

Evaluation of physical and biological properties of conventional glass ionomer cements

Avaliação das propriedades físicas e biológicas de cimentos de ionômero de vidro convencionais

Wellington Luiz de Oliveira Da Rosa*

Arthur Dias Galarça**

Felipe Immich**

Tiago Schlindvein de Araujo**

Adriana Fernandes Da Silva*

Evandro Piva*

Abstract

Objective: to evaluate the physical and biological properties of conventional glass ionomer cements (GICs). **Methodology:** the following GICs were evaluated: Fuji IX (GC Europe, Belgium), Ketac Molar (3M ESPE, United States), Maxxion R (FGM, Brazil) and Vitro Molar (Nova DFL, Brazil). Setting time, dimensional change, radiopacity, water solubility and water absorption were evaluated for all materials. Compressive strength was analyzed after intervals of 1h, 24h, 7 days and 28 days; and release of fluoride ions at 3 am, 24 am and 72 am. Cell viability was assessed after 24 and 48 hours with fibroblast cells. Statistical analysis was performed using SigmaPlot 12 software (Systat Inc, San Jose, CA, USA), with a significance level set at $\alpha = 0.05$. **Result:** only the Fuji IX had an adjustment time within the range recommended by the Standard Specification of ADA 96 (2012) of not exceeding 6 minutes. Vitro Molar and Maxxion R had radiopacity that was not in accordance with the ADA 96 (2012) specification. Maxxion R and Vitro Molar showed a statistically similar dimensional change. As for mechanical properties, Fuji IX was the only GIC that showed an increase in compressive strength during the evaluation period of 28 days. Ketac Molar showed the highest cell viability, while Maxxion R showed severe cytotoxicity and the highest cumulative fluoride release value. **Conclusion:** Fuji IX and Ketac Molar showed the most appropriate physical and biological properties among the evaluated GICs.

Keywords: glass ionomer cements; cytotoxicity; dental materials; *in vitro* techniques.

<http://dx.doi.org/10.5335/rfo.v25i3.10808>

* PhD, Department of Restorative Dentistry, School of Dentistry, Federal University of Pelotas, Pelotas, Brazil.

** Undergraduate Student, School of Dentistry, Federal University of Pelotas, Pelotas, Brazil.

Introduction

Glass ionomer cements (GICs) are used mostly as provisional restorative materials, however, they are also used as liners and bases, luting cements, fissure sealants, bonding agents for orthodontic brackets and as restorative material for atraumatic restorative treatment (ART)¹⁻³. GICs are classified as acid-base cements that contain calcium or strontium aluminofluorosilicate glass powder (base) combined with a water soluble polymer (acid)². The final structure contains filler particles in the form of unreacted glass which helps to reinforce the set cement⁴. In general, GIC can be classified as conventional, and resin modified glass ionomer cements, and could also be classified in subgroups regarding their viscosity, as High Viscosity GICs are more suitable for ART than conventional ones^{2,5}.

Although resin-modified GICs usually have better mechanical and esthetic properties, they may possibly have toxic effects on the dental pulp – since the HEMA present in their composition is capable of crossing dentin and reaching the pulp tissue. Also, the high cost - when compared with conventional – may be disadvantages that limit their application in some cases, such as ART, for

example⁵. Conventional GICs have been extensively used in dentistry mainly due to their low-cost, good biocompatibility, ability to bond chemically to mineralized tooth substrates, bactericidal ability, and fluoride release⁴. It has been reported that GIC are able to release fluoride at a sustained rate for long periods of time (at least 5 years)^{2,6,7}, and could present the potential to promote remineralization⁴.

As there are a high number of different brands of GICs on the market, the demand for them to present better properties for clinical use is also high. However, in spite of the wide variety of GICs, evaluations of their physical and biological properties are lacking. Also, a recent systematic review failed to compare the results between different GICs because of a lack of standardization of the studies, and researchers should adhere to the ISO specifications when planning and performing laboratory experiments⁹. Therefore, the aim of this *in vitro* study was to evaluate physical and biological properties of conventional GICs following ISO and the American Dental Association (ADA) standards. The null hypothesis evaluated was that all materials would present similar setting time, radiopacity, water solubility and water absorption, dimensional change, compressive strength and cell viability.

Materials and methods

The materials evaluated, manufacturer, lot and powder:liquid ratios are presented in Table 1.

Table 1 – Main composition of conventional glass ionomer cements evaluated

Groups	Composition	Powder:liquid ratio (g/g)
Fuji IX (GC Europe, Belgium) Lot. 1401101	Powder: Aluminum fluorosilicate glass and polyacrylic acid powder. Liquid: distilled water, polyacrylic acid, polycarboxylic acid.	3.55
Ketac Molar (3M ESPE, United States) Lot. 593966	Powder: Aluminum fluorosilicate glass, lanthanum and calcium, polyacrylic acid, eudragit, tartaric acid, sorbic acid, benzoic acid and pigments. Liquid: Water, copolymer of acrylic acid and maleic acid, tartaric acid and benzoic acid.	2.55
Maxxion R (FGM, Brazil) Lot. 250515	Powder: Aluminum fluorosilicate glass, polycarboxylic acid, calcium fluoride and water. Liquid: Polyacrylic acid.	1.38
Vitro Molar (Nova DFL, Brazil) Lot. 15010014	Powder: Barium and aluminum silicate, dehydrated polyacrylic acid and iron oxide. Liquid: Polyacrylic acid, tartaric acid and distilled water.	2.9

Source: authors.

