

## Effect of different surface protection materials on microhardness of a resin-modified glass-ionomer cement

Awiruth Klaisiri<sup>1</sup>, Nantawan Krajangta<sup>1</sup>, Lili Yang<sup>1</sup>

### **Abstract**

**Objective:** *The purpose of this study was to measure the microhardness of resin-modified glass-ionomer cement after applying different protective materials for surface protection.*

**Materials and methods:** *Sixty specimens of resin-modified glass-ionomer cement were prepared in stainless steel mold; 6 mm of diameter and 6 mm of height. The specimens were divided into six groups according to the surface coating methods; 1. Non-coat (NC) 2. Fuji varnish (FV) 3. Equia coat (EC) 4. Adper scotchbond multi-purpose adhesive (SM) 5. Adper single bond 2 (SB) and 6. Single bond universal (SU). The specimens were coated with different agents in each group. After initial setting for 10 minutes, all specimens were stored in distilled water at 37°C for 24 hours. The specimens were polished with the polishing machine for 20 seconds at room temperature, and then the surface microhardness was measured by FM-800. The measurements were statistically analyzed by one way ANOVA and Tamhane's post-hoc test.*

**Results:** *The microhardness of coated groups were significantly higher than non-coated group. In the coated group, microhardness of FV, EC and SM were significantly higher than SB and SU.*

**Conclusion:** *The microhardness of resin-modified glass-ionomer cement of all coated groups were significant higher than non-coated group. The best surface protection was observed in Fuji varnish, Equia coat and Adper scotchbond multi-purpose adhesive.*

**Key words:** *Microhardness; Resin-modified glass-ionomer cement; Surface protection material*

<sup>1</sup> Division of Operative Dentistry, Faculty of Dentistry, Thammasat University, Pathumthani

## Introduction

Modification of glass-ionomer cement (GIC) by addition of small quantities of light-polymerizable resin group has been proven to be a successful strategy for water sensitivity reduction. It related to the improvement of physical and mechanical properties of GIC but still retain the advantages of conventional GIC in aspect of ion exchange, adhesion to conditioned enamel and dentin, fluoride release, low interfacial shrinkage stress, improved resistance to microleakage, on-command hardening and immediate finishing as with resin composite, translucency<sup>1,2</sup>.

Resin-modified glass-ionomer cement (RM-GIC) has been developed by addition of small quantities of light-polymerizable resin groups (2-hydroxyethylmethacrylate, or HEMA) within acidic liquid molecules<sup>3</sup>. The result contains complex structure from both newly acquired light polymerized reaction and traditional acid-base setting reaction of conventional GIC<sup>2</sup>. When the resin part is polymerized making strength to the material, it protects ongoing acid/base reaction from dehydration and water sorption. Even though the acquired resin part work, water still play a role for maturation of RM-GIC; water dehydration during the initial setting stages can compromise the physical properties of the restoration<sup>4</sup>. In spite of the resistance to water movement in and out of the restoration, post-finishing sealing of a RM-GIC restoration with

light-polymerized unfilled resin and other coating agents are recommended to protect acid/base components at the restoration's outer surface such as varnish, nail varnish, petroleum jelly, coco butter and nanofilled resin<sup>5-9</sup>.

The surface hardness may be defined as the resistance of a material surface to wear and related to the polymerization of resin-base restorative materials. The surface hardness is important parameter in evaluating dental material, especially restorative materials. Vickers' hardness (VHN) tester is very useful in surface hardness of dental materials such as GIC<sup>10</sup> and resin composite<sup>11</sup>

At the present stage, there are few studies of RM-GIC surface protection, and many limitation in research about surface protection by adhesive resin such as one-step self-etch adhesive and universal adhesive. Meanwhile, the universal adhesive is widely used in the dental clinic, but there still has been no study to evaluate the universal adhesive as the surface protection of RM-GIC.

Therefore, the aim of the present study was to measure the microhardness of RM-GIC after applying different protective materials for surface protection. The influence of Fuji vanish, Equia coat, Adper scotchbond multi-purpose adhesive (3-step etch and rinse), Adper single bond 2 (2-step etch and rinse) and Single bond universal (one-step self-etch, universal adhesive) coated in RM-GIC surface and immersed in distilled water are evaluated.

