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Chapter 2

Physical-mechanical properties of GICs indicated for Atraumatic Restorative Treatment (ART)

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Abstract

Aim: This study evaluated mechanical properties of glass-ionomer cements (GICs) used for Atraumatic Restorative Treatment. Wear resistance, Knoop hardness (Kh), flexural (F_s) and compressive strength (C_s) were evaluated. The GICs used were Riva Self Cure (RVA), Fuji IX (FIX), Hi Dense (HD), Vitro Molar (VM), Maxxion R (MXR) and Ketac Molar Easymix (KME).

Methods: Wear was evaluated after 1, 4, 63 and 365 days. Two-way ANOVA and Tukey *post-hoc* tests (p=0.05) analyzed differences in wear of the GICs and the time effect. F_s, C_s, and Kh were analyzed with one-way ANOVA.

Results: The type of cement (p<0.001) and the time (p<0.001) had a significant effect on wear. In early-term wear and Kh, KME and FIX presented the best performance. In long-term wear, F_s and C_s, KME, FIX and HD had the best performance. Strong explanatory power between F_s and the Kh (r^2=0.85), C_s and the Kh (r^2=0.82), long-term wear and F_s of 24 h (r^2=0.79) were observed.

Conclusions: The data suggest that KME and FIX presented the best in vitro performance. HD showed good results except for early-term wear.

Introduction

Since glass-ionomer cements (GICs) were introduced in the 70's by Wilson and Kent (1) they have undergone constant improvements in order to follow market trends and to fulfill many functional and aesthetic requirements (2, 3). These materials are inexpensive compared to resin composites and less demanding with respect to the clinical application. By increasing the powder/liquid ratio, the high viscous or condensable GICs, with better mechanical properties than tradition al GICs were developed for the Atraumatic Restorative Treatment (ART) (4). GIC is the material of choice for ART due to its physical and chemical properties. Such properties include its adhesion to dental structures, biocompatibility, chemical set reaction, and fluoride release/uptake, which contributes GIC's preventive character (1, 2, 5). One of the major drawbacks of GIC is the relatively low fracture strength and higher occlusal wear rate in comparison to amalgam and modern resin composite materials (6).

Renewed interest in the study of GICs is due to their good performance in recent clinical trials (6-8). Van 't Hof *et al*. (8) concluded based on a meta-analysis, that single-surface ART restorations using high-viscosity GIC in both primary and permanent dentitions showed high survival rates, and that medium-viscosity (traditional) GIC should not be used for ART restorations. The reported clinical failure rates in multi-surface ART restoration are due to gross marginal defect, secondary caries, loss of retention and fracture of ART restorations (9, 10). Gross marginal defects were induced by occlusal forces or insufficient wear resistance of the restorative material (10). Taken this into account, the use of GIC in pediatric restorative dentistry would be still adequate of the relative low occlusal forces applied to the restorations and their reduced time in the oral cavity. Furthermore, GIC releases and uptakes fluoride, adhere chemically to the tooth structure, and can be used in a variety of clinical scenarios (11). Based on these previous arguments, special attention should be paid to the mechanical properties of GICs. Therefore, a better understanding of GICs flexural and compressive strength, wear resistance and hardness is essential.

The development and diffusion of restorative treatment techniques has stimulated dentistry manufacturers expand the number of GICs indicated for ART (high-viscosity and better mechanical properties GICs). In this study, six commercially available conventional GICs were investigated. These included Ketac Molar Easymix and Fuji IX, which were used as reference materials as they are the most frequently reported materials in in vivo and in vitro studies (12-15). The aim of this study was to investigate the mechanical properties of GICs used for ART. The wear resistance, flexural and compressive strength, and Knoop hardness were evaluated.

Material and Methods

The restorative GICs used in this study are listed in Table 1, together with the manufacturer and batch code data. The GICs used are hand-mixed versions and were used in accordance with the procedures supplied by the manufacturers.

Three-body wear was evaluated with the ACTA wear machine (15). This device consisted of two motor-driven cylindrical wheels rolling over each other with a surface slip of 15%, inside a bowl containing a third body medium, consisting of a slurry of rice and

millet seed shells (pH = 7). The specimen wheel and stainless steel were pressed against each other with a spring force of 15 N. A test run consisted of 200,000 cycles (55.5 hours) of the specimen wheel at a rotational speed of 1 Hz (4). The specimen wheel consisted of 10 compartments, each containing approximately 1 g of cement. For Riva, Hi Dense, Vitro Molar and Maxxion R, two compartments were filled with the cement. Ketac Molar Easymix and Fuji IX served as reference material and therefore only one compartment was filled with these cements. The specimen wheel was kept wet at 37°C at all times throughout a period of one year. Four wear runs were performed on this specimen wheel. The first run starting six hours after the preparation of the specimen, and the three subsequent ones started after 4, 63, and 365 days. After each run, 10 tracings were taken at fixed positions on the worn surface of each pair of specimen (PRK profilometer No. 720702, Perthen GmbH, Hannover, GE) so the loss of material (lm) could be measured.