Dimensional change was determined in accordance with methods recommended by the International Organization for Standardization

(ISO) specification number 6876:2012. Radiopacity, compressive strength and setting time were determined according to the American National

Standards Institute/American Dental Association number 96:2012 (ANSI/ADA). Water solubility and water absorption were determined according to the ISO 4049:2000, and cell viability according to ISO 10993-5:2009 specifications.

Setting Time Analysis

Stainless steel molds with an inner diameter of 10 mm, and uniform thickness of 2 mm were fabricated for each material evaluated (n=5). A Gilmore needle (100 g and 2 mm active tip) was vertically pressed against the horizontal surface of the material to observe indentations. Setting time was defined as the time elapsed from the beginning of the mixture until the time when no more indentations were visible on the cement surface.

Dimensional Change

Ten specimens (n=10) of each material were prepared (6 mm high and 4 mm in diameter). All the specimen lengths were measured and afterwards they were stored at 37°C in 1 ml of distilled water for 30 days. Specimens were then removed from the mold and measured again. The dimensional change was calculated as the percentage change between the original length and the value measured after 30 days storage in water.

Radiopacity Analysis

Five samples (n=5) of each material (10 mm in diameter and 1 mm thick) were placed on occlusal radiographic film (Insight, Kodak Company, NY, USA), and radiographed with an X-ray apparatus (Kodak 2200 intraoral X-ray system), operating at 70 kV and 10 mA with exposure time of 0.36 s and a focus-film distance of 30 cm. After processing, optical density or gray tones of images were measured and obtained by means of software ImageJ 1.4 (National Institute of Mental Health, Maryland, USA). Five points of each specimen were randomly selected to obtain the mean radiopacity value (R) in pixels, which was further transformed into mm/Al according to an aluminum scale also present in the radiograph.

Water Solubility (W_{SL}) and Water Absorption (W_{SR}) Analyses

Ten specimens (n=10) of each material were molded (1 mm thick and 6 mm in diameter). The specimens were weighed after 24 h of setting, until a constant initial mass (m1) was obtained. Then the samples were stored in distilled water for one week at 37°C. After removing the samples from water storage, they were weighed (m2). All specimens were weighed on the following days until a constant final mass be obtained (m3). The water solubility ($W_{SL} = [(m1 - m3)/m3] \times 100$) and absorption ($W_{SR} = [(m2 - m3)/m3] \times 100$) were calculated as percentages of the original weight.

Compressive Strength Analysis

Ten specimens (n=10) of each material were prepared by using a split metal (6 mm high and 4 mm diameter). The specimens were stored at 37°C in 1ml of distilled water until the following time intervals of testing: 1 h, 24 h, 7 days, and 28 days. A universal testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil) was used to evaluate compressive strength at a crosshead speed of 0.5 mm/min. The maximum load required to fracture each specimen was determined.

Fluoride Ion Release

The fluoride ion release was assessed at time intervals of 3h, 24h and 72h using an advanced pH/ISE/mV meter (HI5222-01, HANNA Instruments, Brazil). Standard discs (n = 3 Ø = 8 mm; h = 1.5 mm) of each experimental group were individually stored in 1.5 ml Milli-Q water at 37°C throughout the entire test period.

Cell Viability Analysis

Cell viability analysis was performed using mouse fibroblasts L929 (20×10^3 well⁻¹) maintained in Dulbecco's Modified Eagle Medium (DMEM, Lonza, Switzerland). Specimens of each material (n = 5; 5 mm in diameter and 1 mm deep) were stored at 37°C for 7 days to allow all cements to reach the final setting time, and after

this they were sterilized under a UV-light source for 1 h on each side. Then, specimens were placed in 24-well plates with 1 mL of DMEM at 37°C, pH 7.2. After 24 h, 200 µL of eluate from each specimen was transferred to previously prepared 96-well plates and incubated for 24 and 48 h. Control cells without material extracts were considered to have 100% cell viability. MTT (Methylthiazolyldiphenyl-tetrazolium bromide; Sigma Aldrich, United States) was applied to assess cell metabolic function by mitochondrial dehydrogenase activity using the absorbance at 540 nm via a microplate reader (SpectraMax M5; Molecular Devices, Sunnyvale, CA, United States). The level of cytotoxicity of conventional GICs were classified into severe (<30%), moderate (30-60%), slight (60-90%) and non-cytotoxic (>90%).