## Materials and methods

The materials used in this study are shown in Table 1

**Table 1: Materials used in this study**

Materials	Compositions	Manufacturer's recommendation	
<b>Fuji II LC (capsule)</b> Lot No. 1505141 (GC Corporation, Tokyo, Japan)	Powder: aluminosilicate glass Liquid: polyacrylic acid; HEMA; 2,2,4, TMHEDC; TEGDMA	Mechanical mix by amalgamator for 10 seconds at high speed. Light cure for 40 seconds.	strip (3M ESPE, St. Paul,
<b>Fuji varnish</b> Lot No. 1310281 (GC Corporation, Tokyo, Japan)	Isopropylacetate, acetone Copolymer of vinyl chloride & vinyl acetate	Apply and dry by gently blowing with air syringe. Maintain moisture isolation for 2-3 minutes.	
<b>Equia coat</b> Lot No. 1502061 (GC Corporation, Tokyo, Japan)	Urethane methacrylate, Methyl methacrylate, camphorquinone, nanofiller	Apply to the surfaces to be coated and light cure for 20 seconds.	
<b>Adper scotchbond multi-purpose adhesive</b> Lot No. N629415 (3M ESPE, Deutschland GmbH, Neuss, Germany)	Bis-GMA, HEMA, peroxide component of catalyst resin, amine	Apply adhesive and light cure for 20 seconds.	
<b>Adper single bond 2</b> Lot No. N613918 (3M ESPE, St. Paul, Minnesota, USA)	Silica nanofiller, Bis-GMA, HEMA, dimethacrylates, ethanol, water	Apply adhesive and light cure for 20 seconds.	
<b>Single bond universal</b> Lot No. 555323 (3M ESPE, Deutschland GmbH, Neuss, Germany)	MDP phosphate monomer, Dimethacrylate resin, Vitrebond copolymer, HEMA, filler, water, ethanol, initiators, silane	Apply adhesive and light cure for 20 seconds.	

Sixty specimens of RM-GIC [Fuji II LC (capsule), GC Corporation, Tokyo, Japan] were prepared from stainless steel mold (6 mm of diameter and 6 mm of height) following ISO 4049<sup>12</sup>. RM-GIC was automatically mixed encapsulated cements. The molds were filled with RM-GIC, covered with celluloid matrix

Minnesota, USA), and followed by a glass slide. To press this set against the top portion of the mold, a 200 gram weight was placed on top of the set. RM-GIC was light cured at a **light** intensity of 1000 **mW/cm<sup>2</sup>** for 40 seconds on each side (Elipar Freelight 2 LED curing light, 3M ESPE, Minnesota, USA). After setting,

glass slide and celluloid matrix strip were removed. Specimens with voids and uneven rough surface were excluded from the study. The specimens were randomly assigned into 6 groups, 10 specimens each: group1 non-coat (NC), group2 Fuji varnish (FV), group3 Equia coat (EC), group4 Adper scotchbond multi-purpose adhesive (SM), group5 Adper single bond 2 (SB), group6 Single bond universal (SU).

Coating agents were applied on all surface of the specimens in each group according to the manufacturer's recommendation (Table 1). The specimens were immersed in distilled water and stored at 37°C for 24 hours, after that the coatings were removed from the specimens by wet-ground with 1200-grit silicon carbide paper (3M Wetordry abrasive sheet, 3M, Minnesota, USA) on a polishing machine (Nano 2000 grinder-polisher with a FEMTO 1000 polishing head, Pace Technologies, Arizona, USA) for 20 seconds and 600 round per minute at room temperature, to obtain a flat polished surface and without any of the surface protection material. Then, the surface microhardness of the specimens were tested by microhardness

tester (FM-800, Future Tech corp., Kawasaki, Japan). Microhardness indentations were made on the top of specimen surface. The Vickers' microhardness test was performed using a diamond indenter with 100 gram load and 15 seconds dwell time<sup>13</sup> (x40 magnification). Three measurements were accomplished on the top in each specimens, with a 1 mm distance between indentations, and the mean were calculated.

The data were statistically analyzed using one way ANOVA and Tamhane's post-hoc test to determine significant statistical differences ( $p < 0.05$ ) in microhardness of materials in between group.

## Results

The means and standard deviations of the six Vickers' microhardness test groups are given in Table 2. A significant difference was observed between non-coated and coated groups. The microhardness from highest to lowest were found as follow: FV (86.30±3.26), EC (85.65±0.18), SM (85.23±3.28), SB (64.75±3.34), SU (61.82±2.62) and NC (46.45±0.48)

**Table 2: Vickers' microhardness values of resin-modified glass-ionomer cement**

Material (N=60)	Mean
Non-coat (NC)	46.45±0.48 <sup>a</sup>
Fuji varnish (FV)	86.30±3.26 <sup>b</sup>
Equia coat (EC)	85.65±0.18 <sup>b</sup>
Adper scotchbond multi-purpose (SM)	85.23±3.28 <sup>b</sup>
Adper single bond 2 (SB)	64.75±3.34 <sup>c</sup>
Single bond universal (SU)	61.82±2.62 <sup>c</sup>

The value with identical letters indicates no significant difference ( $p < 0.05$ ).

