

# Effect of Nano-filled Protective Coating on Microhardness and Wear Resistance of Glass-ionomer Cements

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## Abstract

The purpose of this study was to investigate the effect of adding a protective coating on the microhardness and wear resistance of glass ionomer cements (GICs).

Specimens were prepared from GIC and resin-modified GIC (RMGI), and divided into 3 groups based on surface protection: (1) no coating (NC), (2) Equia coat coating (EC), and (3) un-filled adhesive coating (AD). All specimens were then placed in distilled water for 24 h. Surface hardness (n = 10) was evaluated on a Vickers hardness testing machine. Wear resistance (n = 10) was evaluated after subjecting the specimen to thermocycling for 10,000 cycles using a chewing simulator. Data were analyzed using a one-way ANOVA and the Kruskal-Wallis test.

Surface hardness was highest in the NC groups, followed by the EC and AD groups. The wear depth of GI + NC was significantly higher than that of all RMGI groups. EC did not significantly lower the wear depth compared to AD.

Based on these results, it was concluded that although EC does not increase the surface microhardness of GIC, it can increase the wear resistance.

**Key words :** Surface protection, Glass ionomer cement, Resin-modified glass ionomer cement, Surface hardness, Wear

## I . Introduction

Glass ionomer cement (GIC) is a tooth-colored restorative material that releases fluoride and chemically bonds to tooth structure[1]. This material has a coefficient of thermal expansion similar to that of a tooth, and is widely used in pediatric dentistry without a bonding agent[2-4].

However, some properties of GIC may limit its application[5]. GIC has low surface hardness, strength, and wear resistance compared to other restorative materials such as amalgam and composite resins[6]. The long setting reaction time and sus-

ceptibility to moisture during setting reaction restricts its widespread clinical use[7].

In order to overcome these drawbacks, resin-modified GI (RMGI) was introduced at the end of the 1980s with improved mechanical properties as well as operability and aesthetics[8]. During the early stage of setting, photopolymerization of the resin component occurs first and is followed by chemical acid group reaction of the glass ionomer component, resulting in a fully matured double polymerized product[9].

During the initial stage, which occurs within first 10 minutes after mixing, GIC is sensitive to water uptake. The second

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stage, slower acid-base reaction, lasts 24 hours and is susceptible to dehydration[10]. Therefore, it is recommended to protect the surface of GIC during the first 24 hours to avoid a decrease in the mechanical properties of the GIC application[5].

In order to protect the restorative material from water contamination, immediate application of a surface coating agent is recommended[11]. These include solvent based and light-cured bonding resins, petroleum jelly, or fluoride varnish[5]. Recently, a new generation of coating for GIC known as the Equia coat, which is a self-adhesive resin containing nanofillers, has been introduced. The manufacturer claims that Equia coat protects the restoration from wear and dissolution, increases the surface hardness, and mechanical stress is dispersed due to the evenly dispersed nanofillers and tough coating layer.

However, a few studies have compared the surface protective coating effect of the Equia coat with non-filled resin adhesive. And none of these studies have evaluated the wear resistance of GI and RMGI under thermocycling conditions. Therefore, this study was carried out to evaluate whether nanofilled resin-based coatings increase the microhardness and wear resistance of GI and RMGI when stored in water for 24 hours, compared to coating with non-filler adhesives and no surface coating.

## II. Materials and methods

### 1. Specimen preparation

The materials used in this study are listed in Table 1. 60 discs with a diameter of 9.0 mm and a thickness of 2.0 mm were fabricated for each specimen. Fuji IX GP EXTRA (GI) and Fuji IX LC (RMGI) were used according to the manufacturer's instructions

and mixed for 10.0 seconds using Rotomix (3 M ESPE, Seefeld, Germany). After injection into the mold, the samples were covered with transparent matrix strips (Matrix-Strips, Orbis, Münster, Germany) and a transparent glass slab was placed over them. GI was allowed to set for 150.0 seconds while RMGI was light cured for 20.0 seconds using a light-emitting diode (LED) with a standard power mode (1000.0 mW/cm<sup>2</sup>).

The specimens were then removed from the mold and the surfaces were ground using #600 grit silicon carbide paper. The specimens were then randomly grouped into 3 batches (20 in each batch for each material). The first batch (NC) was considered as the control and was not subjected to any further treatment. A thin layer of Equia coat was applied to the surface of the specimens in the second batch (EC) using a micro brush and light cured for 20 seconds. An unfilled adhesive, Scotchbond Multi-Purpose (3M Dental Products, St Paul, MN., USA), was similarly applied over the surfaces of the specimens in the third batch (AD) with a micro brush and light cured for 20.0 seconds. All the specimens were labeled, stored, and tested after being immersed for 24 h in distilled water at 37.0°C.

