Mechanical and Bioactive Properties of a Commercial Glass Carbomer: GCP Glass Fill

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Abstract
Glass carbomers, such as GCP Glass Fill, are a type of GICs, which have nanosized apatite added to their composition. The manufacturer describes this material to have remineralizing properties. This study aimed to evaluate the bioactivity of this material and the effect of finishing by thermocuring and gloss on its properties. Bioactivity was measured by Ca and PO4 release studies, SEM, FT-IR, as well as EDX. Mechanical properties were measured by a compressive strength test. Cements of all finishing types (with/without gloss and/or thermocuring) had comparable and acceptable initial compressive strengths. After an incubation period, all strengths decreased. Although GCP Glass Fill should be bioactive, no signs of bioactivity after incubation in SBF were observed. Moreover, the finishing conditions with Gloss and thermocuring do not improve the mechanical properties of the cement. Therefore, this material is not superior to e.g. GICs.

Keywords: Bioactivity, GCP Glass Fill, Glass Carbomer, Gloss, Thermocuring

1. Introduction
Glass ionomer cements (GIC) are very attractive dental restorative materials. They are also used as adhesives since they can adhere to moist dental tissue and base metals. The chemical adhesion to enamel and dentin is achieved by the displacement of phosphate ions of Ap by the carboxylate groups of the polyacrylic acid (PAA). Moreover, free Ca2+ is chelated by these groups and hydrogen bonds with collagen can be formed. This PAA is a component, necessary to form a GIC, by the reaction with the basic aluminosilicate glass (ASG) (1, 2). These GICs also have anticariogenic properties due to the release of fluoride. This fluoride inhibits the metabolism of bacteria that cause caries. Moreover, the release of F- from the glass in the immediate surroundings of the cement can influence the natural demineralization/remineralization process of teeth by the formation of fluorapatite (FAp) instead of hydroxypapatite (HAp). This improves resistance of dental tissue to acid attack (2, 3). GICs also benefit from their thermal compatibility with tooth tissue 2. Among most restorative and luting cements, GICs have the lowest increase in temperature while setting, which improves their biocompatibility, especially in cements that have to be placed in close contact to the pulp (4). Despite these benefits, their applications are limited due to their low mechanical properties, such as compressive strength, flexural strength, (diametral) tensile strength and brittleness. Marginal and bulk fractures are the most common cause of failure (2, 5).

In order to improve these cements, recent studies are aiming at the bioactivation of GICs, so that they would form apatite (Ap) on or on their surfaces. The formation of calcium phosphate (CaP) as a layer on or in the direct surroundings of the cement is a parameter to the remineralization potential of a material and may make GICs even more appropriate for atraumatic restorative treatments (ART) or as filling material in caries-prone teeth (5-8). Those bioactive GIC may also potentially have better mechanical properties than the conventional GIC on the long term. The increased biocompatibility due to the formation of a CaP layer can namely enhance the interaction with bone or dental cells and consequently with natural bone or dentin. On top of the normal chemical bonding of GICs to dental/bone tissue, a mechanical interlocking occurs if GICs are made bioactive (9). In this way GICs could become more suitable for hard tissue replacements, not only in the dentistry field, but also in orthopedics (2). Moreover, a combination of GIC and pure synthetic Ap can improve the mechanical properties and may also improve bioactivity and biocompatibility of these cements. The PAA in the GIC itself aids this process as it partially dissolves HAp,
which releases Ca\(^{2+}\) and PO\(_4^{3-}\) ions that can remineralize and form a firm bond between the HAp and the rest of the GIC (10). However, it was seen that the size of the apatite crystals added to a GIC is important, which makes sense since Ap is prone to dissolution by the PAA. Particles in the nanosize-range decrease initial compressive strength, while those in the micrometer-range don’t alter or slightly improve compressive strength (10). These results are in contradiction with the results of Roche et al. who state that nanoparticles of HAp don’t influence initial strength and fracture toughness (11). Moshaverinia et al. even find that nanosized HAp and FAp increase initial and long-term compressive-, biaxial-, and flexural strength (12). Although it was aimed to obtain bioactive cements by the addition of Ap, studies showed a low remineralization potential of these cements. The limited bioactivity of the GIC, when apatite is added, can be explained by the polyacrylic acid (PAA) not only dissolving apatite, but at the same time consuming Ca\(^{2+}\) to set the cement (13-15).

