits with an adhesive restorative material, such as the currently used GICs. The approach combines a preventive component with a restorative procedure, and has the potential to be minimally invasive and maximally preserve the tooth structure[5,6].

But, due to inadequate physical properties of the glass ionomer materials to resist occlusal forces[7], efforts to improve several aspects of this treatment have been made, involving different kinds of self-cured GICs, such as inclusion of more reactive polyacids (e.g. copolymers of acrylic and maleic acid), by pretreatment of the glass surfaces and with modified glass compositions[8,9].

Besides all the developments in the hybrid systems, there has been a potential development in the field of conventional acid/glass systems with the development of high viscosity GICs, as Fuji IX (GC Corporation)[10,11,8]. The particular ways of improving conventional GICs consisted mainly of optimizing the concentration and molecular weight of the polyacid as well as the particle size distribution of the glass[8].

The compressive and diametral tensile strengths are common tests to determine the mechanical properties of glass ionomers[5,9,11-16]. As Brazilian GIC indicated for ART technique are commercially available and no previous study was performed with these materials, the aim of this study was to compare the compressive and diametral tensile strengths of a traditional Glass-Ionomer Cement (GIC): Fuji IX-GC Corporation, with two Brazilian marketed GICs:

Vitro-Molar (DFL) and Bioglass R (Biodinamica), all indicated for Atraumatic Restorative Treatment (ART) technique.

Table 1. Materials, manufacturers, GIC classification, powder:liquid (P:L) ratio, and batch numbers

Materials	Manufacturers	Classification	P:L ratio	Batch numbers- valid
Bioglass R	Biodinamica – Paraná, Brazil	Restorative-Conventional	3.0:1.0	157/04-03/2006
Fuji IX	GC Corporation-Tokyo, Japan	Restorative Conventional High Viscosity	3.6: 1.0	0309051-09/2006
Vitro Molar	DFL-Rio de Janeiro, Brazil	Restorative-Conventional	3.0:1.0	020144-11/2006

Material and methods

The three chemically-cured glass-ionomer cements (GICs) tested in this study are listed on Table 1.

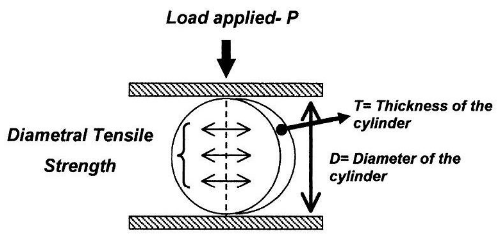
In accordance with ADA specifications 66[197] five specimens were prepared for each material and for each of three periods of time: 1 hour, 24 hours and 7 days, to evaluate compressive (CS) and diametral tensile strengths (DTS). The cylinder dimensions were 6.0mm diameter x 12.0mm height for the CS test and 6.0mm diameter x 3.0mm height for the DTS test.

The powder/liquid ratios were used according to the manufacturers' instructions for all materials. The material necessary to make each specimen was weighed in a precision balance and mixed with a plastic spatula (GC Corporation, Tokyo, Japan) on impermeable paper.

The specimens were made at room temperature of 23±2ºC and relative air humidity of 50±10%, as recommended by ADA specification[197]. After mixing, the materials were inserted with a Centrix syringe (Centrix, Shelton, USA) into metallic matrices, which were previously coated with a thin layer of petroleum jelly (Sidepal, Guarulhos, Brazil). The insertion was done slowly to adapt the material into the matrix and avoid bubble formation. The matrices were slightly overfilled with the GIC; a polyester strip (Proben, Catanduva, Brazil) covered with a thin layer of petroleum jelly was placed on the material and a cover slip was placed on top of it. Hand pressure was then applied for 20 seconds while excess material was extruded from the top of the matrices for DTS test. For CS test matrices were compressed in a device. Two minutes after the start of the mix, the matrices were placed in an oven at 37±1ºC and 95±5% relative humidity, for 15 minutes. Then, the specimens were ejected from the matrices and the excess material was removed with a carver and petroleum jelly was applied to protect the GIC during the initial setting reaction. The specimens were afterward stored in 6mL of deionized water at 37±1ºC. Tests were made in an Emic Universal Testing Machine (Emic- DL 5000/10000, São José dos Pinhais-PR-Brazil) at a crosshead speed of 1.0mm/min for CS and 0.5mm/min for the DTS test.

