



## ORIGINAL ARTICLE

# Ultimate tensile strength and microhardness of glass ionomer materials

Resistência coesiva e microdureza de materiais ionoméricos

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## ABSTRACT

**Objective:** This study evaluated the ultimate tensile strength (UTS) and microhardness ( $\mu$ KH) of conventional (CO) and hybrid resin-modified glass ionomer (RM). **Material and methods:** Nine specimens to UTS and twelve for  $\mu$ KHN of glass ionomer materials were obtained using special molds. The materials were manipulated and CO groups were allowed to self-cure for five minutes and RM were subjected to light-activation as indicated by manufactures through a glass slide. All specimens were dark-stored in 100% relative humidity for 24 h. For UTS test, specimens were tested in tension in a universal testing machine (crosshead speed of 1 mm/min) until failure. For  $\mu$ KHN test a Knoop diamond indenter was used to make five indentations in the upper/light irradiated surface of the specimens. UTS and  $\mu$ KHN data were submitted to one-way ANOVA, followed by Tukey's test ( $\alpha = 5\%$ ). **Results:** The results for UTS were: Ionomaster: 7.0 ( $\pm 1.6$ ) A; Maxxion R: 8.8 ( $\pm 3.7$ ) A Vidrion R: 8.8 ( $\pm 3.9$ ) A; ChemFil Rock: 10.7 ( $\pm 4.6$ ) AB; Vitremer: 13.1 ( $\pm 3.3$ )BC; Vitrofil R: 14.9 ( $\pm 7.8$ )CD; Ionoseal: 14.5 ( $\pm 8.2$ )CD; Resiglass: 16.3 ( $\pm 2.3$ )D. The results for  $\mu$ KH: Ionomaster: 24.3 ( $\pm 6.6$ )B; Maxxion R: 17.7 ( $\pm 4.7$ ) A, Vidrion R: 31.0 ( $\pm 9.4$ ) B; ChemFil Rock: 31.1 ( $\pm 8.5$ )B; Vitremer: 20.3 ( $\pm 3.3$ ) A; Vitrofil R: 16.5 ( $\pm 5.1$ ) A; Ionoseal: 13.1 ( $\pm 8.5$ ) A; Resiglass: 21.6 ( $\pm 5.2$ ) A. **Conclusion:** It was observed that the hybrid resin-modified ionomers generally have higher cohesive strength than conventional ones, but lower microhardness.

## KEYWORDS

Tensile Strength; Hardness; Glass Ionomer Cements.

## RESUMO

**Objetivo:** Este estudo avaliou a resistência coesiva (UTS) e microdureza ( $\mu$ KH) de materiais ionoméricos, convencional (CO) e híbrido de ionômero de vidro modificado por resina (RM). **Material e Métodos:** Nove amostras para UTS e doze para  $\mu$ KHN de materiais ionoméricos foram preparadas utilizando matrizes especiais. Os materiais foram manipulados e grupos CO sofreram a autocura durante cinco minutos e os RM foram submetidos à ativação por luz como indicado pelos fabricantes através de uma lamínula de vidro. Todos os espécimes foram armazenados no escuro, em 100 % de umidade relativa por 24 horas. Para o teste de UTS, os espécimes foram testados em tensão numa máquina de ensaios universal (velocidade de 1 mm/min) até a falha. Para o teste  $\mu$ KH um diamante penetrador tipo Knoop foi usado para fazer 5 endentações na parte superior; irradiada pela luz na superfície dos espécimes. Os dados de UTS e de  $\mu$ KHN foram submetidos a one-way ANOVA, seguido pelo teste de Tukey ( $\alpha = 5\%$ ). **Resultados:** Os resultados para UTS foram: Ionomaster: 7,0 ( $\pm 1,6$ ) A; Maxxion R: 8,8 ( $\pm 3,7$ ) A Vidrion R: 8,8 ( $\pm 3,9$ ) A; Chemfil Rock: 10,7 ( $\pm 4,6$ ) AB; Vitremer: 13,1 ( $\pm 3,3$ )BC; Vitrofil R: 14,9 ( $\pm 7,8$ ) CD; Ionoseal: 14,5 ( $\pm 8,2$ ) CD; Resiglass: 16,3 ( $\pm 2,3$ )D. Os resultados para  $\mu$ KH: Ionomaster: 24,3 ( $\pm 6,6$ ) B; Maxxion R: 17,7 ( $\pm 4,7$ ) A, Vidrion R: 31,0 ( $\pm 9,4$ ) B; Chemfil Rock: 31,1 ( $\pm 8,5$ ) B; Vitremer: 20,3 ( $\pm 3,3$ ) A; Vitrofil R: 16,5 ( $\pm 5,1$ ) A; Ionoseal: 13,1 ( $\pm 8,5$ ) A; Resiglass: 21,6 ( $\pm 5,2$ ) A. **Conclusão:** Observou-se que os híbridos de ionômero de vidro modificados por resina geralmente têm força coesiva mais alta do que os convencionais, mas menor dureza.