Statistical analysis

Statistical analysis was performed with SigmaPlot 12 software (Systat Inc, San Jose, CA, USA). For setting time and dimensional change the data were analyzed using One-way ANOVA followed by the Tukey test. For radiopacity, water solubility and water absorption, the data were

analyzed using the Kruskal-Wallis followed by the SNK test. For compressive strength and cell viability the data were analyzed using Two-way ANOVA followed by the Tukey test. Fluoride ion release was analyzed using the Two-way Repeated Measures ANOVA followed by the Tukey test. The level of significance was set at $\alpha = 0.05$.

Results

Setting Time, Dimensional Change, Radiopacity, Water Solubility (W_{SL}) and Water Absorption (W_{SR})

Fuji IX presented a setting time statistically similar to that of Ketac Molar, as shown in Table 2. Maxxion R and Vitro Molar showed a setting time of longer than 7 minutes. Only Fuji IX had a setting time within the range not exceeding 6 minutes, according to the Technical Specification recommended by the ADA 96 (2012) Standard. Regarding dimensional change, Fuji IX showed values that were statistically similar to those of Ketac Molar ($p > 0.05$), as shown in Table 2. Maxxion R and Vitro Molar also showed a statistically similar dimensional change, with values higher than 3% ($p > 0.05$).

Table 2 – Mean and standard deviation of setting time, radiopacity, water absorption (W_{SR}), water solubility (W_{SL}) and dimensional change of conventional glass ionomer cements

Groups	Setting time (min)	Radiopacity (mmAl)	W_{SR} (%)	W_{SL} (%)	Dimensional change (%)
Fuji IX	5.0 (0.19) ^c	2.64 (0.4) ^a	11.54 (0.96) ^c	4.15 (0.46) ^c	1.19 (0.82) ^c
Ketac Molar	6.3 (0.35) ^{bc}	1.27 (0.13) ^b	13.97 (2.69) ^b	5.99 (0.67) ^b	1.91 (1.18) ^{bc}
Maxxion R	8.6 (0.96) ^a	0.29 (0.05) ^c	20.62 (3.13) ^a	11.47 (1.59) ^a	3.28 (1.25) ^a
Vitro Molar	7.5 (0.80) ^{ab}	0.79 (0.17) ^d	18.99 (1.51) ^a	10.51 (0.96) ^a	3.06 (0.81) ^{ab}

Data followed by different letters are statistically different in the same column ($p < 0.05$).

Source: authors.

Fuji IX was the most radiopaque, followed by Ketac Molar, Vitro Molar and Maxxion R; and all groups differed statistically ($p < 0.05$). Vitro Molar and Maxxion R presented a radiopacity that was not in accordance with ADA 96 (2012) specification, with values not higher than 1mmAl. For water solubility and water absorption, Maxxion R and Vitro Molar showed the highest values and were statistically similar (Table 2). Fuji IX presented the lowest water solubility and water absorption and differed statistically from all the other groups ($p > 0.05$).

Compressive Strength

The compressive strength increased with time only for Fuji IX; and after time intervals of 7 and 28 days a higher and statistically different compressive strength was shown than after 1 and 24h ($p > 0.05$) (Figure 1). Ketac Molar presented a decrease in compressive strength after 1h ($p > 0.05$), but after 24h the values were statistically similar to those shown in the time intervals of 7 and 28 days ($p < 0.05$). Maxxion R and Vitro Molar showed similar compressive strength

in all time intervals. Considering the different time intervals, after 1h Ketac Molar presented the highest and statistically different compressive strength when compared with all the other groups ($p>0.05$). However, after 24h all groups

showed statistically similar values. After the time intervals of 7 and 28 days, Fuji IX showed the highest compressive strength values that differed statistically from those of all the other groups ($p>0.05$).

