Mechanical properties and water sorption of two experimental glass ionomer cements with hydroxyapatite or calcium fluorapatite formulation

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INTRODUCTION

Glass ionomer cements (GICs) have been widely used in clinical dentistry since 1971. They can be used as a lining, bonding, luting, sealing or restorative material. Compared to the conventional restorative materials such as resin-based composites, the advantages of GICs can be listed as fluoride release, anticariogenicity, biocompatibility, similar thermal expansion coefficient to natural tooth, intrinsic radiopaque response and ease of use1-3. On the other hand, their low fracture toughness, slow setting reaction and moisture sensitivity are regarded as the main drawbacks of these cements4,5.

GICs are formed by an acid-base reaction of aluminosilicate glass and an aqueous solution of polyalkenoic acid and set in two stages; the gelation phase which generally takes 10 min after mixing followed by the hardening phase including the slow release of the Ca²⁺ and Al³⁺ within the matrix and the formation of aluminum salt bridges6. The material becomes very sensitive to water uptake that may soften the surface in the first stage, while it is very susceptible to dehydration during the second step6. Early water exposure or dehydration significantly alters the final mechanical strength of the restorations6.

To overcome the disadvantages mentioned above several attempts have been tried over the years. To avoid the problems within the early stage, protection of the GIC’s surface is recommended. Petroleum jelly, different types of varnishes or light-cured resins have been used to sustain moisture levels in GICs. Recently, nanofilled, resin based light cured self-adhesives were introduced to improve the esthetics and physical properties of GICs. The main purpose of coating is, by covering the top surface of the restoration to protect against to saliva or dehydration until the setting reaction is completed and to increase wear resistance6,9.

Furthermore, heat application by a light-curing unit during setting has been also suggested. Heat application is supposed to accelerate the setting reaction of GICs and thus getting over the initial critical setting period before exposure to saliva6,11.

Considerable studies have been continued to enhance the mechanical or biological characteristics of GICs by incorporating several additives such as chlorhexidine, nanoclays, amino-acids, fibers, metallic powders, bioactive glass particles, montmorillonite clay, hydroxyapatite (HA) and fluorapatite (FA) to the GIC12-17.

Recently, it has shown that HA has promising advantages in restorative dentistry, with its hardness similar to natural tooth, intrinsic radiopaque response and biocompatibility. HA has been reported as a successful reinforcement material for adhesive bone cements and resin composites18,19 and has been found to interact with via carboxylate groups in the polyacid of GICs20.

In previous studies, the researchers added HA with nano particle sizes and different weight percentages (10-28%) to GIC powder or polyacid components at different P/L ratios (1.00, 1.25, 1.75, 2.33, 2.50, 3.60) by mixing procedures experimentally and it was shown that the addition of HA to GIC can increase its flexural strength (FS) while maintaining its compressive strength and simultaneously enhancing fluoride ion release14,21,22. Porous, spherical HA particles were found to be the most effective in this regard16,17,23.

Other variables include the powder to liquid ratio, which in the case of GICs has a direct bearing on the concentration of reinforcing glass particles in the set
material. The concentration of reinforcing glass particles together with the presence of voids produced either by air inclusions or inadequate wetting of powder by liquid will have an influence on mechanical properties. Central to the ability to achieve an effective contact between powder and liquid and a set material with low porosity is the method chosen for mixing\(^2\).

On the other hand, FA or fluoridated HA were shown as having excellent biocompatible properties\(^2\). It is envisaged that the presence of HA and FA in the GIC matrix have the ability to increase the mechanical and bond strength of the resulting material. Therefore, the incorporation of HA or FA into GIC may not only improve the biocompatibility of GIC but also have the potential of enhancing the mechanical properties.

Additionally, the strength of GICs is affected by incorporated porosity and this is dependent on the method of mixing. Encapsulated materials have certain inherent advantages over hand-mixed materials. The manufacturer controls the ratio of components and the mixing is performed in a reproducible manner, which is normally quicker and cleaner than hand mixing. At the end of mixing the encapsulated material is often ready for immediate use by injecting directly from the capsule. For hand mixing, the ratio of powder and liquid is variable, due to the differences in cement powder packing densities achieved on rolling the scoop and the manner in which the bottle is held and the drop of liquid created. The vibratory action of the conventional mechanical mixing machines has been reported to incorporate increased porosity into some encapsulated luting cements and GI restoratives compared with their hand-mixed equivalents\(^2\). Recently for the study, prototype GICs having HA or calcium fluorapatite (CFA) already added by the manufacturer to the encapsulated forms were produced as trial materials.