For the DTS test, the specimens were compressed diametrically introducing tensile stress in the material in the plane of the force application by the test (Figure 1).

Figure 1. Schematic illustration of Diametral Tensile Strength

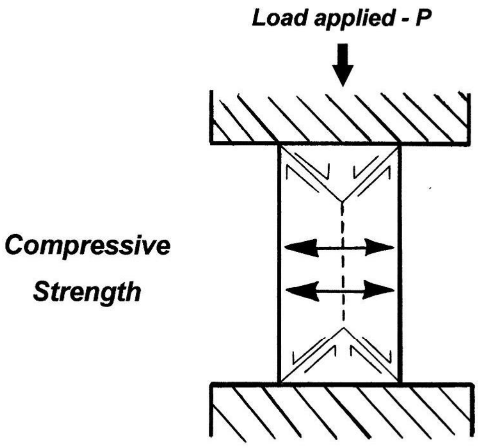


Adapted from Darvell, 2000[3].

How diametral tensile is envisaged ideally tension acting smoothly over the entire diameter, peak at the center[3].

This was calculated by the formula: 2P/ =πDT, where: P= load applied; D= diameter of the cylinder, T= thickness of the cylinder, π= (constant) 3,14. DTS values [kgf/cm^2] were converted into MPa as follows: DTS[MPa]=DTS[Kgf/cm2] x 0.09807. For the CS test, the specimens were placed in vertical position, with force incident on the long axis (Figure 2). The CS was calculated by the following formula: $P/\pi r^2$. Where: P= load at fracture, r= the radius of sample cylinder, and π= (constant) 3,14. CS values [kgf/cm^2] were converted into MPa as follows: CS [MPa]=CS[Kgf/cm^2] x 0.09807.

Figure 2. Schematic illustration of Compressive Strength



Darvell, 2000[3]

The stress and causes of failure in a cylindrical specimen loaded axially are no different from those in the diametral case except that the pattern is radially symetrical[3].

The data were submitted to two-way ANOVA (GICs and time) and Tukey-Kramer test for individual comparison with a 0.05 level of significance.

Results

The CS and DTS test results for the GICs are shown in Table 2 and 3.

Compressive Strength

- All GICs tested presented a significant increase in CS between the 1-hour and 7-day periods ($p<0.001$).
- Bioglass R and Vitro Molar and Fuji IX presented statistically significant difference between 1-hour and 24-hour periods. There were no differences between the 24-hour and 7-day periods.
- There were statistically significant differences between all GICs at the 1-hour period
- Bioglass R and Fuji IX showed statistically significant difference at the 24-hour and 7-day periods.
- Vitro Molar and Fuji IX showed no statistically significant difference at the 24-hour and 7-day periods.
- Bioglass R presented lower strengths than the others GICs included in this study at the 3 tested periods.

Diametral Tensile Strength

Two-way analysis showed significant differences between materials, where Bioglass R < Vitro Molar < Fuji IX ($p = 0.00$) and between periods of evaluation, where 1 hour < 1 day < 1 week ($p = 0.00$).

Table 2. Mean Compressive Strength (CS) of GIC's in MPa and standard deviations (SD)

Glass-Ionomer Cements	1 hour	24 hours	7 days
Bioglass R	42.03 (6.83)$^{A\ 1}$	83.39 (16.60)$^{A\ 2}$	95.67(15.27)$^{A\ 2}$
Vitro Molar	70.26(6.05) $^{B\ 1}$	125.67(6.95)$^{B\ 2}$	148.03(17.80) $^{B\ 2}$
Fuji IX	99.51(7.91)$^{C\ 1}$	147.93(18.18)$^{B\ 2}$	155.47(9.02)$^{B\ 2}$

Results designated with the same superscript characters are not statistically different ($p<0.05$). Letters are for comparisons between GIC's; numbers are for comparisons between times of the same material

Table 3. Mean Diametral Tensile Strength (DTS) of GIC's in MPa and standard deviations (SD)