## PALAVRAS-CHAVE

Resistência a tração; Dureza; Cimento de ionômero de vidro.

## INTRODUCTION

The dental caries are still a concern in Brazilian population [1]. Nearly 27% of children from 18 to 36 months had at least one primary tooth with dental caries experience [1]. Almost 70% of Brazilian children aged 12 years and about 90% of adolescents aged 15 to 19 had at least one permanent tooth with caries experience [1].

For this reason, new techniques have been introduced into clinical procedures using minimal intervention and prevention [2,3]. To make this possible, materials with different characteristics were combined in 1972 in England by Wilson & Kent [4]. They merged the characteristics of the silicate cement and zinc polycarbonate, which lead to the emergence of a hybrid material: the glass ionomer cement [4].

Since its development, the glass ionomer cements, now called conventional, were indicated in the prevention of dental caries, due to its peculiar characteristics of release and uptake fluorides from external environment [5]. The glass-ionomer cements (GICs) have been gaining more and more popularity as restorative materials, due to their favorable biological properties such as its biocompatibility, chemical adhesion, low solubility and its good performance in the long term, taking a significant role in preventive dentistry, also due to bacterial reduction and fluoride release [5].

The comprehension that the strength of the material in the oral environment is one of the factors that determine clinical longevity, in the late 80's an attempt was made to improve the glass-ionomer cements by hybridizing them with resin composite [6]. This combination provided the cariostatic effect of conventional GICs and an increased mechanical strength and more favorable aesthetic of composites, with an ease manipulation and control of activation, which are determining factors for choosing this material compared to conventional, expanding its use for many clinical situations [6-9].

Looking to the large and growing consumption of ionomeric materials in the world

and also in the Brazilian market [7], several companies have concentrated their forces in new formulations with restorative purposes that come to market as new glass ionomer cements and hybrid conventional glass ionomers [7].

Due to the presence of various ionomeric materials on the market [7], it is prudent that their physical and mechanical properties be properly analyzed, pointing to the professional the most safe and suitable materials for each clinical situation.

Because of the increasing use of GICs in dental practice and public programs like Atraumatic Restorative Treatment, these grow in the market stimulated companies to intensify the research in ionomeric materials, launching national and international versions of conventional GICs, reinforced GICs, and resin-modified glass ionomers [7].

As the material behaves in real stress situations (mechanical properties) and their physical behaviors (physical properties) are based on the laws of mechanics, knowledge is necessary for the success of dental materials in the oral environment. Specific tests are conducted to analyze physical and mechanical properties individually or simultaneously. All these properties are necessary for the development of new materials and maintenance and quality control of existing ones, providing the best indications for use and northings professionals. This study evaluated the ultimate tensile strength (UTS) and microhardness ( $\mu$ KH) of conventional (CO) and hybrid resin-modified glass ionomer (RM).

## MATERIAL & METHODS

### *Experimental Design*

The factor under study was the GICs in 8 levels composed of five conventional glass ionomer (CO) materials and tree hybrid of glass ionomer (RM) materials (Table 1). The response variables were the ultimate tensile strength (UTS) in MPa and surface microhardness in KHN.