### 2. Vickers hardness test

10 specimens per group were subjected to a hardness test for 15.0 s at 300.0 g load using a Vickers microhardness test machine (Mitutoyo, Kawasaki, Japan). The Vickers machine comprises a diamond shaped indenter with a square base (Vickers pyramid) and with an opening angle of 136.0° which is pressed vertically on to the surface of the objects being tested. Each specimen was subjected to three indentations and the average values were calculated.

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

Code	Material	Manufacturer	Chemical composition	Material type
GI	Fuji IX GP Extra (A2 shade)	GC Europe (Leuven, BE)	Aluminium-fluoro-silicate glass, polybasic carboxylic acid, polyacrylic acid	Self-cure (conventional) glass-ionomer cement
RMGI	Fuji II LC (A2 shade)	GC Corporation, Tokyo, Japan	Aluminium-fluoro-silicate glass/ Poly-HEMA	Resin-modified glass-ionomer cement
EC	Equia coat	GC America, IL, USA	Methylmethacrylate, colloidal silica, camphoroquinone, urethane, methacrylate, phosphoric ester, monomer	Nanofilled self-adhesive light-cured protective coating
AD	Adper Scotchbond Multi-Purpose adhesive	3M ESPE	HEMA, Bis-GMA, initiator	Unfilled adhesive

HEMA = hydroxyethylmethacrylate, Bis-GMA = bisphenol A-glycidyl methacrylate

### 3. Wear

10 specimens per group were subjected to a wear test. The wear test was conducted using a masticatory simulator (Chewing simulator SC-4.8; SD Mechatronik, Feldkirchen-Westerhan, Germany) that has 8 chambers simulating the vertical and horizontal movements simultaneously in the thermocycling condition (Fig. 1)[12,13]. Each of the chambers consists of an upper metal antagonist and a lower plastic sample holder in which the specimen can be embedded. For fixation, the specimens were embedded in acrylic resin in the lower holder. A 5.0 mm vertical movement and a 3.0 mm horizontal movement were reproduced with a vertical load of 2.0 kg for 10,000 cycles at 1.7 Hz in the presence of simultaneous thermal stress (Table 2).

The wear depths ( $\mu\text{m}$ ) of specimens were determined using a micrometer (Mitutoyo, Kawasaki, Japan).

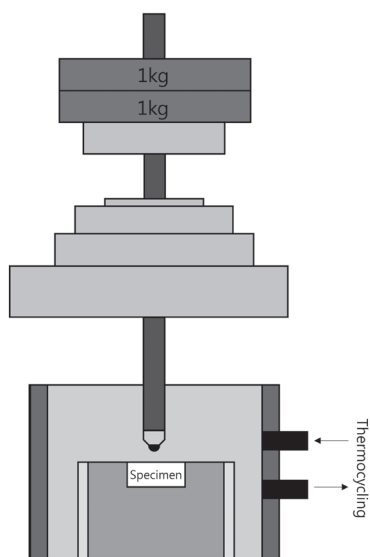


Fig. 1. Cross-sectional illustration of wear test.

Table 2. The experimental conditions of chewing simulator

Parameter	Condition
Cold/Hot temperature	5.0°C/55.0°C
Vertical movement	5.0 mm
Rising speed	60.0 mm/s
Descending speed	30.0 mm/s
Weight per sample	2.0 kg
Dwell time	60.0 s
Horizontal movement	3.0 mm
Forward speed	30.0 mm/s
Backward speed	30.0 mm/s
Cycle frequency	1.7 Hz

### 4. Statistical analysis

Statistical analysis was performed using SPSS 22.0 (IBM Corp., Armonk, NY, USA) software. The Shapiro-Wilk normality test was used to ensure that the normality assumption of all the wear data was met. Mean values and standard deviation of Vickers hardness were calculated and analyzed using a one-way ANOVA. Post hoc analyses among group means were conducted using a Tukey test. The wear test was analyzed with the Kruskal-Wallis test and bonferroni's post hoc test.

## III. Result

### 1. Vickers hardness test

Vickers hardness numbers are shown in Tables 3 and 4. The Vickers hardness number of GI + AD group was the lowest while that of RMGI + NC group was the highest. Both GI and RMGI groups without surface protection showed significantly higher Vickers hardness than those with surface protection.