Glass-Ionomer Cements	1 hour	24 hours	7 days
Bioglass R	5.54($\pm$0.529)$^{A\ 1}$	6.58($\pm$0.808)$^{B\ 1}$	8.74($\pm$1.396)$^{D\ 1,2,3}$
Vitro Molar	8.27($\pm$0.475)$^{A\ 2}$	9.43($\pm$0.822)$^{B,C\ 2}$	10.76($\pm$3.072)$^{D,E\ 2,4}$
Fuji IX	7.24($\pm$0.699)$^{A\ 3}$	11.96($\pm$1.514)$^{C\ 4}$	13.72($\pm$2.834)$^{E\ 4}$

Results designated with the same superscript characters are not statistically different ($p<0.05$). Letters are for comparisons between GIC's; numbers are for comparisons between times of the same material

- Bioglass R and Vitro Molar presented no statistically significant differences between the 3 analyzed periods (1 hour, 24 hours and 7 days)
- Fuji IX presented lower DTS at 1-hour when compared to 24-hour and 7-day periods.
- At 1 hour there were no differences between the materials.
- At 24 hours and 7 days, Bioglass R presented statistically significant lower DTS than Fuji IX
- At 24 hours and 7 days, Vitro Molar presented no statistical difference when compared to Bioglass R and Fuji IX.

Discussion

The resistance to fracture within a restorative material is specified by a fracture stress, which is often referred to as the strength of the material[16]. Two mechanical strength tests (Compressive and Diametral Tensile) were used in this study. The compressive strength (CS) is an important property in restorative materials, particularly in the process of mastication. This test is more suitable to compare brittle materials, which show relatively low result when subject to tension[3,18]. To test compressive strength of a material, two axial sets of force are applied to a sample in an opposite direction, in order to approximate the molecular structure of the material[19].

The diametral tensile strength (DTS) is a critical requirement, because many clinical failures are due to tensile stress[4]. As it is not possible to measure the tensile strength to brittle materials like Glass Ionomer Cements (GICs) directly, the British Standards Institution adopted the diametral tensile strength test[20]. In this test, a compressive force is applied to a cylindrical specimen across the diameter by compression plates. While the stresses in the contact regions are indeterminate, there is evidence of a compressive component that hinders the propagation of the tensile crack[3]. Large shear stresses that exist locally under the contact area may also induce a shear failure before tensile failure at the center of the specimen[3,13].

For all cements, CS values were much higher than DTS values. Compressive strength was about 8-13 times greater than DTS. This may be explained because cohesion between the materials is identical in both compressive and diametral tensile strength tests, but the direction of forces is reversed[16].

The results observed in this study were comparable to those presented in the literature concerning the Fuji IX DTS and CS values[9,14], probably due to standardization of procedures, especially those involved with measuring powder/liquid and manipulation according to manufacturers' instructions. This observation is of great importance to validate the present results and observations. There are studies with lower DTS values for Fuji IX, for example by Iazzetti et al.[21]. This happens due to different variables, as operators and measuring and manipulating the material. It is not possible to perform a statistical analysis between these two studies to check if the lower values are significantly different, but assumptions can be made and the lower values can be attributed to different variants of the study.

In this study, Bioglass R and Vitro Molar showed an increased in CS between 1 hour and 7 days and between 1 hour and 24 hours, but no significant difference in strength was observed between 24 hours and 7 days. This increase in CS can be analyzed by the setting reaction of GICs. The calcium polycarboxylate is formed in the first 5-7 minutes after mixing. The aluminum polycarboxylate, which is more stable and improves the mechanical properties of the cement, takes 24 hours to be formed in the average. The setting reaction continues for at least 24 hours and probably much longer[15,22]. In contrast, the Fuji IX did not show statistically significant differences when CS was evaluated (1 hour, 24 hours and 7 days). This may be explained by the faster setting reaction of the high viscosity GICs (Fuji IX). According to the manufacturer, the relatively higher viscosity is the result of the addition of poly (acrylic acid) to the powder and finer grain-size distribution[10,80] improved the mechanical properties of these cements mainly in the first hours[8]. No significant difference in strengths was observed between Fuji IX and Vitro Molar at 24-hour and 7-day periods.