**Table 1** - Materials, manufacturers, batch number and composition of the glass ionomer cements

Material	Manufacturer Batch	Type	Composition
Ionomaster	Wilcos, Petrópolis RJ, Brazil ADX0971	CO	Powder: calcium-fluoroaluminosilicate glass powder, tartaric acid, citric acid, pigments Liquid: water, polycarboxylic acid, pigments
Maxxion R	FGM, Joinville, SC, Brazil 070211	CO	fluoroaluminosilicate glass, polycarboxylic acid, calcium fluoride, water
Vidrion R	SS White, Rio de Janeiro, RJ, Brazil 20311	CO	Powder: sodium-fluorsilicate, calcium, aluminum, barium sulphate, polycarboxylic acid, pigments. Liquid: tartaric acid, water
Vitrofil R	DFL Rio de Janeiro, RJ, Brazil 10121715	CO	Powder: strontium silicate, aluminum, polyacrylic acid, aluminum, ferric oxide Liquid: polyacrylic acid tartaric acid, water
ChemFil Rock	Dentsply Caulk, Kontanz, Germany 1005004003	CO	Powder: Polycarboxylic acid Liquid: Polycarboxylic acid, tartaric acid
Resiglass	Biodinâmica Ibiporã, PR, Brasil 307/08	RM	Powder: Calcium-fluorosilicate, barium, aluminium, polyacrylic acid, fillers Liquid: dimethacrylate groups, water, catalyst
Vitremer	3M ESPE, St. Paul, MN, USA 20080606	RM	Powder: fluoroaluminosilicate glass powder Liquid: modified polialquenoic acid
Ionoseal	Voco Cuxhaven, Germany 1032024	RM	Bisfenol A glicedil metacrilato, glass ionomer powder, diurethanedimethacrylate, butylated hydroxytoluene

CO: conventional glass ionomer cement.

RM: hybrid of resin and glass ionomer material.

### Specimen preparation

To the UTS test the glass ionomer materials were applied to an addition silicon mold (Panasil, Kettenbach GmbH & Co., Eschenburg, Germany). The silicon mold was created by the insertion of a standard composite resin in an hourglass shape (1 mm thick and 1 mm wide at the constriction region) on a glass plate. After that, the addition silicon was mixed and applied on the hour glass-shaped composite to form the mold [10].

For the hourglass-shaped glass ionomer specimen, a single calibrated operator

manipulated the ionomer materials following manufactures instruction (Table 2) and inserted in the mold with an insertion centrix syringe (DFL, Rio de Janeiro, RJ - Brazil). A mylar strip was placed between the mold and the surface glass plate. The photo activation of RM materials were performed with a LED (Radii Cal, SDI, Bayswater, Victoria, Australia; power density:  $\pm 1,600$  mW/cm<sup>2</sup>), which before and after each 5 samples had its intensity continuously monitored with a radiometer (Cure Rite, Dentsply Caulk, Milford, USA). All conventional glass ionomers specimens were left on the plate for 5 min after mixing.

**Table 2** - Materials, proportion, manipulation, and fotoactivation time

Material	Proportion	Manipulation	Fotoactivation
Ionomaster	1 powder scoop to 2 liquids drops	Hand mixed	-
Maxxion R	1 powder scoop to 1 liquid drop	Hand mixed	-
Vidrion R	1 powder scoop to 1 liquid drop	Hand mixed	-
ChemFil Rock	Pre-dosed	Machined mixed	-
Vitrofil R	1 powder scoop to 2 liquids drops	Hand mixed	-
Resiglass	1 powder scoop to 2 liquids drops	Hand mixed	40 s
Vitremer	1 powder scoop to 1 liquid drop	Hand mixed	40 s
Ionoseal	-	Direct insertion	20 s

To the surface microhardness test a white polytetrafluoroethylene mold, constituted of two parts with a cylindrical window (4 mm in diameter, 2 mm high) was placed on a 10 x 120 x 0.05 mm Mylar strip (Dentart, São Paulo, Brazil) on a matte black background and kept stable with a metal ring. Twelve specimens per group of ionomer materials were prepared following manufactures instruction and they were inserted in a single increment (Table 2). A second strip was placed on the top of the uncured material. A glass slide was put on top of the Mylar strip and a 500 g load was applied for 30 s on the top of the mold in order to provide a smooth surface. The glass plate was removed before exposing the RM materials to light, and the light tip of a LED (Radii Cal; power density:  $\pm 1,600$  mW/cm<sup>2</sup>) was centered on the specimen.

Since storage time could affect the mechanical properties of glass-ionomers [11] and surface protection of the glass ionomer materials may cause some effect on the mechanical properties during early setting reactions, and it is desirable that the cement should be protected from direct water contact for at least 1 h after cement mixing [12], the

specimens were dark-stored in 100% relative humidity at 37 °C for 24 h.

