

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.e-jds.com



Original Article

Flexural strengths and porosities of coated or uncoated, high powder-liquid and resinmodified glass ionomer cements



Nathamon Thongbai-on^a, Danuchit Banomyong^{b*}

^a Research Unit, Faculty of Dentistry, Mahidol University, Bangkok, Thailand ^b Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahido

^b Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahidol University, Bangkok, Thailand

Received 16 October 2019; Final revision received 3 February 2020 Available online 16 March 2020

KEYWORDS Flexural strength; Glass ionomer cement; Micro-computed tomography; Porosity; Resin coating	Abstract Background/purpose: No study has previously investigated and compared whether resin coating could prevent the effect of dehydration on flexural strengths and porosities of high powder-liquid and resin-modified glass ionomer cements (HPL-GIC and RM-GIC). The purpose of this study is to investigate the effect of resin coating on flexural strengths and porosities of HPL-GIC and RM-GIC under a dry condition. <i>Materials and methods</i> : HPL-GIC (<i>Equia Forte Fil</i>) or RM-GIC (<i>Fuji II LC</i>) was mixed and loaded into a mold to create a bar-shaped specimen, n = 12 of each. The specimens were randomly divided into two groups, <i>coated</i> and <i>uncoated</i> , n = 6 f each. In the <i>coated</i> group, a resin coating agent (<i>Equia Forte Coat</i>) was applied and light cured for 20 s. After 72 h, each specimen was dried and scanned to detect porosities (% volume) using micro-computed tomography. After scanning, flexural strength (MPa) of the specimen was tested using a three-point bending method. <i>Results</i> : Porosities of HPL-GIC were significantly higher than RM-GIC, either <i>coated or uncoated</i> group ($p < .05$). Flexural strengths of <i>coated</i> and <i>uncoated</i> HPL-GIC were 41.47 ± 0.89 and 15.32 ± 1.15 MPa that were significantly lower than those of RM-GIC at 104.77 ± 3.97 and 52.90 ± 2.17 MPa ($p < .05$). Flexural strengths of GICs under dry condition. HPL-GIC had higher porosities and lower flexural strength than RM-GIC. © 2020 Association for Dental Sciences of the Republic of China. Publishing services by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).
---	--

* Corresponding author. Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahidol University, 6 Yothi Road, Rajthawee, Bangkok, Thailand. Fax: +66 2 200 7824.

E-mail addresses: danuchit.ban@mahidol.ac.th, danuchitb@gmail.com (D. Banomyong).

https://doi.org/10.1016/j.jds.2020.02.004

1991-7902/© 2020 Association for Dental Sciences of the Republic of China. Publishing services by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

Introduction

Glass ionomer cement (GIC) is a water-based restorative material, composing of basic glass powder and polyacid liquid and setting by an acid—base reaction.¹ Currently, GIC is classified into two main categories- high powder-liquid (HPL) and resin-modified (RM) GIC.² HPL or highly viscous GIC is modified from the conventional material by increasing the powder-liquid mixing ratio to accelerate setting reaction and also improve material's properties.² RM-GIC is modified by adding polymerizable monomers into the liquid and/or powder component, which the polymerization reaction forms the polymer networks that enhance the properties.²

GIC is sensitive to water imbalance during initial setting.¹ To prevent water gain or loss, thin-layer coating on GIC with a protective agent is recommended to improve strength^{3,4} and reduce clinical wear.⁵ Resin coating for GIC has been recently introduced to replace the varnish or petroleum jelly. At long term, GIC is still sensitive to water loss even 6-12 months after placement.⁶ Rubber dam isolation is commonly placed during adhesive and restorative procedure. GIC in the isolation area becomes dehydrated, which induces micro-crack within the material and disintegrates bond to dentin.^{6,7} Coating GIC before rubber dam isolation might protect the material from this drying effect.⁸

Effect of surface coating on the properties of GIC tends to be different between HPL-GIC and RM-GIC.⁹ Formation of polymerized resin networks protects RM-GIC from water gain and loss, which makes the material less sensitive to water sorption or dehydration than HPL-GIC.^{3,4} For this reason, surface coating may be not absolutely mandatory for RM-GIC, and the benefit is still controversial.¹⁰