The flexural strength (Fs) was measured according to the ISO Standard 9917-2 using 25 x 2 x 2 mm bar-shaped specimens (n = 10). After setting (10 minutes), the specimens were removed from the moulds and placed into 37°C paraffin. After 24 hours, the height and width of the specimens were measured using a digital micrometer to an accuracy of 0.01 mm. The specimens were subjected to a three-point-bending test (the distance between the two supports is 20.0 mm) on a universal testing machine (Mini Instron no. 4442, Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/min. The Fs was calculated with the following equation:

$$F_S = \frac{3Fl}{2wh^2}$$

where F is the load at fracture, l the distance between the supports (20.0 mm), w the specimen width and h the specimen height. The compressive strength (Cs) was measured analogue to the flexural strength, e.g., same storage conditions, testing machine, crosshead speed and number of specimens per cement, using cylindrical specimens (n = 10) with 4 mm diameter and 6 mm height (according to the ISO Standard 9917-1). The Cs was calculated with the following equation:

$$C_S = \frac{F}{\frac{1}{4}\pi d^2}$$

where F is the load at fracture, d the diameter, and h the specimen height.

The Knoop microhardness was determined with a hardness test machine (HM 124 – Mitutoyo, Japan) with 25 g load and a 30-second dwell time (16). Two specimens of each GIC were prepared in PVC moulds with a diameter of 8 mm and a height of 4 mm. Each hole was filled with a small excess of cement and after 10 minutes the specimens were stored in paraffin for 24 hours at 37°C (17). Prior to testing, the specimens were polished with 1200 grit paper (Buehler) until the excess was removed. Five indentations were taken in two specimens (n = 10).

Two-way ANOVA and Tukey post hoc tests (p=0.05) were used to test differences in wear of the GICs and the effect of time. The flexural strength, compressive strength and Knoop microhardness were analyzed with one-way ANOVA. Regression analysis was used in order to find the explanatory power (r^2) of each tested properties over another. The software used was Sigma Stat 3.1 (SPSS Inc., Chicago, USA).

Table 1 Materials used in this study.

Code	Product	Manufacturer	Color	Batch No.	Expiring Date
RVA	Riva Self Cure	SDI (Bayswater, VIC, AU)	A2	50303	2008/03
FIX	Fuji IX	GC Europe (Leuven, BE)	A3	510031	2008/10
HD	Hi Dense	Shofu (Ratingen, GE)	*	100630-5	2012/10
VM	Vitro Molar	DFL (Rio de Janeiro, BR)	*	5070823	2007/07
MXR	Maxxion R	FGM (Joinville, SC, BR)	A2	200706	2008/07
KME	Ketac Molar Easymix	3M/ESPE (Seefeld, GE)	A3	243914	2007/10

* There were no color specifications.

Results

The wear of different hand-mixed GICs are summarized in Table 2 and graphically depicted in Figure 1. Two-way ANOVA showed that the type of cement (F=2371.7; $p<0.001$) and the time (F=2965.6; $p<0.001$) had a significant effect on wear. Tukey post hoc test ($p<0.05$) showed that cement wear decreased significantly for all time spans measured in the one-year period.

Table 2 Mean wear and standard deviation in parentheses in μm at different time periods for the investigated materials.

Day	Riva	Fuji IX	Hi Dense	Vitro Molar	Maxxion R	Ketac Molar Easymix
1	124.2 (11.8)	105.0 (2.7)[a]	184.7 (19.7)	198.8 (9.0)	146.2 (6.9)	100.9 (1.1)[a]
4	99.3 (11.3)	85.8 (4.1)	225.9 (5.1)	185.0 (7.3)	111.0 (2.1)	74.4 (1.1)
63	79.0 (2.1)[bc]	74.2 (2.2)[c]	84.4 (6.7)[b]	n/a	80.0 (4.7)[bc]	57.7 (1.6)
365	60.6 (9.6)	44.5 (1.8)[d]	4.4 (1.6)[d]	123.2 (2.1)	68.8 (9.8)	44.9 (4.8)[d]

Means with the same letter are not significantly different (p>0.05).

Ketac Molar Easymix presented the lowest wear in comparison to the other materials, with significant difference in the 4 and 63 days time-frames. For the early-term wear test (1 day), Ketac Molar Easymix and Fuji IX had the lowest wear rate, and Vitro Molar and Hi Dense the highest. On the other hand, for the one year time-frame the best performance was found for Ketac Molar Easymix, Fuji IX and Hi Dense.