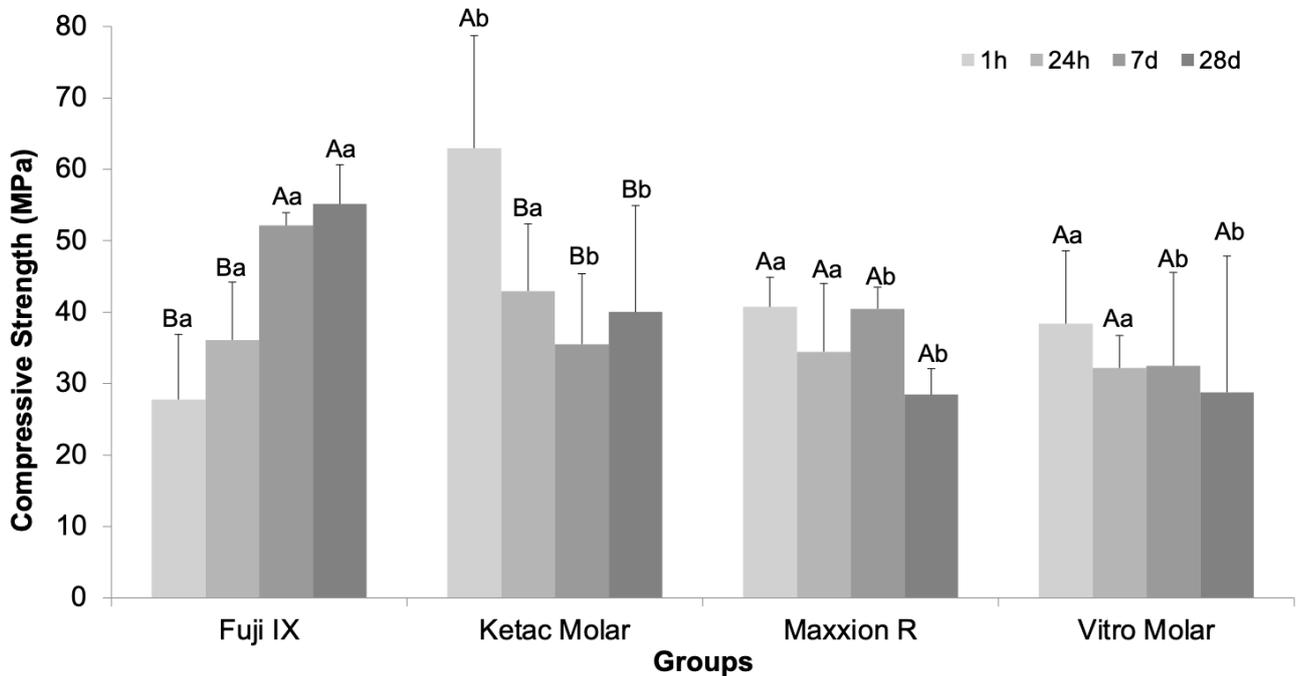


Figure 1 – Compressive strength (MPa) and standard deviation of conventional glass ionomer cements evaluated in time interval up to 28 days. Different uppercase letters indicate statistically significant differences within the same material, and different lowercase letters indicate statistically significant differences within the same time interval ($p<0.05$).

Source: authors.

Fluoride Ion Release

Maxxion R released the highest quantity of fluoride ions, showing values with statistically significant difference from those of all the other materials in all time intervals ($p<0.05$), with a

cumulative release of 27.7 (5.44) ppm. Moreover, the fluoride ion release by Maxxion R increased over time ($p<0.05$). Fuji IX, Ketac Molar and Vitro Molar showed statistically similar values in all time intervals, with no statistically differences observed for the same material over time (Table 3).

Table 3 – Mean and standard deviations (SD) of fluoride ion release (ppm) for different materials and times

Groups	3h	24h	72h	Cumulative ion release
Fuji IX	0.99 (0.16) ^{Cb}	1.00 (0.13) ^{Cb}	0.94 (0.11) ^{Cb}	2.93 (0.43)
Ketac Molar	1.15 (0.10) ^{Cb}	0.87 (0.05) ^{Cb}	0.86 (0.02) ^{Cb}	2.88 (0.17)
Maxxion R	3.46 (0.85) ^{Ca}	9.39 (1.21) ^{Ba}	14.85 (3.38) ^{Aa}	27.7 (5.44)
Vitro Molar	1.32 (0.08) ^{Cb}	0.87 (0.03) ^{Cb}	0.90 (0.15) ^{Cb}	3.09 (1.01)

Different lowercase letters in columns and uppercase letters in rows indicate statistically significant differences ($p<0.05$).

Source: authors.

Cell Viability

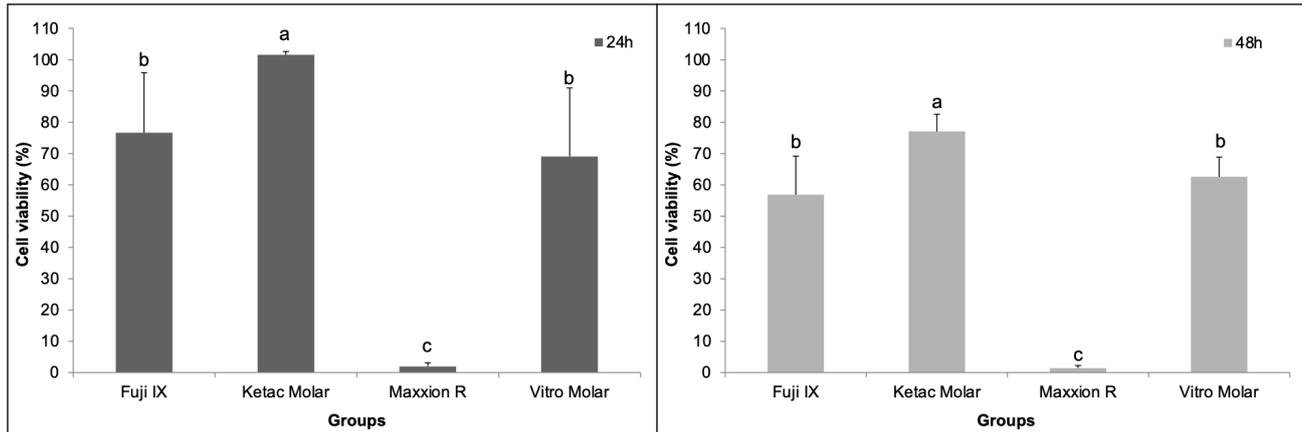


Figure 2 – Cell viability (%) of conventional glass ionomer cements evaluated after time intervals of 24 and 48-hours. Control Group Cell viability was considered equal to 100%. All groups showed no statistically significant differences between 24 and 48 h within the same material. Different letters showed statistically significant difference between groups irrespective of time ($p < 0.05$).