Glass carbomers are a type of glass ionomer cements, which have nanosized FAp and HAp added to their composition. The actual glass phase consists of an aluminosilicate glass, however, it is shown that this glass contains less network modifying ions and thus less non-bridging oxygens than regular ASG (16). During setting, a large amount of the hydroxyapatite can be dissolved and consumed by the PAA; thus, only a small amount of apatite (Ap) is present to induce remineralisation. Recently, glass carbomers are commercialized under the name of GCP Glass Fill, this material may be an interesting subject of research, certainly its bioactive properties as the manufacturer claims this material to have remineralizing properties. If this is the case, GCP Glass Fill could be an interesting reference material for further bioactivity studies. This material comes with very specific guidelines, which state that on top of the material, a gloss should be used, and that the material should be thermocured. The GCP gloss is a silicone-based coat. It protects the surface from exposure to moisture and saliva in the first reaction step as well as prevents dehydration in the second phase (17). In order to improve the mechanical properties, application of heat energy can be used so that the cement sets “on command”. The use of heat is supposed to accelerate the matrix-forming reaction of cGICs and GCP. Kleverlaan et al. measured an obvious relationship between temperature of the samples and compressive strength of cGICs. It was observed that raising the temperature of the surface of the cement to a maximum of 60°C significantly improved the surface hardness of the material after 24 hours (18). Thus, the manufacturer claims that these 2 guidelines lead to superior product characteristics.

The aim of this study is to evaluate the effect of thermocuring and gloss on the mechanical and bioactive properties of GCP Glass Fill cement. If good mechanical and bioactive properties would be observed, this material could be used as a reference for bioactive GICs or other dental filling materials developed in the future. A total of 4 types of finishing are tested: 1 control without gloss and thermocuring, 1 with thermocuring using a LED lamp, 1 with thermocuring at 65°C for an extended time in an oven to heat the core of the material, and 1 finished according to the manufacturer’s instructions with application of a gloss and thermocuring using a LED lamp.

2. Methods

GCP Glass Fill cement (A3, GCP Dental, Batch 7501759) was prepared according to the instructions of the manufacturer (GCP Dental, VD Ridderkerk, The Netherlands) using a GCP CarboMIX CM-02 MIXER (GCP Dental, VD Ridderkerk, The Netherlands). Depending on the evaluated properties, cylinders with different dimensions were prepared (Table 1). After application in the moulds, the cements were subjected to 4 types of finishing. For LED-thermocured cements, a GCP CarboLED CLO Lamp (GCP Dental) was used. One group of cements received a surface coating with 1 droplet of GCP surface gloss (GCP Dental, Batch 1407106). GCP Glass Fill cement, cured at 37°C, without additional finishing, was used as control (Table 1). To obtain flat surfaces, all cements were pressed between 2 glass plates after the cylinders were filled.

| Group          | Bioactive Properties (Ø 5 mm, H 1 mm) | Compressive Strength (Ø 4 mm, H 6 mm) |
|----------------|--------------------------------------|---------------------------------------|
| 37°C (control) | 1 h at 37°C, 85% RH                  | 24 h at 37°C, 85% RH                  |
| 65°C           | 1 h at 65°C                          | 1 h at 65°C                           |
| LED            | 1 min thermocuring                    | 1 min thermocuring at each side        |
| LED + Gloss    | Application of gloss                  | Application of gloss                   |
|                | 1 min thermocuring                    | 1 min thermocuring at each side        |

2.1. Characterization of the Mechanical Properties

Compressive strength was determined with a universal testing machine (LRX plus, Lloyd Instruments, Bognor...
Regis, UK). For that purpose, 18 cement-cylinders were prepared for each group (Table 1) using a split stainless steel mold. A total of 6 cylinders were directly tested (24 hours after mixing), 6 were subjected to an immersion in SBF, and 6 were immersed in H₂O each for 28 days. Cylinders were loaded at a rate of 1 mm/min.

