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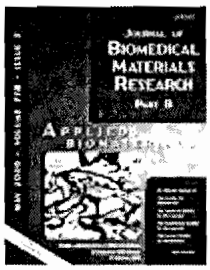

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Environmental degradation of glass-ionomer cements: A depth-sensing microindentation study

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KEYWORDS

mechanical properties ?microindentation ?glass-ionomer

ABSTRACT

This study investigated the effects of various environmental conditions on the hardness and elastic modulus of restorative glass-ionomer cements (GICs). Two resin-modified GICs (RMGICs) (Fuji II LC [FL]; Photac-Fil Quick [PQ]) and three highly viscous GICs (HVGICs) (Fuji IX Fast [FN]; KetacMolar [KM]; KetacMolar Quick [KQ]) were evaluated in this study. Specimens were fabricated according to the manufacturers' instructions and stored under a variety of conditions (n = 7): 100% humidity, distilled water, pH 5 demineralization solution, and pH 7 remineralization solution. The hardness and elastic modulus were measured using a depth-sensing microindentation test after 4 weeks. The results were analyzed using the independent samples T-test and ANOVA/Scheffe's *post hoc* test ($p < 0.05$). HVGICs showed significantly higher hardness and elastic modulus than RMGICs under all storage conditions. Storage in distilled water significantly increased the hardness and elastic modulus of FN, but decreased that of PQ. All HVGICs and RMGICs stored in remineralization solution had hardness values and elastic moduli comparable to those stored in water. Compared to remineralization solution, demineralization solution had no significant effects on the modified GICs with the exception of KQ. The results suggest that the mechanical properties of glass-ionomer restoratives are material-type and storage condition dependent. Therefore, the clinical selection of a glass-ionomer material should be based on the oral environment to which it will be subjected. © 2006 Wiley Periodicals, Inc. J Biomed Mater Res Part B: Appl Biomater, 2006

Received: 10 November 2005; Revised: 9 July 2006; Accepted: 18 July 2006

DIGITAL OBJECT IDENTIFIER (DOI)

10.1002/jbm.b.30697 About DOI

ARTICLE TEXT

INTRODUCTION

Glass-ionomer cement (GIC) and composite resin form the two extremes of the continuum of direct tooth-colored restorative materials. GICs are biocompatible, anticariogenic, and can chemically adhere to tooth structure, properties that are lacking in composite resin.[1] Conventional GICs, however, have inferior strength, aesthetic, and handling properties when compared to composite resin, limiting the use of GICs to deciduous teeth, and nonstress bearing areas of permanent teeth. This has led to a series of modifications to the formulation of conventional GICs.[1] Resin-modified GICs (RMGICs) were developed by incorporating water-soluble resin monomers, while highly viscous GICs (HVGICs) were manufactured by removing excess calcium ions from glass particles, decreasing particle size and increasing the powder to liquid ratio.[2-5] Conventional GICs set via an acid-base reaction between polycarboxylic acids and silicate glass particles. Once set, GICs can be regarded as a composite of polycarboxylate matrix and glass filler particles, in which water is bound to coordination sites of the polycarboxylate matrix and siliceous hydrogel surrounding the glass particles.[6][7] Early water contamination or dehydration during and after the initial setting reaction can result in dissolution or cracking of materials, leading to decreased mechanical properties.[8] Therefore, many manufacturers recommend protection of GICs with a resin coating.[9] As polycarboxylate salt-based materials, GICs may show variable performance in ionic and acidic solutions.[10] Previous studies have

demonstrated that the solubility of GICs increases with a decrease in the pH of the surrounding environment.[11] It is postulated that acids degrade GICs by extracting metal cations from the cement matrix and incorporated glass particles.[12] A commercial HVGIC was however, found to have higher strength with early water exposure.[13] An increase in hardness was also observed when a HVGIC was conditioned in natural saliva.[14] These results suggest that modified GICs may behave differently from conventional GICs under various environmental conditions. From a clinical standpoint, knowledge of the performance of modified GICs under various oral conditions would provide insight to their intraoral application and behavior. To simulate *in vivo* conditions, we examined the performance of various materials after storage under conditions including 100% humidity, water, demineralization (pH 5) and remineralization (pH 7) solutions with levels of calcium and phosphate similar to saliva. The latter two solutions were formulated as recommended by ten Cate and Duijsters.[15]