Source: authors.

All groups showed no statistically significant differences between 24 and 48h within the same material (Figure 2). Ketac Molar showed the highest cell viability after 24 and 48h, of 101.56% (± 1.06) and 77.01% (± 5.52) respectively; and differed statistically from all the other groups ($p > 0.05$). Fuji IX and Vitro Molar showed a statistically similar and slightly cytotoxic effect, with a cell viability of 76.66% (± 19.18) and 69.08% (± 21.87) after 24h, and 56.80% (± 13.33) and 62.55% (± 6.30) after 48h, respectively. On the other hand, Maxxion R showed a severe level of cytotoxicity, with a cell viability of only 1.97% (± 1.13) and 1.45% (± 0.79) after 24 and 48h respectively; which differed statistically from the values of all the other groups ($p > 0.05$).

Discussion

The hypothesis tested was not accepted, because the conventional GICs evaluated showed differences in physical and biological properties. In general, Fuji IX and Ketac Molar showed the shortest setting time, lowest dimensional change, water solubility and water absorption, and the highest radiopacity values. Regarding mechanical properties, Fuji IX was the only GIC that showed an increase in compressive strength during the 28-day time interval of evaluation. Whereas, Ketac Molar showed the lowest cytotoxicity level, while Maxxion R presented severe cytotoxicity.

The setting time of GICs evaluated ranged from 5 to 8.6 minutes. ANSI/ADA specification number 96 (2012) specified a maximum setting time of 6 minutes, and only Fuji IX was within the time recommended. Fuji IX and Ketac Molar showed the shortest setting times, which were in agreement with the findings of another study¹⁰, and may be important for clinical use, since a material with a shorter setting time could shorten the clinical time taken to perform the procedure, and make the material less vulnerable to external challenges, such as those of saliva or water^{1,11}. The initial setting reaction and subsequent matrix formation is a multifaceted phenomenon¹. During setting, there are many reactions that take place, and are associated with various changes in the physical properties known as maturation^{1,11}. In the acid-base reaction, H^+ ions split off the $COOH$ -groups from the acid-chains and react with the ions released from the glass (Al^{3+} , Ca^{2+})^{8,11}. Due to partial dissolution, the outer surface of the glass particles is converted into a gel coat from which Al^{3+} , Ca^{2+} and F^- ions are secreted⁸. The COO^- groups and the Al^{3+} and Ca^{2+} ions released enable the crosslinking of these chains, leading to the formation of a solid network around the glass particles and a hydrated silica gel phase¹.

During the initial setting stage, GICs are more susceptible to hygroscopic change in the environment and they may undergo syneresis and imbibition processes, which are the loss or

gain of water from the external environment, respectively¹². Both Fuji IX and Ketac Molar presented the lowest values of dimensional change, water solubility and water absorption, which could have been affected by the setting and complete maturation of the GICs. The crosslinking reactions may be slow so that it may take a long time before the cements are completely matured, and during this period, the GICs are vulnerable and the material may dissolve, compromising other properties⁸. The highest values of water sorption and solubility were found for Maxxion R and Vitro Molar. These results are in accordance with those reported in another study¹³. On the other hand, Ketac Molar was less sensitive to water sorption than Maxxion R and Vitro Molar, probably because of the large number of carboxylic acid groups in the liquid and the presence of tartaric acid in their composition. These promote a large number of crosslinks that are established between the polymer chains, which reduce the empty spaces, and thus, the water inflow into the material^{13,14}. The higher level of sorption and solubility undergone by Maxxion R was probably related to its inferior results relative to both dimensional change and compressive strength. Furthermore, this higher solubility in an aqueous medium may have influenced its severe cytotoxicity found in this study.