Thus, the aim of this study was to verify if these HA or CFA containing formulated prototype encapsulated GICs show comparable performance to conventional GIC. The effect of coating, heat application, storage medium (artificial saliva and distilled water) and storage duration were also evaluated. The null hypotheses tested were that: (1) the prototype GICs would not show significantly better performances than conventional GIC in terms of mechanical properties (FS and Vickers hardness (VHN)) and water sorption (Ws), (2) application of coating, heating and aging would not change the above-mentioned properties, (3) storage in saliva or water (7 and 30 days) would not result in similar mechanical properties.

**MATERIALS AND METHODS**

The two prototype encapsulated GICs tested were the following: F9ap-sphere (GC, Tokyo, Japan) and F9ap-CFap (GC). Conventional glass ionomer restorative cement Fuji IX GP (GC) was used as control. The materials' properties were summarized in Table 1. Three treatment protocol groups were established: (1) as recommended by manufacturer (without coating-control group); (2) with coating and (3) heat application with LED curing unit (Translux Power Blue, Heraeus Kulzer, Hanau, Germany) without coating during setting.

**Specimen preparation**

Group 1 (n=120): The encapsulated GICs were tumbled

| Materials     | Type                           | Manufacturer | Composition                                                                 |
|---------------|--------------------------------|--------------|----------------------------------------------------------------------------|
| Fuji IX GP    | Conventional encapsulated glass ionomer cement | GC, Tokyo, Japan | Powder: 95% by weight alumino-fluoro-silicate glass with 5% polyacrylic acid powder. Liquid: 50% distilled water, 40% polyacrylic acid, and 10% polybasic carboxylic acid. Powder/liquid ratio: 3.6/1.0 |
| F9ap-sphere   | Experimental encapsulated glass ionomer cement | GC           | Powder: 87 wt% alumino-fluoro-silicate glass, 5 wt% polyacrylic acid powder, 8 wt% spherical hydroxyapatite. Liquid: 50% distilled water, 40% polyacrylic acid, and 10% polybasic carboxylic acid. Powder/liquid ratio: 3.6/1.0 |
| F9ap-CFap     | Experimental glass encapsulated ionomer cement | GC           | Powder: 87 wt% alumino-fluoro-silicate glass, 5 wt% polyacrylic acid powder, 8 wt% calcium fluorapatite. Liquid: 50% distilled water, 40% polyacrylic acid, and 10% polybasic carboxylic acid. Powder/liquid ratio: 3.6/1.0 |
| EQUIA-coat    | Low-viscosity nanofilled surface coating resin | GC           | 40–50% methyl methacrylate, 10–15% colloidal silica, 0.09% camphorquinone, 30–40% urethane methacrylate, 1–5% phosphoric ester monomer |

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\(^2\) Dent Mater J 2019; 38(3): 471–479
for 5 s to aerate the powder inside the capsule before activation. They were then mixed for 10 s using a mixing device (Softly™, Acteon New Zealand and Australia, Rosebery, Australia) according to the manufacturer’s instructions. Bar-shaped test specimens (2x2x16 mm³) were made using a half-split stainless steel mold. The tested materials placed on a glass slab, and covered with a transparent matrix strip. Another glass slab was pressed gently over the mold with finger pressure to evenly spread the material. The glass slab and transparent matrix strip were removed after GIC were set and the excess material was removed using a 1200 grit silicon carbide paper (LECO, St. Joseph, MI, USA)²⁷.

Group 2 (n=120): Specimens of this group were prepared in the same way as group 1. Then the coating material (EQUIA-Coat, GC) was applied with a micro-brush. All surfaces of the specimens were coated with the material without any pressure and polymerized by a LED curing unit (Translux Power Blue) with irradiance of 1,000 mW/cm² for 20 s.

Group 3 (n=120): Specimens were prepared same as group 1. Glass slab was immediately removed after the spread of the GIC, while the strip was maintained. LED curing unit was placed on top of the GIC surface immediately, so that it was in contact with the transparent matrix strip. To produce heat along the top surface of GIC specimens, the LED curing unit irradiance was used for three light exposures (3×20 s) during the setting reaction.