The flexural and compressive strength together with the Knoop hardness are summarized in Table 3. One-way ANOVA showed significant difference for Fs (F=9.2; $p<0.001$), Cs (F=4.8; $p=0.001$) and Knoop hardness (F=24.3; $p<0.001$). The highest flexural and compressive strength was achieved by Ketac Molar Easymix, Fuji IX and Hi Dense, respectively. Both Riva and Vitro Molar had low flexural and compressive strength. Maxxion R performed well in the strength tests, showing no statistical significant difference in the flexural strength in relation to Ketac Molar Easymix, Fuji IX, Hi Dense and Riva. For the compressive strength Maxxion R test, results were different only from Ketac Molar Easymix. The Knoop hardness of Ketac Molar Easymix and Fuji IX was significantly higher than the other cements. Vitro Molar and Riva showed the lower Knoop hardness. A strong explanatory power between the flexural strength and the Knoop hardness ($r^2=0.85$) and the

compressive strength and the Knoop hardness (r^2=0.82) was observed. The explanatory power between the flexural and compressive strength was lower (r^2=0.61). An even smaller explanatory power between the wear at day 1 and the flexural strength, compressive strength and the Knoop hardness was found ($r^2 < 0.52$). The long-term wear after one year showed a strong explanatory power with the flexural strength of 24 hours (r^2=0.79). No other strong explanatory coefficients were found.

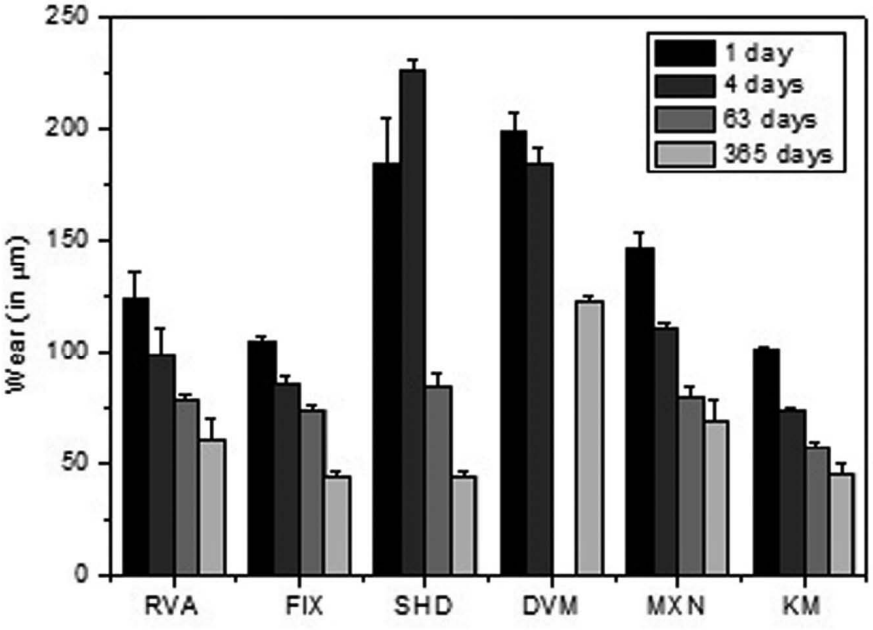

Figure 1 Bar graph showing the wear in µm per 200,000 cycles on the investigated materials at slip of 15% and 15N force.

Discussion

The hand-mixed GICs were chosen for this study as they are commonly used in clinical situations with a lack of electricity. In contrast, encapsulated cements were dispensed beforehand, which minimized operator-induced variability. As the mixing is automatic in this type of GIC, it is possible to add more powder in the mixture, resulting in better mechanical properties, as was suggested in a previous study (4). Some of the GICs used in this study are not available in an encapsulated version; therefore, for standardization all GICs used were hand-mixed versions.

The effectiveness of single-surface ART restorations is already evidence based (7, 8) as the survival rates for the high viscosity GICs reached very acceptable levels. However, for multi-surface restorations the survival rates are reported to be less satisfactory (12 to 76%) (6, 10, 18). The most common reasons given for ART class II GIC restoration failures are gross marginal defect, loss of retention, fracture of ART restorations and secondary caries (9, 10, 13, 14). In a study carried out under ideal clinical conditions, the main reason for failure of class II restorations in permanent teeth was the loss of GIC in the proximal area, leading

to loss of proximal contact (6). Based on this evidence, the search for materials with strong mechanical properties is necessary in order to accomplish better survival rates with multi-surface ART restorations. The current study evaluated flexural and compressive strength, as well as the Knoop hardness and wear resistance for GICs, characteristics that are useful in identifying appropriate materials for ART.