As regards radiopacity, Maxxion R and Vitro Molar showed the lowest values. In another study, Maxxion R also had the lowest radiopacity, followed by Vitro Molar when compared with Vitremer (3M ESPE, United States), Vitrofil LC (DFL, Brazil) and Magic Glass (VigoDent, Rio de Janeiro, Brazil) 15. The presence of silicon oxide on the surface of GICs may be related to the opacity of the material, and Maxxion R and Vitro Molar presented a low quantity of silicon, i.e., 18% and 14.3%, respectively¹⁵. We also complied with ISO standards by using 1 mm thick specimens, and clinically it is common for the thickness of the material to exceed 1 mm. However, it has previously been demonstrated that even with greater thicknesses (up to 6 mm), Maxxion R and Vitro Molar had low radiopacity¹⁵. It is important for the radiopacity of GICs to differ from that of tooth substrates to be able to differentiate them

from tooth tissues or carious lesions. Both Fuji IX and Ketac Molar showed a higher radiopacity than dentin, with Fuji IX being the only GIC evaluated with a radiopacity value higher than that of enamel substrate. Wren *et al.*¹¹ (2013) have also demonstrated that Fuji IX had a higher radiopacity than Ketac Molar. Although the presence of aluminum (Al) may provide radiopacity, the calcium concentration may be replaced by strontium (Sr) or lanthanum (La), which impart radiopacity to the cement¹¹. Ketac Molar contains Al and La to produce radiopacity, while Fuji IX contains Al and Sr¹¹.

Many studies have assessed the mechanical properties of glass ionomer cements, especially those used for ART, such as the materials evaluated in our *in vitro* study. The reason for the low score of compressive strength values found for GICs is due to their poor mechanical properties, such as low fracture strength, toughness, and higher occlusal wear rate¹⁷. For this reason, GICs are not recommended for use as permanent material in adult Class I and II patients. ANSI/ADA specification number 96 (2012) specifies that a minimum compressive strength of 100MPa is required, which it was not achieved by any of the GIC materials.

Although after 1h Ketac Molar showed a higher compressive strength value than other GICs; after time intervals of 7 and 28 days of evaluation Fuji IX showed a higher compressive strength value than other GICs evaluated. Other studies have shown that Fuji IX and Ketac Molar presented the highest performances relative to compressive strengths¹⁸. In general, the compressive strength is evaluated only after 24h¹⁶, the time interval in which all the GICs were evaluated in our study that also showed no differences. Another study⁸ showed that the compressive strength of Fuji IX and Ketac Molar increased when the strength after 1h was compared with the evaluation after 1 and 4 weeks, and also after 3 months. The first 24 h are very critical in the setting of GIC, which could affect the compressive strength, because in this time interval, the material is most prone to wear and dissolution⁸. However, the storage temperature, storage time and storage medium may also have an influence on the compressive

strength of GIC, which may explain the different results obtained in our study.

The compressive strength could also impact the clinical success rates of GICs, which varies among clinical trials^{19,21}. A randomized clinical trial¹⁹ with occlusal ART restorations in primary molars showed that after one year Fuji IX had a better performance when compared with Vitro Molar and Maxxion R. The clinical success rates were of 77.6% for Fuji IX, 61.1% for Vitro Molar and 57.5% for Maxxion R, and restoration loss was the main reason for failure¹⁹. Considering proximal ART restorations, after one year the success rate for Ketac Molar was 50.85%, and 34.48% for Vitro Molar²⁰. After 3 years of follow-up, another study showed the overall success rate for Fuji IX, Hi-Dense (SHOFU Dental GmbH, Germany), and Maxxion R was only 24.4%²¹. In these studies, no statistical differences in clinical success were demonstrated among the GICs^{20,21}.

Although GICs are generally considered to be tissue-compatible, many studies have indicated that GICs might be cytotoxic, and it has been suggested that unreacted components or setting reaction products can affect cell metabolism²². The acid-base reaction is essentially completed in 24h¹, and for these reasons, the cell viability evaluation was performed after 7 days of specimen preparation, to allow GICs to reach the final setting stage. Furthermore, we assessed cell viability in mouse fibroblasts after time intervals of 24 and 48 hours, and no significant differences were found in both time periods, which suggested that most of the cytotoxic effect occurred in the first 24 hours. According to ISO 10993-5:2009, a reduction of over 30% in cell viability is considered a cytotoxic effect, which was demonstrated for Maxxion R in the first 24h, and also for Fuji IX and Vitro Molar after 48h. The results of our study are consistent with those of other *in vitro* studies^{23,24}, and Ketac Molar was the only GIC that presented a cell viability reduction lower than 30%. Marczuk-Kolada *et al.*²⁴ (2017) have demonstrated that Fuji IX presented a low level of cytotoxicity. On the other hand, our study demonstrated that Maxxion R presented severe cytotoxicity, which was in agreement with the findings of another study²³. The reasons for the

cytotoxicity of GICs have not yet been completely elucidated, and the literature contains many explanations, with the effect of low pH during setting and the effects of various components released being the most reported associated factors.²⁴

The severe cytotoxicity of Maxxion R could be linked to fluoride ion release. The effect of different ions on cytotoxicity have previously been investigated, and the results have indicated that F⁻, Al³⁺ and Sr²⁺ levels were too low to affect cell viability²⁵. However, a previous study by Oguro *et al.*²⁶ (1990) reported that *in vitro*, fluoride was cytotoxic to human gingival fibroblasts, which was related to its concentration. Little information is available regarding the effects of fluoride on human pulp cells, and another study by Chang and Chou²⁷ (2001) has demonstrated that there was a decrease in human dental pulp cell viability caused by fluoride. Thus, further studies are still needed to elucidate these questions, and also different cell lines are required to complement data on the biological response to GICs.