Specimens were removed from the molds and stored either in 50 mL artificial saliva (n=60) or in distilled water (n=60) at 37°C, and stored for 7 (n=30) or 30 (n=30) days. The distilled water and artificial saliva were changed twice a week; the volume of solution was 30 days—did not affect the FS values. The highest FS values were affected only from GIC material type difference was found statistically significant (p=0.007). FS values were affected only from GIC material type (η²=0.027, Table 3). The other parameters (preparation protocols: -coating or heating, -storage medium, -distilled water or artificial saliva and -storage duration —7 and 30 days—) did not affect the FS values. The highest FS values were observed when materials were prepared according to manufacturer’s recommendations. The lowest FS values were observed when specimens were coated (Fig. 1b). Specimens stored in distilled water showed higher FS values than in artificial saliva (Fig. 1c). FS values of all tested GICs decreased with the

**RESULTS**

The FS values and standard deviations of all groups were summarized in Table 2. Prototype GIC groups showed higher FS values than Fuji IX GP (Fig. 1a). This difference was found statistically significant (p=0.007). FS values were affected only from GIC material type (η²=0.027, Table 3). The other parameters (preparation protocols: -coating or heating, -storage medium, -distilled water or artificial saliva and -storage duration —7 and 30 days—) did not affect the FS values. The highest FS values were observed when materials were prepared according to manufacturer’s recommendations. The lowest FS values were observed when specimens were coated (Fig. 1b). Specimens stored in distilled water showed higher FS values than in artificial saliva (Fig. 1c). FS values of all tested GICs decreased with the
Table 2  Flexural strength (FS) was detailed in mean values (MPa) and standard deviations (SD)

| GICs       | Control (n=120) | Coat (n=120) | Heat (n=120) |
|------------|-----------------|--------------|--------------|
|            | Distilled water | Artificial saliva | Distilled water | Artificial saliva | Distilled water | Artificial saliva |
|            | 7 days 30 days  | 7 days 30 days | 7 days 30 days | 7 days 30 days | 7 days 30 days | 7 days 30 days |
| Fuji IX    | 9.2 (3.1)       | 11.5 (1.0)   | 11.8 (1.2)   | 11.0 (0.7)     | 11.7 (1.0)   | 11.3 (1.0)     |
| GP         | 8.7 (0.3)       | 10.1 (1.0)   | 10.7 (1.0)   | 11.0 (1.0)     | 10.5 (1.0)   | 11.8 (1.1)     |
| Fuji 9ap-  | 11.9 (0.8)      | 12.3 (0.5)   | 12.5 (0.8)   | 13.3 (0.6)     | 12.3 (0.6)   | 13.3 (0.6)     |
| sphere     | (30.6)          | (0.3)        | (0.9)        | (0.6)          | (0.8)        | (0.8)          |
| Fuji 9ap-  | 12.9 (0.7)      | 12.4 (0.9)   | 12.4 (0.9)   | 12.1 (1.2)     | 12.1 (0.6)   | 12.5 (1.0)     |
| CFap       | (1.2)           | (1.9)        | (1.9)        | (1.9)          | (0.6)        | (0.5)          |

*Same uppercase letter in same column indicates no significant difference (p<0.05).

Table 3  Influence of GIC type, treatment protocol, storage agent and storage duration on flexural strength (FS), Vickers hardness (VHN) and water sorption (Ws)

| GIC type     | FS    | VHN   | Ws   |
|--------------|-------|-------|------|
| Treatment protocols | ns    | 0.067 | ns   |
| Storage agent | ns    | 0.100 | ns   |
| Storage duration | ns    | 0.317 | ns   |

The higher the partial eta-squared values, the higher is the influence of the selected variables on the measured properties (general linear model (η²)).

ns: non-significant.
VHN values and standard deviations of all test groups are summarized in Table 4. The VHN values were affected by GIC material type ($\eta^2=0.331$), storage medium ($\eta^2=0.100$) and storage duration ($\eta^2=0.317$, Table 3). Fuji IX GP showed higher VHN values than experimental GICs (Fig. 2a) and higher VHN values were observed when the materials were prepared according to manufacturer’s recommendation ($\eta^2=0.067$, Table 3, Fig. 2b). These differences were found statistically significant ($p<0.001$). Specimens received heat application showed lowest VHN values (Fig. 2b). Storage in artificial saliva showed higher VHN values than storage in distilled water (Fig. 2c). The VHN values were decreased when the storage duration was increased (Fig. 2d).

The Ws values obtained for tested materials are presented in Table 5. The tested parameters (GIC type, preparation protocols, storage medium and storage duration) did not significantly affect the Ws values (Table 3).

