# Effect of Coating Agents on Surface Microhardness of Highly Viscous Glass Ionomer Cement/Resin Coatings for Atraumatic Restorative Treatment after an Acid Challenge

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**Abstract**

This study evaluated the effect of coating agents during complete setting on the surface microhardness of highly viscous glass ionomer cement/resin coatings after immersion in citric acid solution. A total of 104 specimens (52 specimens per product) of highly viscous glass ionomer cement/resin coating (EF; EQUIA Forte, GC) and highly viscous glass ionomer cement (FN; Fuji IX GP, GC), 10 mm. diameter and 1.5 mm thickness, were divided into 4 groups each (13 specimens/group) according to a coating method; uncoated control groups (EF, FN), resin-coated and polished groups (EFCP, FNCP), petroleum jelly-coated groups (EFP, FNP), and resin-coated groups (EFC, FNC). Specimen microhardness values were measured for baseline data. All specimens were immersed in 5 ml of citric acid solution and microhardness was measured again on day 3 and 7. One-way ANOVA with repeated measurement was used to analyze the analytical statistics and Tukey's HSD and t-test was used for multiple comparisons (α=0.05).

After 7 days of immersion, group FNC had the highest surface microhardness and had a statistically significant difference from other groups (P<0.05).

In conclusion, Fuji IX GP coated with EQUIA Forte Coat (group FNC) was suitable for dental restoration in acid challenge condition.

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## Introduction

Atraumatic restorative treatment (ART) is an alternative method for dental caries management. ART uses only hand instruments, which is the least invasive method, to completely remove necrotic carious tooth tissue and conserve partially demineralized dentine.[1] It aims to prevent caries and stop the progression of caries down to deeper layers.[2] ART is suitable for all situations and can be used either in general clinics or in the field; for example, in nursing homes or the patient's home.[3] The material selected for the ART technique is highly viscous glass ionomer cement,[2] which yields better mechanical properties than other restorative materials.[4-6]

ART protocol consists of a cavity preparation and a restoration procedure with a glass ionomer cement, which requires a mixer to prepare a material. When the material is ready mixed, the material was injected into the cavity and condensed with an index finger against the cavity. The excess material was removed with a carver, and then was applied petroleum jelly to protect the material during the initial setting reaction[7] or coated with the resin using a micro-tip brush and light polymerized for 20 s.[8]

Highly viscous glass ionomer cement requires a surface coating to prevent moisture contamination similar to that of a traditional glass ionomer cement restoration. Widely used coating materials are cocoa butter, petroleum jelly, waterproof varnishes, methyl methacrylate, amide, and filled, light-cured, bonding resins.[9,10] The coating is immediately applied to prevent

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moisture contamination because in the first 10 minutes of the setting process of the glass ionomer cement, calcium ions are released slowly inside the matrix followed by the aluminum ion. During this time, glass ionomer cement is very sensitive to dehydration. If glass ionomer cement receives or loses water in this phase, it could have a significant impact on the final properties of the restoration. Therefore, surface coating is needed to maintain an optimal water balance.[9,10]

In many countries, such as Australia, the Philippines and Thailand, people frequently consume acidic food, sour fruits and drinks resulting in a high incidence of dental erosion.[11,12] The potential erosive effect of these acidic foodstuffs on enamel occurs primarily by dissolution of apatite crystals.[12-14] In an oral environment, both surface degradation and erosion of highly viscous glass ionomer/resin coating restoration for ART may occur. However, there have not been any studies conducted to explain the effect of acidic solutions on the surface alteration of highly viscous glass ionomers/resin coating. This acidity may result in a change in the properties of the material which may lead to failure of dental restoration. Therefore, the aim of this study was to study the effectiveness of different coating materials on highly viscous glass ionomer cement/resin coating and glass ionomer cements after being immersed in citric acid solution. The null hypothesis was that there was no significant difference in surface microhardness values among different coating agents of highly viscous glass ionomer cement/resin coating and glass ionomer cements after citric acid solution immersion.