## Discussion

The surface hardness may be defined as the resistance of a material surface to wear. Considering RM-GIC commonly use in direct restoration<sup>14,15</sup>. Suitable maturation of RM-GIC depends on water balance. Both water contamination and dehydration during the initial setting reaction can compromise the physical properties of RM-GIC<sup>16,17</sup>. The application of surface protection seems to preserve the water balance and provide sufficient early protection to prevent the gain and loss of water from RM-GIC<sup>8,9</sup>.

This study proved that the surface protection materials are very effective to avoid the gain and loss of water during setting of RM-GIC<sup>5-9,18-19</sup>. Fatima *et al.*<sup>8</sup>, proved that the microhardness of non-coated samples RM-GIC was reduced significantly compared to the coated samples. They concluded that surface protecting agents including resin varnish were effective. Brito *et al.*<sup>18</sup>, suggested that the GIC coated by Cavitine (Copal varnish) and Adper single bond 2 were significantly higher than non-coated GIC at 24 hours of storage. Zoergiebel *et al.*<sup>19</sup>, found that GIC showed a significantly higher hardness after applying Equia coat compared to the uncoated group. Mensudar *et al.*<sup>9</sup>, found that RM-GIC coated with Equia coat showed a higher value than non-coated RM-GIC. On the contrary, Shintome *et al.*<sup>20</sup>, revealed that no significant difference was observed among the type of varnish protecting agent and non-protected of GIC (Fuji IX) at 24 hours of storage. Varnish for surface protection, whether specific or not, did not prevent the movement of water from the GIC to external environment, probably due to evaporation of the solvent that is present in its composition, which makes the varnish porous, thus allowing the movement of water into the material. Bagheri *et al.*<sup>21</sup>, found that RM-GIC

showed a significantly lower hardness after applying Equia coat compared to the uncoated group at 24 hours of distilled water storage.

According to the results of the present study, microhardness of the RM-GIC, with the protected surface were significantly higher than the unprotected group. Thus, it has been justified from the results that the protecting agents can prevent water contamination and dehydration within 24 hours.

The comparison of microhardness between Fuji varnish, Equia coat, Adper scotchbond multi-purpose adhesive, Adper single bond 2 and Single bond universal found that Fuji varnish, Equia coat and Adper scotchbond multi-purpose adhesive were shown to be the best protection for RM-GIC. Owing to varnish consists of acetone which performs solvent, when it evaporates, the remaining constituents oxidize to form a durable transparent film. Varnish prevents GIC surface from desiccation with a consequent slowing of the rate of desorption<sup>22</sup>. In Equia coat, main composition is methacrylate monomer which is hydrophobic monomer. Adper scotchbond multi-purpose adhesive, the main composition is Bis-GMA (A-diglycidyl ether bisphenol dimethacrylate) related to its resistance to disintegration, low permeability, hydrophobic nature<sup>23</sup> and low viscosity. Low viscosity of Adper scotchbond multi-purpose adhesive favors the formation of a contact angle that allows good adaptation to RM-GIC, thus providing good sealing. Its protective effect of hydrophobic monomer from extrinsic water may allow complete maturation of the RM-GIC reaction with delayed water exposure, thus possibly creating a stronger material. Moreover, the infiltration of Equia coat<sup>24</sup> and Adper scotchbond multi-purpose adhesive fills porosities by increasing the fracture toughness and strengthening the RM-GIC. The dispersion of nanofiller<sup>25</sup> in Equia coat reinforces the outer

layer, which against wear. That's the reason why it can prevent RM-GIC from water imbalance and increase hardness of RM-GIC. For these reasons, Fuji varnish, Equia coat and Adper scotchbond multi-purpose adhesive are recommended to apply for surface protection of RM-GIC.

Adper single bond 2 and Single bond universal have been shown to be a good surface protection for RM-GIC. The microhardness of Adper single bond 2 and Single bond universal groups were lower than the Fuji varnish, Equia coat and Adper scotchbond multi-purpose adhesive groups, because both adhesive agents are basically composed of highly hydrophilic resin monomers<sup>23</sup>. Malacarne *et al.*<sup>26</sup>, shown a positive correlation between the magnitude of water sorption and the degree of hydrophilic adhesives. The hydrophilic adhesive was not enough to prevent fluid transudation. Nguyen *et al.*<sup>27</sup>, observed that increasing the number of coats can only extend the time for water to permeate completely these coatings, but it did not impede water to move across them. Therefore, its decrease the microhardness of RM-GIC. The light cured hydrophilic bonding is a few effective of limiting water movement across the surface of RM-GIC. Thus, it is noticed that coating with Adper single bond 2 and Single bond universal had a small advantage of preventing water movement over RM-GIC.