Hardness and elastic modulus are fundamental mechanical properties of restorative materials. Hardness refers to the plastic deformation of restoratives under occlusal stress, and relates to functional parameters such as resistance to deformation, friction, and abrasion.[16] Hardness is usually measured using an indentation test in which a sharp diamond indenter is pressed into the material. The hardness number is then calculated by dividing peak load over the projected contact area of the indent impression.[16] Elastic modulus is responsible for the integrity of the interface between restoratives and the tooth structure.[17] Because of the brittleness of dental materials, the elastic modulus is commonly measured using the three-point bending method documented in ISO 4049. This method requires a large beam specimen (2 ?2 ?25 mm^3) of high surface quality.[18] Recently, a depth-sensing microindentation technique was used by Yap et al. to obtain hardness and elastic modulus measurements of dental materials in a single test.[19] In the depth-sensing microindentation test, the indentation load (P) and corresponding indenter penetration depth (h) were measured continuously. The hardness and elastic modulus were then calculated from the loading and unloading portions of the P-h curve, respectively.[20] Some of the advantages associated with the microindentation test include ease of specimen preparation, small specimen size, and the ability to perform repeated measurements on the same specimen. Since hardness and elastic modulus can be simultaneously determined in a single test, considerable experiment time and material are saved. In a separate study, Chung et al. found a significant strong and positive correlation between this method and ISO 4049 flexural test results.[21]

The aim of this study was to investigate the influence of various environmental conditions on modified restorative GICs using the depth-sensing microindentation technique. The null hypothesis tested was that hardness and elastic modulus of these modified GIC restoratives are not reduced by water, ions, and acids present in the intraoral environment.

MATERIALS AND METHODS

Representative GIC restoratives including two RMGICs (Fuji II LC [FL]; Photac-Fil Quick [PQ]) and three HVGICs (Fuji IX Fast [FN], KetacMolar [KM]; KetacMolar Quick [KQ]) were investigated. The material profiles are listed in Table I. All materials were in capsule form and prepared according to the manufacturers' instructions. The mixed materials were injected into the square recesses (3 mm long ?3 mm wide ?2 mm deep) of custom-fabricated acrylic molds and covered with acetate strips (Hawe-Neos Dental, Bioggio, Switerzland). A glass slide was placed over the acetate strip and pressed to extrude excess materials. The light-cured materials (FL and PQ) were then polymerized using a Spectrum light unit (Dentsply/Caulk, Delaware, US) with mean intensity greater than 400 mW/cm^2. All specimens were subjected to 37℃ and 100% humidity for 1 h before being placed under the various storage conditions. This protocol is commonly used for *in vitro* studies on GICs and has been reported to give GIC samples optimal properties.[22] Twenty-eight specimens of each material were randomly divided into four groups (n = 7) and stored for 4 weeks at 37℃ under one of the following conditions: (a) 100% humidity, (b) distilled water, (c) remineralization solution (1.5 m*M* $CaCl_2$, 0.9 m*M* KH_2PO_4, 20 m*M* HEPES, 150 m*M* KCl, pH 7), or (d) demineralization solution (1.5 m*M* $CaCl_2$, 0.9 m*M* KH_2PO_4, 50 m*M* acetic acid, 150 m*M* KCl, pH 5).

Table I. Technical Profiles of Materials Evaluated in Present Study

Material	Classification	Manufacturer	Shade	Curing Time	Compositions
Fuji II LC (FL)	RMGIC	GC, Tokyo, Japan	A2	Light cured (20 s)	Powder: AFS-glass, PAA. Liquid: PAA, HEMA, TEGDMA, TMHMD, water
Photac-Fil Quick (PQ)	RMGIC	3M-ESPE, Seefeld, Germany	A2	Light cured (20 s)	Powder: CAFS-glass, PAMA Liquid: PAMA, HEMA, water
Ketac-Molar Quick (KQ)	HVGIC	3M-ESPE, Seefeld, Germany	A2	Auto cured (3 min 30 s)	Powder: CAFS-glass, PAMA. Liquid: PAMA, TA, water
Ketac-Molar (KM)	HVGIC	3M-ESPE, Seefeld, Germany	A1	Auto cured (4 min 30 s)	Powder: CAFS-glass, PAMA. Liquid: PAMA, TA, water
Fuji IX Fast (FN)	HVGIC	GC, Tokyo, Japan	A2	Auto cured (3 min)	Powder: AFS-glass, PAA. Liquid: PAA, TA, water

AFS-glass, alumino fluoro silicate glass; CAFS-glass, calcium alumino fluoro silicate glass; PAA, poly(acrylic acid); PAMA, copolymer of acrylic and maleic acid; TA, tartaric acid; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; TMHMD, 2,2,4-trimethyl hexamethylene dicarbonate.