## Materials and methods

### Specimen preparation

A total of one hundred and four disc-shaped specimens (52 specimens per product; 10.0 mm in diameter and 1.5 mm in thickness) of a highly viscous glass ionomer cement/resin coating and a highly viscous glass ionomer cement (shade A3, Table 1) were prepared. The capsule (powder and liquid) was mixed in a mixer (model HSM3, Monitex Industrial, New Taipei City, Taiwan (ROC)) for 10 s and loaded in a polytetrafluoroethylene cylindrical mold on a glass plate covered with a mylar matrix strip. The mylar strip was then covered with a second glass plate. A static load of 20 N was applied to extrude excess materials and to achieve a smooth and flat surface on each specimen.

| Material | Type | Composition | Filler size (nm) | Manufacturer |
|---|---|---|---|---|
| EQUIA Forte Fil | Highly viscous glass ionomer cement/resin coating | Powder: fluoroaluminosilicate glass, surface-treated glass<br>Liquid: aqueous polyacrylic acid | 100-200 | GC Corp., Tokyo, Japan |
| Fuji IX GC Gold Label IX EXTRA | Highly viscous glass ionomer cement | Powder: strontium fluoroalumino-silicate glass<br>Liquid: aqueous polyacrylic acid | 1000 | GC Corp., Tokyo, Japan |
| EQUIA Forte Coat | Nano-filled resin | Silica, methyl mathacrylate photoinitiators | 40 | GC Corp., Tokyo, Japan |
| 0.03% citric acid solution (pH = 3) | - | Powder: 0.29 g of citric acid monohydrate (MW = 210.14 g/mol)<br>Liquid: 1000 mL of water | - | Elago Enterprises, NSW, Australia |

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

The specimens were then left for setting for 150 s according to the manufacturer's instructions. After setting, the mylar strip and the glass plate on the top and bottom of the mold were removed. Consequently, fifty two specimens of each material were divided into 4 groups of 13 specimens according to the coating method as follows:

Group EF (EQUIA Forte Fil); no coating as the control group,

Group EFCP (EQUIA Forte Fil + EQUIA Forte Coat and polished coat); applied EQUIA Forte Coat and light polymerized (LED Curing Light, Elipar DeepCure, 3M ESPE, MN, USA) for 20 s,

Group EFP (EQUIA Forte Fil + petroleum jelly); petroleum jelly was applied,

Group EFC (EQUIA Forte Fil + EQUIA Forte Coat and no polished coat); applied EQUIA Forte Coat and light polymerized (LED Curing Light, Elipar DeepCure, 3M ESPE) for 20 s,

Group FN (Fuji IX); no coating as the control group,

Group FNCP (Fuji IX + EQUIA Forte Coat and polished coat); applied EQUIA Forte Coat and light polymerized (LED Curing Light, Elipar DeepCure, 3M ESPE) for 20 s,

Group FNP (Fuji IX + petroleum jelly); petroleum jelly was applied,

Group FNC (Fuji IX + EQUIA Forte Coat and no polished coat); applied EQUIA Forte Coat and polymerized (LED Curing Light, Elipar DeepCure, 3M ESPE) for 20 s.

All groups of the specimens were kept at 37ºC in an incubator (Memmert, model BE500; Memmert GmbH, Schwabach, Germany) for 24 hr (for complete setting reaction of glass ionomer cement[15]). Specimens of groups EFCP, EFP, FNCP, and FNP were then mechanically polished (Phoenix 4000; Buehler GmbH, Dusseldorf, Germany) under running water using 1,200-grit silicon carbide paper (3M ESPE) to remove coating agents before surface microhardness testing.

**Surface microhardness measurement**

Before citric acid immersion, disc specimens were measured with a microhardness tester (Buehler Micromet II; Buehler Ltd, Lake Bluff, IL, USA) using a Vickers diamond tip under a 100-g indentation load for 10 s. Five indentations per specimen were performed on the top surface and the mean value of each specimen was calculated. Subsequently, each specimen was immersed in 5 ml of citric acid solution at 37ºC for 7 days. Surface microhardness measurement was performed on day 3 and 7. The citric acid solution was daily changed every 24 hr.

**Surface morphology analysis**

To examine the effect of citric acid solution on surface morphology, three specimens from each group were examined under scanning electron microscopy (SEM). The specimens were rinsed with distilled water for 5 minutes, dried, and fixed onto an aluminum cylinder, 13 mm in diameter and 10 mm in height. Subsequently, the specimens were sputter coated with a gold-palladium alloy (SPI Module Sputter system; SPI Supplies, West Chester, PA, USA). The specimens were then examined under SEM (JSM-5800LV Scanning Electron Microscope; JEOL Ltd, Tokyo, Japan) before and after day 3 and 7 of citric acid solution immersion.