Table 4  Vickers hardness (VHN) ($\text{N/mm}^2$) was detailed in mean values and standard deviations (SD)

| GICs      | Control (n=120) | Coat (n=120) | Heat (n=120) |
|-----------|-----------------|--------------|--------------|
|           | Distilled water | Artificial saliva | Distilled water | Artificial saliva | Distilled water | Artificial saliva |
|           | 7 days  | 30 days | 7 days  | 30 days | 7 days  | 30 days | 7 days  | 30 days |
| Fuji IX GP | 172.8  | (10.6)a | 152.2  | (31.8)a | 145.1  | (4.0)a | 127.9  | (36.5)a | 160.1  | (6.0)a | 135.3  | (12.6)a | 144.5  | (6.9)a | 128.8  | (26.5)a | 157.2  | (6.4)a | 131.6  | (22.0)a |
| Fuji 9ap-sphere | 125.3  | (5.2)b | 104.3  | (11.2)b | 134.4  | (8.0)b | 113.5  | (16.1)b | 146.9  | (5.3)b | 121.8  | (16.9)b | 129.3  | (6.3)b | 109.2  | (16.1)b | 146.7  | (6.8)b | 121.4  | (22.4)b |
| Fuji 9ap-CFap | 130.9  | (7.1)b | 110.9  | (21.1)b | 129.4  | (4.6)b | 109.8  | (15.1)b | 131.1  | (5.3)b | 106.3  | (14.0)b | 120.9  | (6.2)b | 100.6  | (22.9)b | 141.1  | (6.4)b | 116.7  | (20.9)b |

*Same uppercase letter in same column indicates no significant difference ($p<0.05$).

**Fig. 2**  Comparison of VHN values according to (a) GIC types, (b) treatment protocols, (c) storage medium, (d) storage duration.
DISCUSSION

Present study examined the effect of heat, coating, storage medium and aging on the mechanical behavior and WS of two experimental GICs containing HA or CFA in comparison with a well-known commercial GIC. According to the manufacturer, these experimental GICs should present increased mechanic properties compared to traditional GICs.

In 1984, Yamamoto added HA to conventional GIC to improve the biocompability of GIC31. Later, Yap et al. showed that the addition of HA increased the hardness of GIC and recommended the use of this bioactive cement32. Arita et al. reported that GICs containing 4 wt% HA substitution clearly increased fracture toughness37, whereas Yap et al. indicated that, the addition of the HA powder resulted in a marked deterioration in the compressive fracture strength of GICs31, 37, whereas Yap et al. showed that the incorporation of HA particles resulted in a marked deterioration in the compressive fracture strength and diametral tensile strength following 24 h and 1 week water storage31. In the present study, experimental GICs showed higher FS values than Fuji IX GP. A chemical reaction

Table 5 Water sorption (µg/mm²) was detailed in mean values and standard deviations (SD)

| GICs   | Control (n=60) | Coat (n=60) | Heat (n=60) |
|--------|---------------|-------------|-------------|
|        | Distilled water | Artificial saliva | Distilled water | Artificial saliva | Distilled water | Artificial saliva |
|        | 7 days | 30 days | 7 days | 30 days | 7 days | 30 days | 7 days | 30 days |
| Fuji IX | 36.8 | 83.4 | 24.6 | 57.1 | 54.1 | 80.5 | 44.4 | 83.2 |
| GP     | (25.3)a | (8.1)a | (20.9)a | (44.2)a | (20.1)a | (8.3)a | (18.6)a | (29.2)a |
| Fuji 9ap-sphere | 37.7 | 92.2 | 34.1 | 68.5 | 43.9 | 93.8 | 58.1 | 119.8 |
|        | (12.3)a | (8.6)a | (18.3)a | (12.6)a | (17.8)a | (7.6)a | (22.1)a | (24.5)a |
| Fuji 9ap-CFap | 42.0 | 97.2 | 56.3 | 110.1 | 44.8 | 80.3 | 59.4 | 128.8 |
|        | (10.6)a | (4.0)a | (19.8)a | (23.0)a | (20.0)a | (40.9)a | (19.9)a | (30.9)a |

*Same uppercase letter in same column indicates no significant difference (p<0.05).
between the HA, glass powder and polyacid or the lower dissolution rate of FA may be the cause of increase in the flexural strength. Moreover, this increase in FS can be attributed to the specific properties of HA or CFA of the experimental GIC particles; such as the size of the particles, surface area, porosity of the surface and capacity of crystallinity.

All the tested GICs showed higher VHN values when prepared according to manufacturer’s recommendation but Fuji XP GP showed higher VHN values than experimental GICs which is in accordance with the studies of Woolford[38], Menne-Happ, Ilie[11] and Dehurtevent et al.[39]. Both HA and CFA are involved in the acid-base reaction of GIC and react with inorganic/organic components of the GICs via their phosphate and calcium ions but the mechanical strength of these cements were seemed decreased. This is due to the gradual resorption of HA or FA particles incorporated into the cements after prolonged soaking in distilled water or artificial saliva. Resorption of HA or FA is time dependent and governed by the stability of the particles used. A previous study with a relatively lower crystallinity HA showed a significant decrease in the mechanical properties of GIC after 1 week[40]. It is possible to expect that HA or FA resorption may lead to the degradation of GIC interface, thus deteriorating the mechanical properties[32].