All specimens were subsequently subjected to a depth-sensing microindentation test using an Instron Microtester system (Model 5848 Instron, MA) fitted with a custom-designed indentation head unit and a digital imaging system. In this study, a four-sided diamond Vickers indenter was used and specimens were indented at a rate of 0.0005 mm/s until a maximum load of 10 N was attained. The peak load was then held for 10 s and unloaded fully at 0.0002 mm/s. The indentation load (P) and corresponding indenter displacement (h) were recorded continuously during the loading-unloading cycle. The hardness and elastic modulus were derived using software developed in-house (PhCalculator Version 1.0). The hardness (H) was determined by dividing the peak load over the maximum contact area, while the elastic modulus (E_{in}) was calculated using an unloading contact stiffness analysis according to the method of Oliver &

Pharr. The equations used are as follows:

$$H = \frac{P_{\max}}{A_{\max}} \quad (1)$$

$$E_{in} = \frac{1 - \nu_{in}^2}{\left(1.142\frac{\sqrt{A_{max}}}{S} - \frac{(1-\nu_0^2)}{E_0}\right)} \quad (2)$$

$$A_{\max} = 24.56\left(h_{\max} - \frac{3P_{\max}}{4S}\right)^2 \quad (3)$$

where P_{max} is the maximum indentation load, A_{max} is the maximum projected contact area, h_{max} the indentation depth at maximum load, E_0 the elastic modulus of the indenter, v_0 the Poisson ratio of the indenter, v_{in} the Poisson ratio of the indented material of interests, and S is the unloading contact stiffness.

A digital imaging system was used to capture images of the indent impressions for later comparison. This system consisted of a custom-designed optical transmission unit (Moritex, Tokyo, Japan), an illumination source (MHF-M1002, Moritex), a microscope digital camera system (DP12, Olympus Optical, Tokyo, Japan) and image analysis software (Micro Image 4.0, Media Cybernetics).

All statistical analysis was carried out at a significance level of 0.05. Comparisons between RMGICs and HVGICs from each storage condition were performed using the independent sample T-test. One-way ANOVA and Scheffe's *post hoc* tests were used to compare the effects of the various storage media on the hardness and elastic modulus of each material.

RESULTS

Results for the mean hardness and elastic modulus of modified GICs stored under different environmental conditions are presented in Figures 1 and 2. The mean hardness ranged from 44.0 to 44.2 HV (RMGICs) and 62.1 to 98.2 HV (HVGICs). The mean elastic modulus ranged from 9.0 to 10.8 GPa and 14.4 to 19.3 GPa for RMGICs and HVGICs, respectively. HVGICs (KM, KQ, and FN) showed significantly higher hardness values and elastic moduli than RMGICs (FL and PQ) ($p < 0.01$) under all storage conditions. With regard to the effect of environmental conditions, as shown in Figures 1 and 2, storage in water significantly lowered the hardness and elastic modulus of PQ ($p < 0.05$), but significantly increased the hardness and elastic modulus of FN ($p < 0.05$). All modified GICs stored in remineralization solution showed comparable hardness and elastic modulus with their counterparts stored in water. With the exception of KQ, all modified GICs stored in demineralization solution showed no significant difference from their paired samples stored in remineralization solution ($p < 0.05$).

Figure 1. Hardness (HV) of modified GICs. RMGICs: FL and PQ; HVGIVs: KM, KQ, and FN. Vertical lines stand for standard deviations. (a,b) Same letter indicates no significant differences in hardness between environmental conditions for each material. Results of one-way ANOVA/Scheff's *post hoc* test ($p < 0.05$).
[Normal View 23K | Magnified View 81K]

Figure 2. Elastic modulus (GPa) of modified GICs. RMGICs: FL and PQ; HVGIVs: KM, KQ, and FN. Vertical lines stand for standard deviations. (a,b) Same letter indicates no significant differences in elastic modulus between environmental conditions for each material. Results of one-way ANOVA/Scheff's *post hoc* test ($p < 0.05$).
[Normal View 24K | Magnified View 86K]

Typical indent impressions of modified GICs stored in water are presented in Figure 3, clearly illustrating the phenomenon of "sink in" and indistinct indentation contour exhibited by some GICs.

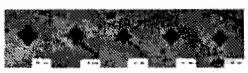

Figure 3. Indent impressions of modified GICs in water (magnification ?0). The arrows indicate undistinguished boundary (B) and sink-in indentation shape (S).
[Normal View 28K | Magnified View 93K]

DISCUSSION

From a method point of view, determination of the contact area at maximum load is critical for the microindentation test. With the nondepth-sensing microindentation method, the hardness is commonly derived from measurements of the indentation diagonal. In this study, the indent images of some GICs were blurred and showed an indistinct boundary. This made it difficult to identify the diagonal and precisely obtain a hardness value. Another factor influencing calculation of hardness is the "pile-up" or "sink-in" effect. During indentation tests, materials around the contact area may deform upwards (pile-up) or downwards (sink-in) with respect to the original surface plane. Sink-in decreases and pile-up increases the contact area, thus the true contact area may be over- or under-estimated. The Oliver & Pharr method works for hard materials which predominantly show "sink-in," but fails with materials that display extensive "pile-up."[20] In this study, "sink in" was clearly visible in the indentation images of modified GICs. This observation supported use of the Oliver & Pharr method for calculating the indenter contact area, avoiding measurement error through consideration of the "pile-up" and "sink-in" effects.