2.2. Characterization of the Bioactive Properties

Simulated body fluid (SBF) was prepared with NaCl (VWR Prolabo 27810.295), NaHCO₃ (Merck 6329), KCl (Merck 4936), K₂HPO₄ (Merck 5104), MgCl₂·6H₂O (Merck 5833), CaCl₂ (Merck 2239), Na₂SO₄ (Merck 6647), and Tris (hydroxymethyl) aminomethane (VWR Prolabo 103156X). These components were dissolved in deionized water (Millipore, Milli-Q Academic, Bedford, MA, USA) as described in the protocol of Kokubo et al. (19). The pH was adjusted to 7.4 with a 1M HCl solution.

Cements were made as described previously and transferred into a mold (Ø 5 mm, H 1 mm). The maturation conditions for the cements in the molds are described in Table 1. Two times, 2 discs of the same cement batch were stored in a plastic container with 25 mL SBF at 37°C for 28 days. For the first 2 discs, SBF was not changed. For the other 2 discs, SBF was changed every 2 days. And for 2 weeks, the old SBF was collected.

The collected SBF was used to determine the phosphate and calcium uptake from SBF by the cement discs. PO₄³⁻ was determined with a differential spectrophotometric method using a Pye Unicam PU 8670 VIS/NIR spectrophotometer (Philips Scientific Equipment, Brussels, Belgium). Ca²⁺ was determined with atomic absorption spectrometry (AAS) (Varian SpectrAA-30, Agilent Technologies, Santa Clara, USA) with an air-acetylene flame (20). The cumulative phosphate and calcium uptake from SBF (in %) was calculated.

FT-IR spectra of crushed cements (Ø 5 mm, H 1 mm) were recorded before and after 28 days of incubation in SBF using a Spectrum One spectrometer (Perkin Elmer Instruments, U.S.) for wavelengths between 4000 and 400 cm⁻¹. In particular, the formation of calcium phosphate was investigated. Therefore, the height of the phosphate peaks at 603 cm⁻¹ and 561 cm⁻¹ were measured in absorbance with Spectrum vs.0.1. software (Perkin Elmer Instruments, U.S.). In order to be able to compare the height of the peaks, identical amounts of crushed cements were mixed with KBr.

Scanning electron microscopic (SEM) images were taken of the surface and fracture surface of the discs (Ø 5 mm, H 1 mm) with a scanning electron microscope (FEI, Quanta FEG, Hillsboro, OR, USA with EDAX silicon-drift detector) and analyzed for the presence of calcium phosphate layer after 28 days of incubation in SBF. The cements, only matured 24h in RH, were used as control. EDX (FEI, Quanta FEI, Hillsboro, OR, USA with EDAX silicon-drift detector) was further conducted on specific structures seen on SEM to determine the atomic composition of these structures.

2.3. Statistical Analysis

The compressive strength was evaluated as a function of the type of finishing with ANOVA. Significant differences between means were determined with a multiple comparison Bonferroni test.

3. Results

3.1. Characterization of the Physico-Mechanical Properties

ANOVA (Table 2) shows that the compressive strength is significantly influenced by the finishing (P = 0.000) and incubation type (0.003), however, apparently the effect of the incubation on the compressive strength does not depend upon the finishing of the GIC (P = 0.808). Nonetheless, there is no significant difference in the initial compressive strength between the groups (P = 1.000) (Figure 1). When the discs are subjected to an immersion in SBF or H₂O, compressive strengths decrease, however, only significantly for cements cured with lamp as well as gloss (P < 0.03) and control cements without additional finishing, immersed in H₂O (P = 0.025). There is no difference between compressive strengths after incubation in SBF or H₂O for each group separately (P = 1.000). After incubation for 28 days, there is also no significant difference in compressive strengths between groups (P > 0.416).