This study tried to replicate oral cavity conditions such as 37 degree celcius temperature and waterish. Nonetheless, there were some limitations such as the role of artificial saliva that didn't take into consideration.

## Conclusion

The microhardness of resin-modified glass-ionomer cement of all coated groups were

significantly higher than non-coated group. Fuji varnish, Equia coat and Adper scotchbond multi-purpose adhesive groups showed the best surface protection in resin-modified glass-ionomer cement.

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ผู้รับผิดชอบบทความ

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ผศ.ทพ. อวิรุทธ์ คล้ายศิริ

โทรสาร 02-9869205,

99 หมู่ 18 ถนนทันตแพทยศาสตร์

อีเมล: [Dentton@hotmail.com](mailto:Dentton@hotmail.com)

มหาวิทยาลัยธรรมศาสตร์ ศูนย์รังสิต ถ. พหลโยธิน

อ. คลองหลวง จ. ปทุมธานี 12121

## ผลของสารเคลือบผิวต่างชนิดต่อความแข็งผิวระดับจุลภาคของกลาสส์ไอโอโนเมอร์ซีเมนต์ชนิดดัดแปรด้วยเรซิน

อวิรุทธ์ คล้ายศิริ<sup>1</sup>, นันทวรรณ กระจ่างตา<sup>1</sup>, Lili Yang<sup>1</sup>

### บทคัดย่อ

**วัตถุประสงค์:** การวิจัยนี้เพื่อศึกษาค่าความแข็งผิวระดับจุลภาคของกลาสส์ไอโอโนเมอร์ซีเมนต์ชนิดดัดแปรด้วยเรซินหลังเคลือบผิวด้วยสารเคลือบผิวต่างชนิด

**วัสดุและวิธีการศึกษา:** เตรียมกลาสส์ไอโอโนเมอร์ซีเมนต์ชนิดดัดแปรด้วยเรซินจำนวน 60 ชิ้นจากแม่พิมพ์สแตนเลสเส้นผ่าศูนย์กลาง 6 มิลลิเมตร สูง 6 มิลลิเมตร แบ่งเป็น 6 กลุ่ม ๆ ละ 10 ชิ้น ตามวิธีการเคลือบผิวด้วยสารเคลือบผิวดังนี้ 1. ไม่ใช้สารเคลือบผิว (NC) 2. Fuji varnish (FV) 3. Equia coat (EC) 4. สารยึดติดของ Adper scotchbond multi-purpose (SM) 5. Adper single bond 2 (SB) และ 6. Single bond universal (SU) หลังจากทาสารเคลือบผิวทิ้งไว้ 10 นาทีเพื่อให้เกิดการก่อตัวเริ่มต้น จากนั้นนำไปแช่น้ำกลั่นที่อุณหภูมิ 37 องศาเซลเซียส เป็นเวลา 24 ชั่วโมง ขัดผิวหน้าของชิ้นทดสอบด้วยเครื่องขัด 20 วินาที แล้วนำไปวัดค่าความแข็งผิวระดับจุลภาคด้วยเครื่อง FM-800 วิเคราะห์ข้อมูลทางสถิติด้วยการทดสอบความแปรปรวนทางเดียว และเปรียบเทียบความแตกต่างระหว่างกลุ่มด้วยแทมเฮนย์

**ผลการศึกษา:** กลุ่มที่ทาสารเคลือบผิวจะมีค่าความแข็งผิวระดับจุลภาคสูงกว่ากลุ่มที่ไม่ทาสารเคลือบผิวอย่างมีนัยสำคัญทางสถิติ โดยกลุ่ม FV, EC และ SM จะมีค่าความแข็งผิวระดับจุลภาคสูงกว่ากลุ่ม SB และ SU อย่างมีนัยสำคัญทางสถิติ

**สรุป:** ความแข็งผิวระดับจุลภาคของกลาสส์ไอโอโนเมอร์ซีเมนต์ชนิดดัดแปรด้วยเรซินของกลุ่มที่ทาสารเคลือบผิวมีค่าสูงกว่ากลุ่มที่ไม่ทาสารเคลือบผิวอย่างมีนัยสำคัญทางสถิติ โดยพบว่า Fuji varnish, Equia coat และสารยึดติดของ Adper scotchbond multi-purpose เป็นสารเคลือบผิวที่ดีที่สุด

**คำสำคัญ:** ความแข็งผิวระดับจุลภาค; กลาสส์ไอโอโนเมอร์ซีเมนต์ชนิดดัดแปรด้วยเรซิน; สารเคลือบผิว

<sup>1</sup>สาขาวิชาทันตกรรมหัตถการ คณะทันตแพทยศาสตร์ มหาวิทยาลัยธรรมศาสตร์ จังหวัดปทุมธานี