### 2. Wear

The mean and standard deviation values of the different samples are shown in Tables 5 and 6. The GI + NC group had the highest wear rate while RMGI + EC had the lowest wear rate. All GI groups had a higher wear rate than that of the RMGI groups.

A significantly higher amount of wear was noted with GI + NC compared to RMGI + NC ( $p = 0.030$ ), RMGI + EC ( $p = 0.000$ ), and RMGI + AD ( $p = 0.010$ ). No significant difference was observed within the GI and RMGI groups. GI + AD showed a significantly higher wear rate than that of RMGI + EC ( $p = 0.012$ ). The wear rate of GI + EC was not significantly different from that of RMGI + EC, which had with the lowest wear rate ( $p = 0.286$ ).

Table 3. Mean vickers hardness number and standard deviation of materials

	Mean Vickers Hardness Number $\pm$ SD	
	GI	RMGI
NC	21.35 $\pm$ 1.93	51.57 $\pm$ 3.03
EC	16.12 $\pm$ 2.42	32.37 $\pm$ 3.11
AD	12.95 $\pm$ 4.20	26.32 $\pm$ 3.85

GI = glass ionomer, RMGI = resin-modified glass ionomer, NC = no coating, EC = equia coat, AD = adhesive

**Table 4.** Correlation between values of vickers hardness number

	GI + NC	GI + EC	GI + AD	RMGI + NC	RMGI + EC	RMGI + AD
GI + NC						
GI + EC	0.007					
GI + AD	0.000	0.245				
RMGI + NC	0.000	0.000	0.000			
RMGI + EC	0.000	0.000	0.000	0.000		
RMGI + AD	0.012	0.000	0.000	0.000	0.001	

GI = glass ionomer, RMGI = resin-modified glass ionomer, NC = no coating, EC = equia coat, AD = adhesive  
One-way ANOVA, Tukey test

**Table 5.** Mean and standard deviation of materials wear depth

	Mean wear depth ± SD	
	GI	RMGI
NC	55.25 ± 31.67	19.30 ± 10.17
EC	26.35 ± 16.87	11.25 ± 7.15
AD	30.50 ± 10.09	16.30 ± 9.86

GI = glass ionomer, RMGI = resin-modified glass ionomer, NC = no coating, EC = equia coat, AD = adhesive

**Table 6.** Correlation between results of the wear test

	GI + NC	GI + EC	GI + AD	RMGI + NC	RMGI + EC	RMGI + AD
GI + NC						
GI + EC	0.526					
GI + AD	1.000	1.000				
RMGI + NC	0.030	1.000	0.685			
RMGI + EC	0.000	0.286	0.012	0.172		
RMGI + AD	0.010	1.000	0.306	1.000	1.000	

GI = glass ionomer, RMGI = resin-modified glass ionomer, NC = no coating, EC = equia coat, AD = adhesive  
Kruskal-Wallis test, Bonferroni's post-hoc test

#### IV. Discussion

The setting reaction of GI is an acid-base reaction, which is caused by the interaction of the poly acid liquid with the glass powder. The acid attacks the glass network and releases cations such as Al<sup>3+</sup> and Ca<sup>2+</sup> or Sr<sup>2+</sup>. Within the first 10 minutes after mixing, calcium polyacrylate, which is vulnerable to hydrolysis, is formed. This matrix is subsequently converted to a more stable form (aluminum polyacrylate) over the first 24 hours[14-16]. Therefore, the cement surface must be protected from water contamination during the setting reaction to

prevent the dissolution of metal cations. Reduced mechanical properties and increased surface corrosion and wear tendency has been reported in water contaminated (during early setting) GIC restorations [17].

Resin-modified GICs were developed in the 1980s to enhance the weak physical properties of conventional GICs. With the addition of 2-hydroxyethylmethacrylate (HEMA), a hydrophilic resin monomer, and a photoinitiator[18], RMGI had a higher compressive strength when compared with conventional GI during the initial setting stage (within the first 24 h)[10]. However, since RMGI still has the properties

of conventional GIC, it is important to prevent early water contamination and dehydration. Miyazaki *et al.*[19] claimed that the surface of RMGI should be protected from water contamination for at least 1 hour after cement mixing.

In this study, compared to non-surface protected specimens, both GI and RMGI specimens with surface protection showed significantly lower surface hardness. This result is opposed to the manufacturer's claim that the nanofilled coating increases surface hardness of the restoration. The surface hardness of the surface protection materials was lower than that of the restorations. Faraji *et al.*[20] reported that conventional GI with nanofilled coating exhibited lower Vickers hardness than GI without coating. This may be due to the thickness of the coating itself, since the coating was applied in accordance with the manufacturer's instructions (light cured without additional air drying). In general, protective coatings do not have the required mechanical properties of a suitable restorative material.