![Figure 1. Compressive Strength of GCP Cements as a Function of Type of Finishing Before and After 28 Days Incubation in H₂O and SBF](image)

3.2. Bioactive Properties

Ca-uptake profiles show that the highest amount of Ca²⁺ is taken up by GCP finished at 65°C for 1h. In contrast, finishing according to the manufacturer’s instructions (with application of gloss and LED thermocuring)
showed the least Ca-uptake. For the other 2 groups, Ca-uptake does not differ from each other, taking the error on the measurement in account (Figure 2A).

Phosphate uptake is highest for the control GCP cement (without additional finishing). Uptake decreases when the LED lamp was used to heatcure the cement. Phosphate is released when cement was finished at 65°C and is highest when the cement is handled according to the manufacturer's instructions (Figure 2B).

FT-IR spectra show (Figure 3) that PO4-3 peaks of GCP powder at 561cm⁻¹ are approximately 3x higher than those of the cements, irrespective of the cement finishing type. After an incubation period of 28 days in SBF, PO4-3 peak height does not increase or decrease significantly on or in the cements.

Figure 4A is a SEM image of GCP Fill powder. Two types of particles can be distinguished, marked with S (smooth) and small I (irregular) particles, lying on a smooth particle. EDX of these glass particles shows that S has a composition typical for ASG (Figure 5A), while I contains more Ca and P (Figure 5B).

Figure 4B illustrates cement surfaces before incubation (left) and after incubation in SBF (right), visualized by SEM. The surfaces of the cements show no depositions before and after incubation in SBF. All surfaces have a “glazy” appearance due to the formed matrix. Surfaces of cements with a LED + Gloss finishing are more irregular.

The inner core of all cements show small spherical structures (Figure 4C), which are most abundant in cements cured at 65°C before and after incubation and are characterized as calciumphosphate by EDX (Figure 5C).

4. Discussion

SEM analysis of the glass phase of GCP glass fill showed 2 different types of particles. The large particles with smooth surfaces were identified as aluminosilicate glass by EDX analysis. The smaller particles with irregular appearance showed a high Ca and P content and were identified as calciumphosphate. FT-IR analysis of the glass phase clearly showed the presence of Ap. Since Si and Al were still detected by EDX, the irregular particle might merely be an ASG-particle covered with small Ap particles.

All experiments were executed with cements of the same batch number of GCP Glass Fill. Differences in mechanical and bioactive properties can thus only be ascribed
All cements had comparable initial compressive strengths. Moreover, compressive strength of GCP Glass Fill is comparable to that of high viscosity GICs (21). Chen et al. even found that high viscosity GICs had better retention rates and a higher caries preventive effect in vivo than glass carboxomers. Added energy to the high viscosity GICs also did not lead to significantly better results (22-24). After 28 days of maturation in H$_2$O or SBF, compressive strengths of all cements decreased. This is in line with one of our previous experiments with GICs (25), however, contrary to the outcomes of other research groups on conventional GICs (2, 26). The compressive strengths of the cements with both gloss applied and thermocured decreased significantly. This gloss does not set by thermocuring. Therefore, despite the fact that the gloss should protect against early moisture sensitivity, the decrease of strength can thus be ascribed to the outer layer of the cements, which is diluted by the gloss (17). As such, ions are more easily leached and cannot contribute to the strength of the cements anymore. Literature shows that the overall survival rate of carbomer fillings seems to be lower than that of composites or high viscosity glass ionomer cements (22-24). This may also be a result of decreased strength and/or marginal leakage as all cements (except those with gloss applied, Figure 4B) show cracks all over the surface and enamel interface, probably caused by local dehydration (27) or the formation of a weak Ca$^{2+}$ rich matrix. This is in contrast to conventional GIC, which are strongly chemically bonded to tooth structure. When gloss is applied, which should protect against dehydration, although the mouth environment is moist, those cracks are not visible on the plain surface. However, with a silver nitrate solution, the cracks are visible and filled with gloss. As previously explained, also in these cracks, the gloss may dilute part of the cement (28). GCP, without a finishing treatment, also showed a significant decrease in strength after 28 days of incubation in H$_2$O. Although some studies state that heat-curing does not seem to have an effect on both marginal leakage, compressive or flexural strength and survival rate of GICs or GCP (17, 22, 23, 28), cements thermocured by LED (without gloss applied) or at 65°C did not decrease as much on maturation compared to cements without additional finishing. At higher temperatures, a stronger matrix may be formed, which is less easily dissolved upon immersion in water.