GIC is a two-component material that requires mixing before use. Porosities are detected in the mixed GIC by the air is trapped inside the material during mixing. The higher viscosity of GIC, the more internal porosities are detected.¹¹ The strength of restorative GIC has an invert relationship to the amount of porosities.^{12,13} HPL-GIC, either hand- or machine-mixed, contains similar amount of porosities.¹³ In contrast, hand-mixed RM-GIC contains higher porosities than the machine-mixed material.¹² Nevertheless, no previous study directly compared between the two categories of GIC in term of porosities. In addition, the porosities of dried GIC may be altered due to water loss. All of previous studies

investigated the porosities of GIC in the humid condition. $^{11-13} \ \ \,$

Micro-computed tomography (micro-CT) is a nondestructive tool that comprehensively investigate three dimensional (3-D) details of the scanned objects.¹⁴ Recent micro-CT tool is able to detect micro gaps and voids with the smallest effective voxel size of approximately 6 microns.¹⁵ From our pilot study, we found that micro-CT could create a dry condition in the chamber during 3-D scanning. In addition, the dry condition was standardized if the scanning time was controlled.

Most of recent studies investigated the effect of coating agent on the properties of GIC in the storage conditions without dehydration.^{3,4,16} No study has previously investigated and compared whether coating could prevent the negative effect of dehydration on the strength and porosities of HPL-GIC and RM-GIC. Therefore, the objective of this study was to investigate flexural strengths and porosities of resin-coated or uncoated, HPL-GIC and RM-GIC under the simulated dry condition.

Materials and methods

HPL-GIC (Equia Forte Fil, GC, Tokyo, Japan) and RM-GIC (Fuji II LC, GC, Tokyo, Japan) were tested in this study, which the details are present in Table 1. The encapsulated material was mixed using a triturator for 10s according to the manufacturer's instruction and loaded into a stainless steel mold size $25 \times 2x2$ mm covering with two glass slides to create a bar-shaped specimen, n = 12 of each material. The specimen of HPL-GIC was left for 5 min to ensure the initial set from the acid-base reaction. The specimen of RM-GIC was light cured from the top site using an LED lightcuring unit (Demi Plus, Kavo-Kerr, Brea, CA, USA) with the 8-mm light guide for 20 s at each area. The set material was kept in a water bath at 37 ± 1 °C for 15 min and then removed from the mold. The specimens were measured and polished with 400-grit silicon carbide paper to obtain the length 25 ± 0.2 mm and the width/thickness 2 ± 0.1 mm. The twelve prepared specimens of each material were randomly divided into two groups-coated and uncoated, n = 6 of each. In the *coated* group, a resin-based coating agent (Equia Forte Coat, GC, Tokyo, Japan) was applied on the surfaces of specimen using a micro-brush and light

and a resin coating agent.					
Materials	Manufacturer	Main compositions	Lot numbers		
Equia Forte Fil (high powder-liquid)	GC corp., Tokyo, Japan	Powder: strontium fluoroalumino-silicate glass, polyacrylic acid powder Liquid: polyacrylic acid, polycarboxylic acid, tartaric acid	1611153 and 1701241		
Fuji II LC (resin-modified)		Powder: fluoroalumino-silicate glass, polyacrylic acid powder Liquid: polyacrylic acid, water, HEMA	1704052 and 1510031		
Equia Forte Coat (resin coating)		Methyl methacrylate, camphorquinone	1502061		

 Table 1
 Manufacturer, main compositions and lot numbers of high powder-liquid and resin-modified glass ionomer cements, and a resin coating agent.

cured for 20 s at each area. In the *uncoated* group, no coating was applied, but the specimen of RM-GIC was also light cured for 20 s at each area to standardize the light-curing time. All specimens were kept in an incubator at 37 ± 1 °C and 100% humidity for 72 h to allow maturation of GIC before testing.

Each specimen was air dried and scanned to observe any internal porosities of the material using micro-computed tomography (micro-CT, SkyScan 1173, Bruker, Kontich, Belgium) set at 80 kV, 100 μ A. The scanning concurrently created the dry condition to GIC for an approximate period of 20 min. The temperature in the operating room was set at $25 \pm 2 \,^{\circ}$ C with a humidity-controlled air conditioner. The 3-D images were reconstructed and analyzed using CT analyzer software (CTAn 1.16; Bruker). Volumes of the materials and porosities were identified using the CT volume software (CTVol 2.3.2.0; Bruker). Porosities were calculated and reported as percentages of the total material volume.