### Ultimate tensile strength test

For the UTS test, the hourglass-shaped specimen was attached to the grips of a microtensile testing jig with cyanoacrylate (Loctite Super Bonder Gel, Henkel, Düsseldorf, Germany) and tested in tension in a universal testing machine (EZ Test, Shimadzu Co, Kyoto, Japan) at a crosshead speed of 1 mm/min until failure [10]. After testing, the specimens were carefully removed from the fixtures with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital micrometer (Series 406; Mitutoyo America Corp., Aurora, USA). The UTS data were expressed in MPa.

### Knoop microhardness test

After 24 h of setting, a Knoop diamond indenter in a hardness tester machine (Panambra, São Paulo, SP, Brazil), was used to make five indentations 30  $\mu$ m one of each other, in the center of the upper or light irradiated surface of the specimens, with a 10 g load for 5 s. The

mean of the five microhardness evaluations in KHN of each specimen was used as the specimen microhardness value.

### Statistical analysis

Data were submitted to a one-way ANOVA followed by a post hoc Tukey's test at a pre-set alpha of 5%, using statistical software (BioEstat 5.0 - Belem, Brazil).

## RESULTS

The results of maximum tensile strength (MPa) were analyzed by single factor ANOVA and Tukey's test. The CO materials Ionomaster, Maxxion R, and Vidrion R showed lower tensile strength than RM materials. The RM materials Resiglass, Ionoseal, and Vitremer showed the high tensile strength value as the CO Vitrofil R material. ChemFil Rock showed intermediary and statistically differed only from results Resiglass (Table 3).

**Table 3** - Mean and standard deviation of Ultimate Tensile Strength (in MPa).

MATERIAL	Type	Mean of Ultimate Tensile Strength
Ionomaster	CO	7.0 (± 1.6) A
Maxxion R	CO	8.8 (± 3.7) A
Vidrion R	CO	8.8 (± 3.9) A
ChemFil Rock	CO	10.7 (± 4.6) ABC
Vitremer	RM	13.1 (± 3.3) BC
Vitrofil R	CO	14.9 (± 7.8) CD
Ionoseal	RM	14.5 (± 8.2) CD
Resiglass	RM	16.3 (± 2.3) D

The results of surface microhardness (in Knoop Hardness number) were analyzed by single factor ANOVA and Tukey's test. The CO Vitrofil R and Maxxion R did not statistically differed from RM Ionoseal, Vitremer and Resiglass materials. The CO materials Ionomaster, ChemFil Rock, and Vidrion R showed statistically significant higher microhardness than the others (Table 4).

**Table 4** - Mean and standard deviation of Ultimate Tensile Strength (in MPa).

MATERIAL	Type	Mean of Microhardness
Ionoseal	RM	13.1 (± 8.5) A
Vitrofil R	CO	16.5 (± 5.1) A
Maxxion R	CO	17.7 (± 4.7) A
Vitremer	RM	20.3 (± 3.3) A
Resiglass	RM	21.6 (± 5.2) A
Ionomaster	CO	24.3 (± 6.6) B
Vidrion R	CO	31.0 (± 9.4) B
ChemFil Rock	CO	31.1 (± 8.5) B

## DISCUSSION

The ability of restorative dental materials to withstand functional forces is an important requirement for their long-term clinical performance [3]. Although, there are different types of mechanical strength tests, it has not been identified which in vitro tests can be considered with clinical validity to reproduce the survivability of glass ionomers materials [11].

Prosser et al. (1986) considered that the measurement of compressive strength had no fundamental meaning to glass ionomer materials, since they would only fracture at the anatomic level by tensile or shear failure, and reported that flexural strength is the most appropriate measurement to a glass ionomer material strength [11]. In this reason there are innumerable studies evaluating the flexural and biaxial strength, but there are no studies researching the ultimate tensile strength [11-14].

The data of maximum tensile strength showed that the RM materials Resiglass, Ionoseal, and Vitremer resulted in higher tensile strength than the CO materials Ionomaster, Maxxion R, and Vidrion R, and are in accordance to Bonilha et al. (2000), who showed that the fracture toughness of CO was lower than that of the RM [15].