While it is recognized that *in vitro* analyses cannot reproduce complex *in vivo* interactions, they are extremely useful as the first level of investigation because of the ability to control environmental factors and to test components individually²⁸. However, the results of laboratory experiments should not be directly extrapolated to clinical conditions. Therefore, within the limitations of this study, Fuji IX and Ketac Molar were shown to have the most suitable physical and biological properties among conventional GICs evaluated.

Conclusion

Fuji IX and Ketac Molar showed the lowest setting time, dimensional change, water solubility and water absorption, and the highest radiopacity. As regards the mechanical properties, Fuji IX was the only GIC that showed an increase in compressive strength during the 28-day evaluation. On the other hand, Ketac Molar showed the lowest cytotoxicity, while Maxxion R was the most cytotoxic GIC evaluated, and presented the highest fluoride release. In conclusion, Fuji IX

and Ketac Molar showed the most suitable physical and biological properties among conventional GICs evaluated.

Acknowledgement

The authors would like to thank the Foundation of Rio Grande do Sul -Brazil (DOCFIX 18/2551-0000515-0). This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (Capes) – Brasil – Finance Code 001.

Resumo

Objetivo: avaliar as propriedades físicas e biológicas dos cimentos de ionômero de vidro convencionais (CIVs). Metodologia: foram avaliados os seguintes CIVs: Fuji IX (GC Europe, Bélgica), Ketac Molar (3M ESPE, Estados Unidos), Maxxion R (FGM, Brasil) e Vitro Molar (Nova DFL, Brasil). O tempo de presa, a alteração dimensional, a radiopacidade, a sorção e a solubilidade em água foram avaliados para todos os materiais. A resistência à compressão foi analisada em intervalos de 1h, 24h, 7 dias e 28 dias; e liberação de íons fluoreto em 3h, 24h e 72h. A viabilidade celular foi avaliada após 24 e 48 horas com células de fibroblastos. A análise estatística foi realizada por meio do *software* SigmaPlot 12 (Systat Inc, San Jose, CA, EUA), com nível de significância estabelecido em $\alpha = 0,05$. Resultado: apenas o Fuji IX teve um tempo de presa dentro da faixa recomendada pela Especificação Padrão da ADA 96 (2012), não superior a 6 minutos. Vitro Molar e Maxxion R apresentaram radiopacidade que não estava de acordo com a especificação ADA 96 (2012). Maxxion R e Vitro Molar mostraram uma alteração dimensional estatisticamente semelhante. Quanto às propriedades mecânicas, o Fuji IX foi o único CIV que apresentou aumento da resistência à compressão durante o período de avaliação de 28 dias. O Ketac Molar apresentou a maior viabilidade celular, enquanto o Maxxion R apresentou citotoxicidade severa e o maior valor cumulativo de liberação de flúor. Conclusão: Fuji IX e Ketac Molar apresentaram as propriedades físicas e biológicas mais adequadas entre os CIVs avaliados.

Palavras-chave: cimentos de ionômero de vidro; citotoxicidade; materiais dentários; técnicas *in vitro*.