**Statistical methods**

The data were statistically analyzed. A one-way analysis of variance (ANOVA) with repeated measurements was performed to assess the surface microhardness values. Within-groups analysis with Tukey's HSD and *t*-test were also performed (α=0.05).

## Results

The surface microhardness values of the materials used before and after the citric acid solution are presented in Table 2. For before immersion, group FNCP had the most surface microhardness values and had statistically significant difference from other groups (P<0.05). After 7 days immersion, group FNC had the most surface microhardness values and had statistically significant difference from other groups (P<0.05).

| Material | Coating agent groups | Mean surface microhardness (kg/mm²) (SD) at different time (days) | | |
|---|---|---|---|---|
| | | Before immersion | 3 | 7 |
| EQUIA Forte | Control (EF) | 55.83 (16.27)$^{b,C,D}$ | 19 (8.19)*$^{,b,B}$ | 6.31 (0.73)*$^{,b,c,D}$ |
| | EQUIA Forte Coat and polishing coat after setting (EFCP) | 89.29 (10.63)$^{a,A,B}$ | 20.67 (3.25)*$^{,b,B}$ | 7.23 (1.09)*$^{,b,C,D}$ |
| | Petroleum jelly (EFP) | 59.05 (17.7)$^{b,C,D}$ | 15.43 (4.48)*$^{,b,B}$ | 4.92 (0.45)*$^{,c,D}$ |
| | EQUIA Forte Coat and non-polishing coat after setting (EFC) | 66.26 (7.03)$^{b,C}$ | 70.65 (7.06)$^{a,A}$ | 67.38 (3.01)$^{a,B}$ |
| Fuji IX GP | Control (FN) | 41.17 (18.97)$^{c,D}$ | 15.11 (6.35)*$^{,b,B}$ | 5.51 (0.61)*$^{,c,D}$ |
| | EQUIA Forte Coat and polishing coat after setting (FNCP) | 93.89 (8.72)$^{a,A}$ | 21.67 (5.18)*$^{,b,B}$ | 10.22 (0.91)*$^{,b,C}$ |
| | Petroleum jelly (FNP) | 67.76 (23.68)$^{b,C}$ | 19.70 (5.38)*$^{,b,B}$ | 5.73 (0.76)*$^{,c,D}$ |
| | EQUIA Forte Coat and non-polishing coat after setting (FNC) | 70.94 (8.71)$^{b,B,C}$ | 75.44 (9.69)$^{a,A}$ | 72.63 (6.22)$^{a,A}$ |

**Table 2.** Mean microhardness of materials immersed in citric acid solution at different times.
*indicates statistically significant difference (in rows) from the before experiment value according to the *t*- test (P<0.05); $^{a-d}$ indicates statistically significant difference (in columns) among groups (in column) for each material according to Tukey's HSD test (P<0.05); $^{A-G}$ indicates statistically significant difference (in columns) among groups and materials (in column) according to Tukey's HSD test (P<0.05).