When studying the wear resistance of GICs, de Gee *et al*. (15) found high early wear rates with different GICs compared with the present study. The authors observed that wear rates declined over time and a reduction in wear was still evident even between 4 months and 1 year. Our results also showed decreasing values from the first to the last experiment with the exception of Hi Dense between 1 day and 4 days. The reason for a high value for the 4 days measurement of Hi Dense can be explained by its composition (19). Hi Dense is a silver reinforced GIC. The silver particles in the powder interfere in the setting reaction, making it lengthy. Concurrently, the final mixture is also thought to suffer from a lack of cohesion. Therefore, it is assumed that the interfacial bonding between the particles and the polymer matrix can be influenced by the different powder particles (16). This could be why Hi Dense presented high wear test results in the first experiments. But even with these characteristics in the early-term wear, Hi Dense presented similar wear rates to the reference material in the long-term. Preliminary studies have shown that highly viscous GICs have either comparable or superior mechanical properties and wear resistance to metal-reinforced cements (20, 21). The Vitro Molar wear at two months (63 days) could not be measured due to failure of the specimen reference. In the wear experiment, approximately 80 per cent of the surface of the specimen wheel was subjected to three body wear by the antagonist wheel, leaving two unworn ridges on the sides of the specimen wheel. These unworn ridges serve as references. If the investigated material is weak or has a very high wear rate, fracture of such a ridge is sometimes observed. This fact can be translated to a clinical situation where the restoration will fail because of a fracture or a large loss of material.

Table 3 Mean flexural strength (F_s in MPa), compressive strength (C_s in MPa), and the Knoop hardness (in MPa) and standard deviations in parentheses for the investigated materials (after 24 hours).

Material	Flexural Strength (F_s)	Compressive Strength (C_s)	Knoop hardness (Kh)
Riva	23.9 (10.7)[ab]	126.5 (18.5) [a]	38.7 (12.9)[ab]
Fuji IX	33.3 (6.1) [c]	166.7 (31.3)[ab]	68.7 (10.9) [d]
Hi Dense	33.3 (3.9) [c]	159.2 (26.7)[ab]	55.8 (11.5) [c]
Vitro Molar	19.2 (4.0) [a]	135.7 (48.3) [a]	37.0 (5.5) [a]
Maxxion R	29.5 (3.6)[bc]	130.3 (23.2) [a]	50.4 (5.3)[bc]
Ketac Molar Easymix	34.5 (7.2) [c]	177.8 (28.2) [b]	73.8 (9.4) [d]

Means with the same letter, within F_s, C_s or Kh are not significantly different ($p>0.05$).

Compressive strength is often used as a measure of the ability of a material to withstand masticatory forces. The obtained compressive strength for Ketac Molar Easymix

and Fuji IX matched with previous reported values (22), but were lower compared to values obtained by Peez and Frank (23). The three-point-bending test can be regarded as representative of a clinical situation of the forces exerted by the opposing cusp (24). The flexural strength values found, corresponded well with previous reported values (20).

Nevertheless, the low flexural strength for Riva and Maxxion R of 23.9 and 19.2 MPa, respectively, can be viewed as the Achilles heel of these materials when used in multi-surface restorations. The microhardness can be defined as the resistance of a material to indentation or penetration. In concordance with the flexural strength, the hardness of Riva and Maxxion R was also significantly lower than the other materials studied. The reported microhardness values of Xie *et al*. (16), which investigated the Knoop hardness for similar materials with the same methods but a storage time of 7 days, were higher than our values. This can be explained by the longer storage line.

A weak explanatory power was found between flexural and compressive strengths; this finding is in line with Xie *et al*. (16). Between the long-term wear and the flexural strength a strong explanatory power was found, suggesting that it is possible to forecast the long-term wear by using the flexural strength results. The ART technique was developed, in principle, for areas that lack electricity, in underprivileged communities of developing countries (25). Considering the success of single surface ART restorations, the technique has spread throughout the public dental health service and academic areas.

Conclusions

Within the limitations of this in vitro study, Ketac Molar Easymix and Fuji IX presented the best performance in all the tests. Hi Dense, the metal reinforced GIC, although presenting good strength results, showed very weak wear resistance. The worst performance was found for Vitro Molar. As Maxxion R achieved, in general, a satisfactory performance in comparison to Ketac Molar Easymix and Fuji IX, this material should be evaluated in clinical situations. The manufacturer should improve this material in order to make it possible for use on a larger scale by applying it in social projects, clinical research and public dental health services.

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