References

1. Moberg M, Brewster J, Nicholson J, Roberts H. Physical property investigation of contemporary glass ionomer and resin-modified glass ionomer restorative materials. 2018; 23(3):1295-1308.
2. Croll TP, Nicholson JW. Glass ionomer cements in pediatric dentistry: review of the literature. *Pediatr Dent* 2002; 24(5):423-9.
3. Virupaxi S, Pai R, Mandroli P. Retentive strength of luting cements for stainless steel crowns: A systematic review. *J Indian Soc Pedod Prev Dent* 2020; 38(1):2-7.
4. Faridi MA, Khabeer A, Haroon S. Flexural Strength of Glass Carbomer Cement and Conventional Glass Ionomer Cement Stored in Different Storage Media Over Time. *Med Princ Pract* 2018; 27(4):372-7.
5. Amorim RG, Frencken JE, Raggio DP. Survival percentages of atraumatic restorative treatment (ART) restorations and sealants in posterior teeth: an updated systematic review and meta-analysis. *Clin Oral Investig* 2018; 22(8):2703-25.
6. Spezzia S. Cimento de ionômero de vidro: revisão de literatura. *J Oral Investig* 2018; 6(2):74.
7. Forsten L. Fluoride release from a glass ionomer cement. *Scand J Dent Res* 1977; 85(6):503-4.
8. Forsten L. Fluoride release and uptake by glass-ionomers and related materials and its clinical effect. *Biomaterials* 1998; 19(6):503-8.
9. Menezes-Silva R, Cabral RN, Pacotto RC. Mechanical and optical properties of conventional restorative glass-ionomer cements - a systematic review. *J Appl Oral Sci* 2019; 27.
10. Algera TJ, Kleverlaan CJ, Prah Andersen B, Feilzer AJ. The influence of environmental conditions on the material properties of setting glass-ionomer cements. *Dent Mater* 2006; 22(9):852-6.
11. Wren AW, Coughlan A, Hall MM, German MJ, Towler MR. Comparison of a SiO₂-CaO-ZnO-SrO glass polyalkenoate cement to commercial dental materials: Ion release, biocompatibility and antibacterial properties. *J Mater Sci Mater Med* 2013; 24(9):2255-64.
12. Santos RL dos, Pithon MM, Leonardo JBP, Oberosler ELC, Vaitsman DS, Ruellas AC de O. Orthodontic cements: Immediate protection and fluoride release. *Dent Press J Orthod* 2012; 17(4):5-10.
13. Lima RBW e, Farias JFG de, Andrade AKM, Silva FDS da CM e, Duarte RM. Water sorption and solubility of glass ionomer cements indicated for atraumatic restorative treatment considering the time and the pH of the storage solution. *Rev Gaúch Odontol* 2018; 66(1):29-34.
14. Nicholson JW. Chemistry of glass-ionomer cements: a review. *Biomaterials* 1998; 19(6):485-94.
15. Stona P, Bertella SM, Rockenbach MIB, Holderbaum RM, Weber JBB. Radiopacities of Glass Ionomer Cements Measured With Direct Digital Radiographic System. *J Dent Child* 2012; 79(2):59-62.
16. Walia R, Jasuja P, Verma K, Juneja S, Mathur A, Ahuja L. A comparative evaluation of microleakage and compressive strength of Ketac Molar, Giomer, Zirconomer, and Ceram-x: an *in vitro* study. *J Indian Soc Pedod Prev Dent* 2016; 34(3):280.
17. Lohbauer U. Dental glass ionomer cements as permanent filling materials? -Properties, limitations and future trends. *Materials (Basel)* 2010; 3(1):76-96.
18. Bonifácio CC, Kleverlaan CJ, Raggio DP, Werner A, De Carvalho RCR, Van Amerongen WE. Physical-mechanical prop-

erties of glass ionomer cements indicated for atraumatic restorative treatment. *Aust Dent J* 2009; 54(3):233-7.

19. Olegário IC, Pacheco AL de B, de Araújo MP, Ladewig N de M, Bonifácio CC, Imparato JCP, *et al.* Low-cost GICs reduce survival rate in occlusal ART restorations in primary molars after one year: A RCT. *J Dent* 2017; 57:45-50.
20. Pacheco AL de B, Olegário IC, Bonifácio CC, Calvo AFB, Imparato JCP, Raggio DP. One year Survival Rate of Ketac Molar versus Vitro Molar for Occlusoproximal ART Restorations: a RCT. *Braz Oral Res* 2017; 31.
21. Bonifácio CC, Hesse D, Raggio DP, Bönecker M, Van Loveren C, Van Amerongen WE. The effect of GIC-brand on the survival rate of proximal-art restorations. *Int J Paediatr Dent* 2013; 23(4):251-8.
22. De Caluwé T, Verduyck CWJ, Ladik I, Convents R, Declercq H, Martens LC, *et al.* Addition of bioactive glass to glass ionomer cements: Effect on the physico-chemical properties and biocompatibility. *Dent Mater* 2017; 33(4):186-203.
23. Mota ACC da, Motta LJ, Santos EM, Guedes CC, Alfaya TA, Bussadori SK. Cytotoxic effect of glass ionomer cements in cell culture. *Rev Assoc Paul Cir Dent* 2014; 68(2):166-9.
24. Marczuk-Kolada G, Luczaj-Cepowicz E, Pawinska M, Holownia A. Evaluation of the cytotoxicity of selected conventional glass ionomer cements on human gingival fibroblasts. *Adv Clin Exp Med* 2017; 26(7):1041-5.
25. Stanislawski L, Daniau X, Lautié A, Goldberg M. Factors responsible for pulp cell cytotoxicity induced by resin-modified glass ionomer cements. *J Biomed Mater Res* 1999; 48(3):277-88.
26. Oguro A, Cervenka J, Horii K -i. Effect of Sodium Fluoride on Growth of Human Diploid Cells in Culture. *Pharmacol Toxicol* 1990; 67(5):411-4.
27. Chang YC, Chou MY. Cytotoxicity of fluoride on human pulp cell cultures *in vitro*. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2001; 91(2):230-4.
28. Lewis J, Nix L, Schuster G, Lefebvre C, Knoernschild K, Caughman G. Response of oral mucosal cells to glass ionomer cements. *Biomaterials* 1996; 17(11):1115-20.

Corresponding address:

Prof. Dr. Evandro Piva
Federal University of Pelotas, Faculty of Dentistry
Department of Restorative Dentistry
Gonçalves Chaves St., 457, room 503
Zip Code: 96015-560 – Pelotas, RS, Brazil
E-mail: evpiva@gmail.com
Phone: +55 53 32256741 (134)

Recebido: 03/04/2020. Aceito: 29/07/2021.