RM materials presents in their formulations the 2-hydroxyethyl methacrylate (HEMA) polymer and other low molecular weight species

such as additives and co-initiators [16]. HEMA is incorporated as the resin component in RM formulations and it helps enhance water sorption of the methacrylate-containing polyacids because HEMA bears both hydroxyl and methacrylate groups [16]. So far almost all the commercially available RM contain HEMA, such as the RM studied Vitremer. This way, the cure of RM can be produced by an acid-base chemical mechanism similar to CO ones, by a thermal activated initiator, by a photochemical mechanism through the photoinitiator activated by blue light within the range of 400-500 nm wavelength, or simultaneously with both mechanisms [17].

Due to the presence of polymerizable components, McKenzie et al. (2003), found that RM Vitremer showed significantly higher biaxial flexure data than others CO materials, also they stated that this effect increase the ability to undergo flexure without fracturing, and hence raise the overall strength [18]. This result is in accordance to ours results, since RM materials RM materials presented the higher tensile strength than the CO materials.

However CO Vitrofil R material did not differ from RM materials, and this outcome may be due to differences in the filler ratio or size. Particle size may influence the strength of glass ionomer materials. The smaller particle size results in greater surface volume for polymeric acid and glass interaction lead to a faster maturation [19]. Also, the use of smaller particles increased the setting reaction, however may compromised the material strength [19].

The chemistry of the setting reaction of all CO is essentially an acid/base reaction [3], with the formation of a siliceous hydrogel resulted from a reaction between a leachable glass and a polyarilic acid liquid. Initially, the polyarilic acid attacks the surface of the fluoroaluminosilicate glass particles, resulting in the leaching of available cations (e.g.  $Al^{3+}$ ,  $Sr^{2+}$  and  $Ca^{2+}$ ). The thickening and gelatination of the solution arises from the formation of a cross-link hydrogen-bonding between the leached cations and the polymer network within the cement matrix [20].

The CO ChemFil Rock showed intermediary tensile strength data and statistically differed only from RM Resiglass. According to the manufacturer, this material has high molecular weight polyacids to improve the gelation due to hydrogen bond formation and a novel reactive zinc-modified fluoro-alumino-silicate glass filler [21]. The leached zinc ions form zinc-polyacid complexes which are stronger than of other bivalent strontium or calcium cations resulting in an accelerated build-up of flexural strength [21].

Then, the filler glass and powder/liquid ratio may influence the material compressive and tensile strength [14], and in the surface microhardness. Shintome et al. (2009) [7] reported that CO Fuji IX, which has a higher powder/liquid ratio, showed higher microhardness than Maxxion R and Vidrion R.

The CO materials ChemFil Rock and Vidrion R indicated to restorative purposes and the Ionomaster indicated to cementation, showed statistically significant higher microhardness than RM. The highest results observed in the CO material may be due to the Aluminum polycarboxylate formation which is a more stable and improves the mechanical properties of the cement takes a mean of 24 h to be formed [12].

Comparing the studied materials, the RM Resiglass and Ionoseal, that is indicated as lining, and to fissure sealing and restoration of smaller lesions, showed high cohesive strength and low microhardness probably due to its composition be close to composite resin, since this materials do not cure without light. Also, the RM Vitremer that present similar indications, and composition is close to conventional GICs which also exhibits an acid-base reaction showed similar behavior [22]. However, the Vitremer light activation promotes a rapid polymer network formation that strongly reduces the salt formation rate on acid base reaction [22]. Then, it can be supposed that the photo-activation reaction may be responsible for the low microhardness data observed to Vitremer.

However, the RM Vitremer microhardness data are very close to Cassoni et al. (2011), which

found a mean of 26.0 KHN after 24 hours of light-activation [23]. Also, in the Cassoni et al. (2011) study there was an increase of microhardness after 6 months of storage [23]. It reflects the state of cure of this material and the presence of an ongoing acid-base reaction and its maturity [23], and also this increase could be expected in our specimens in future evaluations.

By the other side, CO Vitrofil R and Maxxion R showed lower microhardness than the others CO materials, which may also be related to chemistry of the setting reaction and filler size. Also, these differences in the formulation may explain the different behavior of Vitrofil R, which showed low microhardness data and high tensile strength.

## CONCLUSION

It was observed that the resin-modified ionomers generally have higher cohesive strength than conventional ones, but lower microhardness.

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