After micro-CT scanning, flexural strength testing using a three-point bending method was performed according to the ISO 4049-2009. A static load was applied at the center using a 2-mm diameter cylindrical tip at a rate of 1 mm/s until the specimen was fractured, and the force (N) was recorded. Flexural strength (MPa) was calculated according to the formula- $3Fl/2wh^2$, while *F* is the force until fracture, *l* is the length, *w* is the width, and *h* is the height of specimen. For statistical analysis, unpaired t-test was used to compare the flexural strengths and porosities between the *uncoated and coated* groups, as well as between HPL-GIC and RM-GIC.

Results

Flexural strengths of *uncoated* and *coated* HPL-GIC were 15.32 ± 1.15 and 41.47 ± 0.89 MPa while those of RM-GIC were 52.90 ± 2.17 and 104.77 ± 3.97 MPa (Table 2). Flexural strengths of *coated* HPL-GIC or RM-GIC were significantly higher than those of the *uncoated* materials (p < .05). Flexural strength of RM-GIC was significantly higher than HPL-GIC, either *uncoated* or *coated* (p < .05).

The amount of porosities of HPL-GIC were significantly higher than RM-GIC (p < .05), either in *coated* ($0.20 \pm 0.10\%$ vs. $0.07 \pm 0.03\%$) or *uncoated* group ($0.18 \pm 0.02\%$ vs. $0.05 \pm 0.02\%$) (Table 3). The porosities were not significantly different between the *coated and uncoated* groups ($p \ge .05$).

The representative micro-CT images of *uncoated* and *coated* GIC materials are present in Fig. 1. The specimens of HPL-GIC showed many porosities distributed within the material. In contrast, the specimens of RM-GIC only contained

Table 2Flexural strengths (MPa) of the coated and uncoated specimens of high powder-liquid and resin-modified glass ionomer cements.

Flexural strengths (MPa)	Uncoated	Coated
Equia Forte Fil	15.32 ± 1.15^{a}	41.47 ± 0.89^{b}
	52.90 ± 2.17	104.77 ± 3.97

The superscripts with different *small* letters indicate a significant difference in flexural strength between the groups.

Table 3Porosities (% volume) of the coated and uncoatedspecimens of high powder-liquid and resin-modified glassionomer cements.

Porosities (% volume)	Uncoated	Coated
Equia Forte Fil Fuji II LC	$\begin{array}{c} 0.18 \pm 0.02^{a} \\ 0.05 \pm 0.02^{b} \end{array}$	$0.20 \pm 0.10^a \\ 0.07 \pm 0.03^b$

The superscripts with different *small* letters indicate a significant difference in % porosities between the groups.

few porosities within the material. The patterns of porosities were similar between the *uncoated* and *coated* specimens.

Discussion

Under the simulated dry condition, the flexural strength of GICs coated with a resin coating agent were significantly higher than the uncoated cements. GIC is a water-based material that is sensitive to water loss, so the properties of the cements are negatively affected in the dry condition.^{1,2} Coating on GIC prevents water loss⁸ and, as a result, increases the flexural strength in this study.

When comparison between HPL-GIC (*Equia Forte Fil*) and RM-GIC (*Fuji II LC*), the flexural strength of the former was much significantly lower than the latter, regardless of coating or non-coating. Current HPL-GIC is clinically set within 5 min, yet the acid—base reaction still progresses until the maturation stage.¹⁷ HPL-GIC at early stage is partially mature with limited strength, as reported in our study. Hence, HPL-GIC strictly requires protection from resin coating after restoration placement¹⁷ or whenever water imbalance is expected on the restoration, such as after rubber dam isolation.

In addition to the polyacid salt matrix formation in HPL-GIC, the polymerized resin networks in RM-GIC protect the material from water gain and loss,¹ and also improved the strength of the material.² Our results also showed the strength of RM-GIC was much higher than HPL-GIC, regardless of coating. However, coating RM-GIC is still required since the coating significantly improved the strength of RM-GIC under the dry condition, as reported in our study. The importance of coating to RM-GIC is confirmed in the other studies that investigated the effect on gap formation⁶ and fracture toughness.¹⁷

In our study, the porosities of GICs were not significantly affected by coating. Coating could prevent water loss from GICs under the dry condition, but it had no effect on the formation of porosities. However, we found that the amount of porosities of HPL-GIC was significantly higher than RM-GIC. The result is in correspondence to the study that investigated gaps and voids of HPL-GIC and RM-GIC when used as a base in restoration of endodontically treated teeth.¹⁵ This phenomenon might be explained by the difference in viscosity between the two materials. From the specimen preparation, the author noticed that the viscosity of the HPL-GIC was relatively higher than the RM-GIC, which might cause more voids or air bubbles inside the material during mixing.¹¹ Furthermore, the higher amount of porosities in HPL-GIC might also take a part in the lower flexural strength when compared to RM-GIC.¹