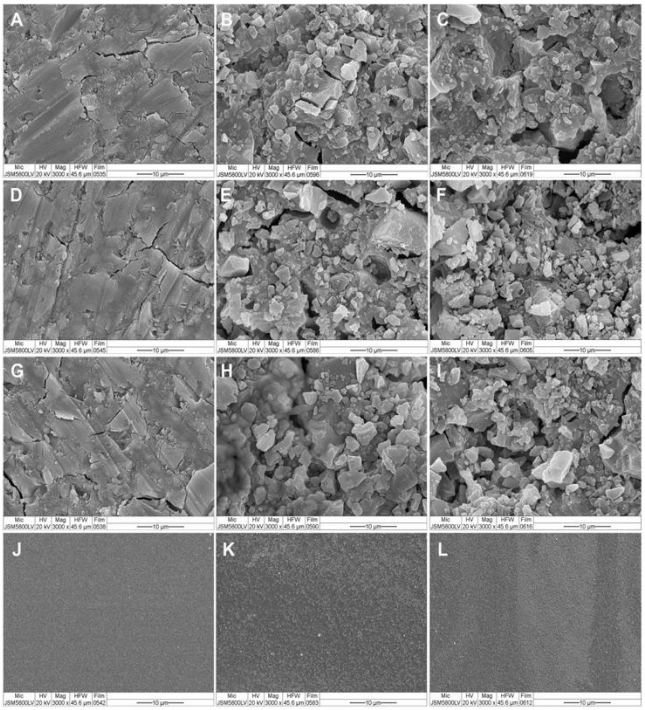


**Figure 1.** SEM photomicrographs of EQUIA Forte Fil (×3000). (A-C) group EF- no coating at before immersion, day 3 and day 7, respectively; (D-F) group EFCP- EQUIA Forte Coat and polished coat at before immersion, day 3 and day

7, respectively; (G-I) group EFP- petroleum jelly at before immersion, day 3 and day 7, respectively; (J-L) group EFC- EQUIA Forte Coat and no polished coat at before immersion, day 3 and day 7, respectively.

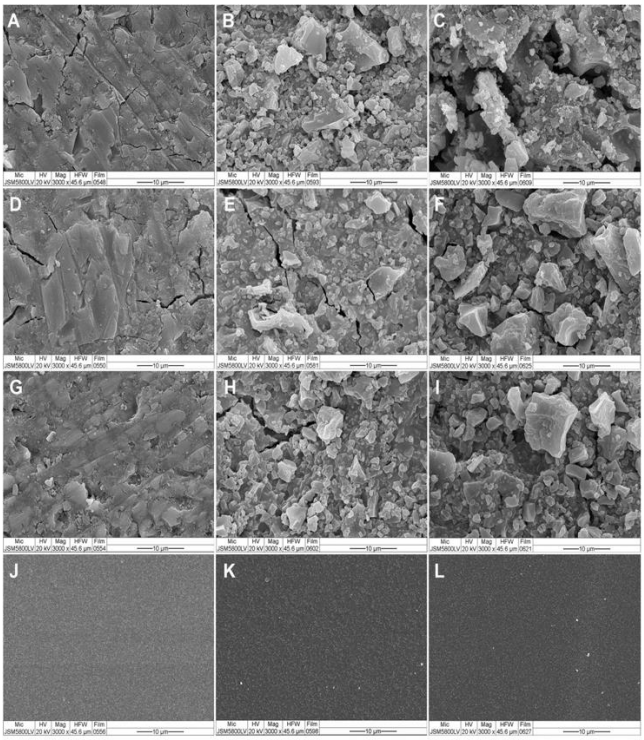


**Figure 2.** SEM photomicrographs of Fuji IX GC (×3000). (A-C) group FN- no coating at before immersion, day 3 and day 7, respectively; (D-F) group FNCP- EQUIA Forte Coat and polished coat at before immersion, day 3 and day 7, respectively; (G-I) group FNP- petroleum jelly at before immersion, day 3 and day 7, respectively; (J-L) group FNC- EQUIA Forte Coat and no polished coat at before immersion, day 3 and day 7, respectively.

SEM photomicrographs of the examined materials, before immersion and after 3 and 7 days immersion in citric acid solution are presented in Figures 1 and 2. Before immersion, all groups (except groups EFC; fig. 1J, and FNC; fig. 2J) showed rough, scratched and cracked surfaces. After citric acid solution immersion, all groups (except groups EFC; figs. 1K and 1L, and FNC; figs. 2K and 2L) presented rougher surfaces, however, they were time dependent.

## Discussion

The results of the present study support rejection of the null hypothesis. After 7 days of citric acid solution immersions, there was significant difference in surface microhardness values among different coating agents of highly viscosity glass ionomer cement/resin coating and glass ionomer cements. In the oral cavity, restorative materials must be exposed to various conditions such as temperature and pH change from food or beverages. Therefore, the restorative materials used in the oral cavity should withstand or exhibit minimal change in these situations. In this study, the degradation of highly viscous glass ionomer cement restorative materials coated with various materials was simulated by continuous immersion in citric acid solution for 7 days, and surface microhardness wase determined. The continuous immersion in the acid solution for 7 days which simulated the worse degradation to the restorative materials, equals the continuous consumption of acidic food and beverages for 6 months.[16]