Matrix composition, the size of filler content and distribution, mixing method, and different immersion medium as well as the powder to liquid ratio could affect the water absorption of GICs[32,42]. Storage time and medium can be influential factors on Ws kinetics. Small molecular-sized HA or FA particles can gradually resorb in a time-dependent manner, which can also deteriorate the mechanical properties of GICs after prolonged soaking[32]. Moreover, water molecules can penetrate into the interpolymer chains thereby can cause deterioration of strength[40].

Water absorption levels of GICs tested in this study were within the similar range. Therefore, the effect of HA or CFA addition into the GIC composition could not be interpreted. Similarly, Aliping-Mckenzie et al. reported that the Ws characteristics were found to be unaffected by the nature of the storage medium[41].

Water uptake or loss during the setting reaction would reduce the mechanical properties of GICs to preserve the balance between water uptake and loss[44,45]. It has been shown that the coating increases the FS via maintenance of the water balance and the inhibition of hygroscopic expansion, leading to less dimensional change and can also be beneficial in preventing the formation of surface porosities and cracks[46-48].

Bonifacio et al. investigated the effect of coating on a GIC (Fuji IX Extra) and reported significant difference in FS when coating was used[47]. On the other hand, Zoergiebel and Ilie examined the effect of coating on micro-mechanical properties of different GICs and reported a non-significant improvement in the hardness[48]. In the present study, coating did not improve the FS or VHN values of GICs. This result might be due to the chemical composition of the experimental GICs tested or the storage medium or storage duration of the specimens.

The setting process of GIC is characterized by an interaction between polyacid liquid and a glass powder. The use of heat is supposed to improve the mechanical properties by accelerating the matrix-forming reaction[38]. The heat energy can be supplied by LED, halogen lamp or ultrasonic excitation[11].

Woolford[38], Menne-Happ and Ilie[11] and Dehurtevent et al.[39] reported no-effect on mechanical properties, when materials were heated during setting reaction. On the other hand, Fabián Molina et al. reported that heating the GICs with an LED curing increased the biaxial FS value of GICs[11]. In the present study, heat application through the use of an LED 1,000 mW/cm² curing unit during setting did not show an improvement on the mechanical properties. This may also originate from the chemical formulation of the GICs used. In another study, it was reported that heat application enhances the reaction rate but does not influence the mechanical properties of the GICs[11].

Two different storage media were used in the present study and storage of GICs in artificial saliva showed higher VHN values than storage in water. Artificial saliva used in the experiment was composed of various chemical components simulating oral conditions. The higher VHN values may be due to salivary components, including calcium and phosphate diffusing into GIC structure. Okada et al. also investigated the effects of saliva and distilled water used as storage media and the storage period on the surface hardness of Fuji IX[40]. They reported that Fuji IX did increase in VHN with time at both storage conditions but the increase rate of saliva was higher than distilled water.

In the present study, VHN values of the all GICs were negatively affected from prolonged water storage in correlation with the study of Silva et al.[7]. On the other hand, Ellakuria et al. also examined the effect of storage time on microhardness of GICs and reported that the increase in storage time produced an increase in microhardness[39].

This study has highlighted the possible potential for improving the performance of GIC when using HA or CFA as reinforcement. The addition of HA or CFA in experimental GICs did not show better performances than the conventional GIC in terms of VHN. FS values of experimental GICs were higher than the conventional GIC whereas Ws values were similar for all tested GICs. Thus, the data support partly rejection of the first null hypothesis. Coating or heating did not influence the FS but VHN values of the GICs were affected by heat application. Aging decreased both of the mechanical properties. So, the second null hypothesis was also partly rejected. Storage in artificial saliva or distilled water did not show similar mechanical properties; thus, third null hypothesis was accepted but as this study was generated under laboratory conditions the results were far removed from clinical practice.
CONCLUSIONS

Within the limitation of this in vitro study the following conclusions were drawn:

1. Addition of HA or CFA to GIC may enhance the FS of GIC,
2. Inclusion of HA or CFA in GIC did not seem to improve the micro hardness of tested experimental GICs,
3. WS was not affected by all tested parameters,
4. Coating or heating has no influence on FS of the tested GICs,
5. Microhardness of the GICs was seemed to be affected by heat application,
6. Aging seemed to affect FS and VHN negatively,
7. FS and VHN of the tested GICs could be affected differently from storage media.

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