None of the cements within 1 group of finishing treatment showed a difference in strength when incubated in SBF or when incubated in H$_2$O. This is in contrast with 1 of our previous studies where BAG was added to a GIC and Ap was formed as a layer on the cement surface upon immersion in SBF (29). Therefore, a difference in decrease in strength between immersion in SBF and H$_2$O was at-
Figure 4. SEM of the glass Particles Distinguished in the GCP Glass Fill powder (A); Surface of the Cements Cured at 37°C, at 65°C, with the Lamp and with Lamp and Gloss (B); Fracture Surface of Cement Cured at 65°C (C), Incubated in H\textsubscript{2}O and SBF for 28 Days.

Figure 5. EDX of the Smooth (A) and Irregular (B) Powder Fractions and of the Spherical Structures on the Inner Side of the Cements Cured at 65°C (C).

Contributed to the growing Ap layer in SBF. Since strengths are comparable between immersion conditions in the present study, it may be assumed that no Ap layer is formed.

GCP from the same batch was used in these exper-
iments. Therefore, the phosphate and calcium release –and/or uptake profiles may indicate the most reactive and thus bioactive cements. The results must however be interpreted with caution. Normally, a highly bioactive cement will release a high amount of Ca\(^{2+}\) and take up a low amount of phosphate (30). In this study, none of the cements released Ca\(^{2+}\), which corresponds with the findings of SEM and long term compressive strength that no bioactivity was observed on/in the GCP cements. Cements prepared according to the manufacturer’s finishing instructions (gloss + lamp) had the lowest Ca\(^{2+}\)-uptake and the highest PO4\(^{3-}\) release. These results can be substantiated by the loss of ions due to the dilution of the outer layer of the cement in gloss (17). Further in time, the applied gloss may form a barrier for the release and/or uptake of calcium and phosphate ions. This is in line with literature as the release of fluoride from a GIC is inhibited as well when a bonding agent is applied (31). Cements cured at 65°C take up most Ca\(^{2+}\) and release PO4\(^{3-}\) and should thus be the least bioactive according to Baghbani et al. (30). The fast formation of the cement matrix at high temperatures might indeed impede bioactivity. The fact that these cements set faster than cements cured at lower temperatures is clear from the fact that agglomerates of Ap are visible in the fracture surfaces on SEM. In cements with longer maturation, the PAA has more time to react with the Ap. Finally, cements without additional finishing and cements heat-cured with a LED lamp equally take up calcium and phosphate, thus, both lack bioactivity. All phosphate and calcium release –and/or uptake profiles, however, are in a similar range and indicate that on none of the cements Ap is formed.

FT-IR also substantiates these findings as the increase or loss of apatite after 28d incubation in SBF is fairly insignificant. This fits with the literature, as Zainuddin et al. showed that after 10 months ageing, still no sharp apatite peaks were visible with NMR of carbomer cements (16).

4.1. Conclusions

In conclusion, it can be seen that GCP Fill cement has comparable and acceptable initial compressive strengths, irrespective of the type of finishing. Only when cements are cured, according to the manufacturer’s instructions, strength decreases significantly after 28 days incubation. None of the GCP Fill cement with different finishing treatments showed bioactivity and in this respect this dental filling material cannot serve as reference material to compare with bioactivity of experimental GICs.

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