Figure 1 A–D Micro-computed tomography images of uncoated (A, C) and coated (B, D) glass ionomer cements (grey) with internal porosities (colored with yellow, blue, purple or orange in each specimen). HPL-GIC (*Equia Forte Fil*) (A, B) showed the higher amounts of internal porosities than RM-GIC (*Fuji II LC*) (C, D), regardless of non-coating or coating specimens. The amounts of porosities in the uncoated and coated GICs were similar.

In conclusion, coating with a resin-based agent significantly increased the flexural strength of either HPL-GIC (*Equia Forte Fil*) or RM-GIC (*Fuji II LC*) under the dry condition. RM-GIC had a significantly higher flexural strength than HPL-GIC, regardless of coated or uncoated. The porosities of HPL-GIC were significantly higher than RM-GIC. On the contrary, the porosities of coated and uncoated GICs were not significantly different.

Declaration of Competing Interest

The authors deny any conflict of interest.

Acknowledgements

None.

References

- 1. Sidhu SK, Nicholson JW. A review of glass-ionomer cements for clinical dentistry. *J Funct Biomater* 2016;7:e16.
- Tyas MJ, Burrow MF. Adhesive restorative materials: a review. Aust Dent J 2004;49:112–21.
- Bagheri R, Palamara J, Mese A, Manton DJ. Effect of a selfadhesive coating on the load-bearing capacity of toothcoloured restorative materials. *Aust Dent J* 2017;62:71–8.
- Bagheri R, Taha NA, Azar MR, Burrow MF. Effect of G-Coat Plus on the mechanical properties of glass-ionomer cements. *Aust Dent J* 2013;58:448–53.
- Diem VT, Tyas MJ, Ngo HC, Phuong LH, Khanh ND. The effect of a nano-filled resin coating on the 3-year clinical performance of a conventional high-viscosity glass-ionomer cement. *Clin Oral Invest* 2014;18:753–9.

- Sidhu SK, Sherriff M, Watson TF. The effects of maturity and dehydration shrinkage on resin-modified glass-ionomer restorations. J Dent Res 1997;76:1495–501.
- Sidhu SK, Pilecki P, Sherriff M, Watson TF. Crack closure on rehydration of glass-ionomer materials. *Eur J Oral Sci* 2004; 112:465–9.
- Nicholson JW, Czarnecka B. Kinetic studies of the effect of varnish on water loss by glass-ionomer cements. *Dent Mater* 2007;23:1549–52.
- 9. Percq A, Dubois D, Nicholson JW. Water transport in resimmodified glass-ionomer dental cement. *J Biomater Appl* 2008;23:263-73.
- Wang XY, Yap AU, Ngo HC. Effect of early water exposure on the strength of glass ionomer restoratives. *Operat Dent* 2006; 31:584–9.
- **11.** Fleming GJ, Farooq AA, Barralet JE. Influence of powder/liquid mixing ratio on the performance of a restorative glass-ionomer dental cement. *Biomaterials* 2003;24:4173–9.
- **12.** Covey DA, Ewoldsen NO. Porosity in manually and machine mixed resin-modified glass ionomer cements. *Operat Dent* 2001;26:617–23.
- Nomoto R, Komoriyama M, McCabe JF, Hirano S. Effect of mixing method on the porosity of encapsulated glass ionomer cement. *Dent Mater* 2004;20:972–8.
- 14. Swain MV, Xue J. State of the art of Micro-CT applications in dental research. *Int J Oral Sci* 2009;1:177–88.
- 15. Thongbai-On N, Chotvorrarak K, Banomyong D, Burrow MF, Osiri S, Pattaravisitsate N. Fracture resistance, gap and void formation in root-filled mandibular molars restored with bulkfill resin composites and glass-ionomer cement base. J Investig Clin Dent 2019;10:e12435.
- Leirskar J, Nordbo H, Mount GJ, Ngo H. The influence of resin coating on the shear punch strength of a high strength autocure glass ionomer. *Dent Mater* 2003;19:87–91.
- Alvanforoush N, Wong R, Burrow M, Palamara J. Fracture toughness of glass ionomers measured with two different methods. J Mech Behav Biomed Mater 2019;90:208–16.