In relation to the DTS, also theoretically Fuji IX should be stronger at all time intervals, as the maturation of the cement takes place at a faster rate. The use of smaller particles to increase the setting reaction may, however, have a compromising effect on strength. The smaller irregularly shaped particles used could increase the risk for local stress concentrations and as a result of that facilitate local crack growth and decrease strength. This may be attributed to no significant differences observed in DTS between the three GICs tested at 1 hour.

At 24-hour and 7-day periods, Bioglass R presented statistically significantly lower DTS than Fuji IX, but Vitro Molar presented no statistical difference when compared to Bioglass R and Fuji IX. This may be explained in part by the low cohesive condition[19]. The DTS measures the cohesive strength of the material, and the most brittle the material, the faster will be the occurrence of fracture. This influences the load are the cohesive properties of the material, independently of the deformation values.

The CS of amalgam is in the range of 300-450MPa, while that for composite resin is between 210-340 MPa[13]. In addition, the DTS of amalgam and composite resin has been reported to be between 43-58MPa and 40-70MPa respectively[13]. In this study, the mean CS and DTS at 24 hours of the GICs tested was still lower than that of the amalgam and resin composite, between 83.39 –147.93MPa and 6.58-11.96MPa, respectively. Although, it must be reiterated that, of the GICs tested, only Bioglass R showed a CS below the minimum strength at 24-hours periods of 125MPa required by British Standards[20]. The mean CS of Bioglass R at 1 hour, 24 hours and 7 days was very low, namely 42.03 - 83.39 - 95.67MPa, respectively.

Conclusions

The CS and DTS tests showed no significant difference between Fuji IX and Vitro Molar, except for CS test at 1-hour period. Bioglass R had lowest mean value for CS of the cements tested. In DTS test Bioglass R presented no statistically significant difference when compared to all others tested GICs at 1-hour period and Bioglass R presented no difference at 24-hour and 7-day periods when compared to Vitro-Molar. Further studies to investigate other physical properties such as fracture toughness and wear resistance as well as chemical composition and biocompatibility are now needed to better understand the properties of these new Brazilian GICs.

摘要

该项研究的目的在于对传统的高粘度玻璃离子结合剂 **Fuji IX （GC Corporation）**与两种新型的巴西玻璃离子接合剂：**Vitro-Molar (DFL)** 和 **Bioglass R (Biodinamica)**在不同时段的耐压强度和径向抗张强度进行比较，所有三种接合剂都用于防损填充性治疗（**ART**）技术。
每种玻璃离子接合剂都制作了**15**个圆盘状样本（**6.0mm** 直径 **x 3.0mm** 高度）用于径向抗张强度（**DTS**）试验和**15**个圆柱状样本（**6.0mm** 直径 **x 12.0mm** 高度）用于耐压强度（**CS**）试验。
样本储存于摄氏**37**度的除电离水中，并存于**100%**湿度的温室中直至试验。
每个时段，亦即**1**小时、**24**小时和**7**天，均有每种玻璃离子接合剂的**5**个样本呈做**CS**和**DTS**的试验。样本在试验机（**Emic**）中测试，十字头速度为**CS**试验**1.0mm/**分钟，**DTS**试验**0.5mm/**分钟，直到出现失败。数据呈交于双向**ANOVA**和**Tukey**试验（**α=0.05**）。试验时段从**1**小时至**7**天，**CS**平均值为**42.03**到**155.47MPa**，**DTS**平均值为**5.54**到**13.72MPa**。
除**1**小时时段的**CS**试验外，**Fuji IX** 与 **Vitro Molar** 比较，**CS**试验和**DTS**试验未显示统计数字上的显著差异。在所有测试的接合剂中，**Bioglass R**的**CS**平均值最低。在**DTS**测试中，**Bioglass R**在**1**小时时段未显示与所有其他测试接合剂存有显著统计数字差异，在**24**小时及**7**天时段，与 **Vitro-Molar** 相较无差异。当前需进一步调查这些新型巴西接合剂的其他物理属性例如断裂韧度和抗磨损性能，以及化学组成和生物适应性，以便更好地理解其各种属性。 *首次发表于J Appl Oral Sci 2004; 12: 344-8。*