The manufacturers of Equia coat report that it contains nanofillers. Fillers are the strongest content in resin components that are added to strengthen the composite resin and decrease the percentage of resin monomer. Kim *et al.*[21], noted that increasing the filler content enhanced the mechanical properties of the restorative material. Shinkai *et al.*[22], reported that restoration containing small sized fillers exhibited better wear resistance compared with that of restorations containing large fillers. Therefore, the higher surface microhardness in specimens coated with the Equia coat in this study is thought to be due to the filler content compared to the filler-free adhesive.

All GI groups had lower Vickers hardness number than that of the RMGI groups. Addition of the resin component in RMGI not only decreases initial hardening time and handling difficulties, but also increases physical strength of the cement[23].

Wear resistance is an important property for all restorative dental materials. This is the ability of the restoration to withstand the grinding force of the opposing tooth and food, while maintaining its function. Despite some beneficial properties, GICs have been proven unsuitable for stress-bearing areas due to their poor wear resistance[24,25]. The use of EC has been recommended by its manufacturer as it strongly bonds to GI and improves abrasion resistance. The chewing simulator is a two-body wear test machine and the main wear mechanism acting in this study was abrasion in combination with surface fatigue[26].

In both GI and RMGI, surface protection increased wear

resistance compared to NC, but the difference was not statistically significant. This seemed to be due to the protection from water contamination during the first 24 hours of initial hardening. Hotta *et al.*[27] reported that the use of light-polymerized agents can restrict water movement across the cement surface. In previous studies, the surface protection significantly increased wear resistance, but in this study, wear resistance between coated groups and uncoated groups did not show statistically significant differences. This may be due to the small sample size and large deviation of results of this study.

In this study, EC increased the wear resistance compared to AD in both GI and RMGI. This may be due to the microfiller contained in the EC. However, the difference was not statistically significant. Therefore, it would be expected that the effect of the nanofiller is less than that of water contamination prevention which is increased the wear resistance.

The RMGI has improved physical properties because of its resin monomer, and shows excellent initial strength. Croll and Nicholson[23] reported that fracture toughness, fracture resistance, and resistance to wear are all improved in the resin-modified glass ionomer. However, Lohbauer[10] claimed that RMGI are more prone to abrasive wear due to a weak filler-matrix coupling. In this study, uncoated GI showed the lowest wear resistance. However, no significant difference was observed between all RMGI groups and GI when surface protection was applied to GI, regardless of the coating type. These results suggest that GI is more sensitive to early water contamination than RMGI, and therefore it is thought to have a greater effect on coatings.

There are a few limitations of this study. The hardness of only the outermost surface protection material was assessed and it does not reflect that of the actual restorative material. Further, this study was conducted in the laboratory without recreating the complete oral environment. Additionally, the specimens were stored in water for only 24 hours, and hence the long-term effect on the surface coating could not be evaluated.

## V. Conclusion

Application of surface protection did not have a major influence on the hardness of the two types of GI cements. However, the wear resistance of GI increased following the use of surface protection and was similar to that of RMGI. The presence of nanofillers did not significantly affect wear resistance.

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국문초록

## 나노필러가 함유된 표면보호재가 글라스 아이오노머 시멘트의 미세경도와 마모저항성에 미치는 효과

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이 연구의 목적은 글라스 아이오노머 시멘트의 표면 보호가 미세경도와 마모저항성에 미치는 효과를 알아보고자 하는 것이다. 글라스 아이오노머와 레진강화형 글라스 아이오노머를 사용하러 각각 60개의 시편을 제작하였다. 각 시편을 표면 보호를 시행하지 않은 경우, 나노필러가 함유된 표면 보호재, 필러가 함유되어 있지 않은 표면 보호재에 따라 20개씩 나누었다. 37°C 증류수에 24시간 보관한 후 각 군당 10개의 시편은 비커스 미세경도를 측정하였고, 10개의 시편은 마모 시험을 시행한 후 마모된 깊이를 측정하였다. 표면 보호를 시행한 군들보다 표면 보호를 시행하지 않은 군들이 높은 표면경도를 보였다. 글라스아이오노머와 레진강화형 글라스아이오노머 모두에서 표면 보호를 시행한 경우에 마모저항성이 더 증가하였지만 유의한 차이는 아니었다. 나노필러의 유무는 마모도에 유의한 영향을 미치지 못하였다.