The restorative materials selected in this study are the popular materials used in the ART technique[17] and are used to compare both in vitro and in vivo studies.[18] The present results showed that the citric acid solution could decrease the surface microhardness of both materials as also seen in SEM photomicrographs, which corresponded to an other study.[19] SEM photomicrographs showed high corrosiveness of the materials tested. This is the result of the hydrogen ions in the citric acid solution penetrating the metal ions, which act as a binder between the carboxylic acids in the glass ionomer cement, forming free metal ions (especially trivalent aluminium) and leaching outside the material surface. When the metal ions in the glass ionomer cement are reduced, the polysalt matrixes (results from the formation of contact cation-anion ion pairs or complexes between the carboxylic groups of the polyalkenoic acid and metallic ions) of the set cement are destroyed.[19,20]

Regarding comparison between two highly viscous glass ionomer cements, EQUIA Forte Fil and Fuji IX GC, the current results presented that Fuji IX GC had higher surface microhardness values than EQUIA Forte Fil, even after citric acid solution immersion, which was in agreement with an other study.[21] The

reason was possibly due to the concentration of the fillers in the materials and firm adhesion of the fillers to the matrix, thus increasing the surface microhardness.[22] In addition, the size and shape of the fillers might make a difference in surface microhardness values.[22] Composition of Fuji IX GC also included strontium (Sr) as an element, which increases the mechanical properties of the material.[23]

This study investigated the surface microhardness of highly viscous glass ionomer cements coated with different agents during complete settings after exposure to citric acid solution. Therefore, an experiment was designed to study the efficacy of coating agents during complete settings by polishing specimens to remove the coating agent with sandpaper after 24 hr. Surface microhardness values of the materials were measured and then immersed in citric acid solution, which was different from an other study[19] where efficacy of the coating agent was not studied. Surface microhardness measurement in this study showed the real microhardness values of the materials, not the microhardness values of the coating agents (except in groups EFC and FNC). The present results obtained from the surface microhardness values demonstrated that surface microhardness was significantly reduced after citric acid immersion, when compared with no coating agent removal (groups EFC and FNC). On the other hand, this result showed that a coating agent prevented surface degradation of the materials (groups EFC and FNC), providing better surface microhardness values. Surface microhardness obtained from these groups after immersion in citric acid solution also did not reduce significantly over the time period, which consistent with a study by Brkanovic et al.[24] This might be because the resin coating decreased rigidity, increased material flow (increased loss modulus), reduced early water absorption, and reduced the water-loss process which increased the mechanical properties of the materials over the time period.[25-29]

Regarding the results of petroleum jelly coating (groups EFP and FNP), the surface microhardness values significantly decreased over the time period compared with the other groups except in the control groups (groups EF and FN). Petroleum jelly is used for coating in the ART technique because it is easy to use.[7] However, this study showed that citric acid solution decreased microhardness values of highly viscous glass ionomer cements coated with petroleum jelly over the time period, which is consistent with another study.[30] Therefore, if resin coating could be provided instead, petroleum jelly might not be useful for coating the surface in the ART technique.

This study was an in vitro study. It was difficult to simulate an oral environment with other factors related to the degradation of highly viscous glass ionomer cements such as buffer capacity, flow rate of saliva or the acidity of saliva that may reduce the severity of the acid solution. This study also did not take into account the abrasion factor from the occlusion system. It is difficult to simulate situations similar to complex oral conditions. Therefore, the results obtained from this study might be exaggerated since it did not allow for any of the above factors. However, the results of this study could serve as a basis data for further study. Further studies should be required with the addition of the above factors or an in vivo study.

## Conclusions

Within the limitations of this study, the following conclusions could be drawn:

1. After the complete setting (24 hr), group FNCP had the highest surface microhardness values and had a statistically significant difference from all other groups ($P<0.05$) except group EFCP ($P>0.05$).
2. After citric acid solution immersion, surface hardness values of all groups significantly decreased ($P<0.05$) except Groups EFC and FNC.
3. For the ART technique, group FNC was suitable for restoration in patient with acid challenge conditions.

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## Declaration of Interest

The authors report no conflict of interest.

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