Resumen

Este estudio tiene como propósito comparar, en diferentes periodos de tiempo, las resistencias compresiva y tensil diametral del cemento de vidrio de alta viscosidad [Fuji IX (GC Corporation)], y de dos nuevos cementos de ionómero de vidrio brasileros [Vitro-Molar (DFL) y Bioglass R (Biodinámica)], indicados para la técnica de Tratamiento Restaurador Atraumático (ART). Por cada ionómero de vidrio a ser probado, se prepararon quince discos de especímenes (6.0mm diámetro x 3.0mm alto) para la prueba de resistencia tensil diametral (RTD), y quince especímenes cilíndricos (6.0mm diámetro x 12.0mm alto) para la prueba de resistencia compresiva (RC). Los especímenes fueron almacenados hasta el momento de la prueba, en un horno, en agua desionizada a 37ºC y una humedad del 100%. Para cada periodo, es decir, 1 hora, 24 horas y 7 días, se sometieron cinco especímenes de cada ionómero de vidrio a las pruebas de RC y RTD. Los especímenes se probaron en una máquina de pruebas (Emic) a una velocidad 'crosshead' de 1.0mm/min para la prueba de RC y 0.5mm/min para la prueba de RTD, hasta que se presentó una falla. La información fue sometida a pruebas de doble-sentido ANOVA y a pruebas Tukey (α=0.05). Los

valores promedio para RC variaron de 42.03 a 155.47MPa y para RTD de 5.54 a 13.72 MPa, con periodos de prueba desde 1h a 7 días. Las pruebas de RC y RTD no mostraron diferencia estadística importante entre Fuji IX y Vitro Molar, a excepción de la prueba de RC de 1 hora. De los cementos probados, el Bioglass R tuvo el valor promedio más bajo para la prueba de RC. En la prueba de RTD, el Bioglass R no presentó diferencia estadística importante cuando se le comparó con los otros ionómeros de vidrio probados por espacio de una hora, y el Bioglass R no presentó diferencia en periodos de 24 horas y 7 días al ser comparado con el Vitro-Molar. Para comprender mejor las propiedades de estos nuevos cementos de ionómero de vidrio brasileros, se requieren estudios posteriores que investiguen otras propiedades físicas tales como resistencia a fractura y a desgaste, así como la composición química y bio-compatibilidad. *Publicado primero en J Appl Oral Sci 2004; 12:* 344-8.

Resumo

Comparou-se a Resistência à Compressão (RC) e à Tração Diametral (TD) de um cimento de ionômero de vidro de alta viscosidade [Fuji IX (GC Corporation)] e de dois novos cimentos Brasileiros [Vitro Molar (DFL) e Bioglass R (Biodinamica)], recentemente lançados no mercado, ambos indicados para o Tratamento Restaurador Atraumático (ART), em diferentes períodos de tempo. Foram confeccionados quinze corpos-de-prova com 6,0 mm de diâmetro x 3,0 mm de altura para o teste de TD e quinze com 6,0 mm de diâmetro e 12,0 mm de altura para o teste de RC, para cada ionômero a ser testado. Os corpos-de-prova foram armazenados em recipientes plásticos, com água deionizada, e mantidos em estufa a 37ºC e 100% de umidade, até a realização dos testes. Cinco corpos-de-prova de cada material foram submetidos aos testes de TD e RC em cada período de tempo: 1-hora, 24-horas e 7-dias, em uma máquina de testes universal (EMIC – DL 500) a uma velocidade de 1,0 mm/min para RC e 0,5mm/min para TD. Os dados obtidos foram submetidos aos testes ANOVA a dois critérios e Tukey (α=0,05). Os valores médios de RC e TD variaram de 42,03 a 155.47 MPa e de 5,54 a 13,72 MPa, respectivamente para os períodos analisados. O Fuji IX e o Vitro Molar não apresentaram diferenças em relação aos testes de RC e TD, exceto para RC no período de 1-hora. O Bioglass R apresentou os menores valores de RC dos cimentos testados. Na TD o Bioglass R não apresentou diferença em relação aos outros cimentos testados no período de 1-hora e não foi diferente do Vitro-Molar nos períodos de 24-horas e 7-dias. Mais estudos são necessários para avaliar outras propriedades mecânicas desses novos cimentos de ionômero de vidro brasileiros, tais como: tenacidade e desgaste, bem como composição química e biocompatibilidade

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