In the present study, HVGICs showed greater hardness and elastic modulus than RMGICs regardless of storage conditions. This was in agreement with the results of a previous one-year study of GICs.[23] The improved hardness and elastic modulus of HVGICs may be accounted for as follows: Firstly, excess calcium ions in HVGICs are removed from the surface of glass particles in order to form more insoluble aluminum polycarboxylate at early setting times.[4][5] The insoluble aluminum salts can reduce the loss of matrix-forming ions under aqueous conditions and improve the mechanical properties of HVGICs.[24] In contrast, RMGICs consist of a polymer and polycarboxylate matrix network. The polymer matrix of HEMA (2-hydroxyethyl methacrylate) in RMGICs may retard H^+ mobility, inhibit further acid-base reaction and therefore discourage complete formation of a polycarboxylate matrix.[3] Secondly, a larger glass particle size and higher powder to liquid ratio may contribute to the superior mechanical properties of HVGICs.[5] It has been shown that a dense surface texture with tightly packed glass particles may result in higher surface hardness for GICs.[25] Thirdly, in RMGICs, HEMA polymer entangled with polycarboxylate matrix may lead to an inferior interface between glass particles and matrices.[26] Possible phase separation can occur, leading to degradation of the composite structure and inferior mechanical properties.

With regard to the effect of intraoral conditions, the previously stated null hypothesis was partially rejected. Results showed that the influence of various environmental conditions on modified restorative GICs was dependent on the material type. In this study, HVGICs presented higher or comparable hardness and elastic modulus in water when compared to 100% humidity. This indicated that an aqueous environment is critical for retaining the properties of set HVGICs, which mainly consist of polycarboxylate matrix. However, RMGICs showed decreased mechanical properties when exposed to water. Water absorption of hydrophilic HEMA in matrices may cause plasticization of RMGICs and be responsible for a reduction in hardness and elastic modulus.[27][28] These results suggest that HVGICs may not require resin protection after initial set and should be prevented from dehydration, while RMGICs should be sealed with a resin after placement.

In neutral ionic remineralization solution and water, similar hardness values and elastic moduli were observed for each modified GIC. Results indicated that the modified GIC restoratives are durable in a neutral ionic environment, which is representative of the common intraoral environment. Regarding acidic conditions (pH 5 demineralization solution), RMGICs showed comparable hardness and elastic modulus when compared with neutral remineralization solution. This may be due to incorporation of HEMA resin into RMGICs. HEMA resin may obstruct movement of H^+ and thus improve the resistance to acid attack.[3] For HVGICs, demineralization solution had no effects on FN and KM, but reduced the hardness of KQ. Previous studies have shown that the aesthetics and radiopacity of FN is improved when calcium is replaced by strontium. The strontium salts of polycarboxylic acids are thought to be more stable than the calcium salts.[29] In KQ, copolymers of acrylic and maleic acids are used. The resultant matrix derived from maleic acid may be more susceptible to attack by acids.[30] The disparity in performance between KM and its fast set version KQ, however, is not clear and warrants further investigation. Clinically, the composition of the GIC as well as the oral condition of the patient should be considered when a GIC filling treatment is suggested.

CONCLUSIONS

The effect of various environmental conditions on the hardness and elastic modulus of modified glass-ionomer restoratives was examined using a depth-sensing microindentation approach. HVGICs were significantly harder and stiffer than RMGICs. When exposed to water, the hardness and elastic modulus of HVGICs increased or remained the same. In contrast, RMGICs showed comparable or decreased hardness and elastic modulus in water. Neutral ionic storage conditions did not affect surface mechanical properties of any of the modified GICs. With the exception of KQ, acidic conditions (pH 5) had no significant effects on the GICs evaluated. Results suggest that the performance of modified GICs is material-type and environmental condition dependent. Preventing HVGIC restoratives from dehydration and protecting RMGIC restoratives with a waterproof coating after initial setting is recommended. Clinical selection of GICs should be based on the oral environment to which they will be subjected.

Acknowledgements

We would like to thank GC and 3M-ESPE for the donation of materials used in this study.

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