The effects of nanofilled resin-based coatings on the physical properties of glass ionomer cement restorative materials

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ABSTRACT

Objectives: To determine the effect of two resin-based coatings on the water sorption/solubility and colour change of three conventional and two resin modified glass ionomer cement restorative materials.
Methods: Five glass-ionomer cement (GIC) restorative materials and two nanofilled resin-based coatings were studied. Disc-shaped specimens of each material were prepared and divided into three groups, uncoated and coated with either of the two coatings tested (n = 8). Water sorption and solubility were measured in accordance with ISO 4049 and ISO 6876 instructions, respectively. For colour change, the specimens were immersed in distilled water for 24 h, then immersed for another 24 h and an extra week in the three solutions of lactic acid, coffee, and distilled water. The specimens were subjected to colour measurements, using a spectrophotometer after 24 h and 7 days of immersion. The colour change (ΔE) was calculated using a specific formula. ANOVA tests were used for data analysis.
Results: Two-Way ANOVA revealed a significant interaction between materials and coatings for the water sorption/solubility values. Fuji Bulk showed the lowest water sorption/solubility and the coated groups showed a lower mean sorption/solubility and ΔE. The ΔE varied depending on the materials and the solutions.
Conclusions: Coating of GIC restoratives reduced the water sorption/solubility and ΔE of almost all materials with a significant decline in most of the materials.
Clinical significance: Coating of GIC restorations in the oral environment with resin-based coating may protect the restorations from early water sorption/solubility and discolouration.

1. Introduction

The demand for an ideal tooth coloured restorative material as a substitute for amalgam is of great concern in dentistry. Resin composites and glass ionomer cements (GICs) were developed to accomplish this goal [1]. GICs or "glass polyalkenoate cements" are direct restorative materials, which can bond to enamel and dentin without the need for bonding agents. The ability to prevent caries by releasing fluoride over time, biocompatibility, and chemical adhesion to tooth structures are among the favorable features of these tooth-coloured amalgam substitutes [2]. Having the ability to prevent water contamination in an environment filled with moisture is essential. The setting reaction of glass ionomer cements consists of two phases; the first 10 min is the formation of the polyacrylate matrix resulting in water uptake and the second 24 h is the continuation of the acid-base reaction leading to dehydration [3]. Thus, this sensitivity to moisture

has an adverse effect on the early mechanical strength of the GIC, which limits the application of this material to Class V cavities and the sandwich technique [4,5].

In the early 1990s, resin-modified glass ionomer cement (RMGIC) was developed to overcome the shortcomings of conventional glass ionomer cement (CGIC) [6]. Although low mechanical properties inhibit these materials from being ideal alternatives of amalgam in load bearing areas [7], the application of these restorative materials has become increasingly common in children and low stress bearing areas such as Class III and Class V cavities in adults [4].

Since these types of restorations are generally placed in aesthetic zones and due to their widespread use, it is vital to maintain intrinsic colour stability and resistance to surface staining. Earlier approaches [8] have shown that GICs alike resin composites are susceptible to discolouration in various staining media. However, Bagheri et al. [9], in their study on assessing the influence of food-simulating solutions on

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susceptibility to staining of aesthetic restorative materials, reported that a GIC (Fuji IX) showed the least susceptibility to discolouration compared to the resin composites examined.

The introduction of coating agents was intended to prevent moisture contamination, reduce gingival microleakage and improve the mechanical properties of GICs [10–13]. Hence, protective agents such as copal cavity varnish, light cured bonding resins, nail varnish and petroleum jelly have been developed to protect the GIC's surface [14,15]. Moreover, some coating agents such as G-Coat Plus (G-CP) and Equia Forte Coat (EFC) (GC Co, Europe) have been marketed and claim they provide a clear, glossy surface for glass ionomers and restore the aesthetic appearance and marginal integrity when used to repair the interface between the restorations and marginal tooth structure.

While several studies suggested that the application of G-CP improves mechanical properties such as fracture toughness, flexural strength and shear punch strength of GICs [12,16–18], the effect of coating on physical properties of GICs such as water sorption and solubility is limited. Recently, Hankins and colleagues [19] applied G-CP on extracted premolars restored with Fuji IX GP Extra and found that the uncoated restorations absorbed water, whereas the coated material prevented water absorption. The authors suggested that this might have resulted in the coated restorations' being drier, thus exhibiting a higher shrinkage.

Although a number of studies have been conducted to evaluate the effect of coating on staining susceptibility of resin composites, few data are available investigating the effect of nanofilled resin-based coatings on the water sorption, solubility and colour change of GICs after immersion in food simulating solutions (FSS). Therefore, the objectives of this study were to determine the water sorption, solubility and susceptibility to staining of conventional and resin modified GICs immersed in three FSS, and to evaluate the effect of two nanofilled resin-based coatings on those properties. The null hypothesis is that the physical properties of the restorative materials are independent of the surface coating with resin-based coatings.

2. Materials and methods

Three CGICs: riva self cure – SDI Ltd. (RSC), Fuji BULK – GC Australia (FB), and Equia Forte Fil – GC Europe (EFF), two RMGICs; riva light cure – SDI Ltd. (RLC) and Fuji II LC – GC Japan (FLC)), and two nanofilled resin-based coatings; G-CP and EFC from GC Europe were used in this study (Table 1). Three FSS for colour change measurements, including 15 g/500 mL Coffee (Espresso 43; Nestlé Australia Ltd, NSW, Australia) with pH of 5.01, distilled water (DW) with pH 6.8 and 0.01 mol/L lactic acid (LA) of pH 4 were used.

For each of water sorption and solubility tests, 24 disc-shaped specimens of 10 ± 0.1 mm in diameter and 1.0 ± 0.1 mm thickness were prepared for each material. The light-cured specimens were irradiated for the recommended exposure time through Mylar strips using LED curing unit at a wavelength range of 440–480 nm and an emitting light intensity of 1500 mW/cm2 (Radii plus, SDI, Victoria, Australia). The irradiance level of the light was monitored periodically with an International Light, IL1700 radiometer (International Light Technologies; Newburyport, Massachusetts, USA) to ensure an output of at least 1200 mW/cm^2.

2.1. Water sorption test

In accordance with ISO 4049 [4], the specimens were instantly transferred to an incubator maintained at 37 °C with relative humidity not less than 95%. After 60 min, the specimens were removed from the mould and the periphery was trimmed manually against 1000 grit silicon carbide paper on a non-rotating grinding table.

The specimens of each material were randomly divided into two groups, 16 coated and eight uncoated. For the coated group of each material, eight specimens were coated with G-CP, and eight with EFC. All surfaces of the specimens were coated with the resin-based coatings in the "coated" groups. The specimens were immersed in distilled water at 37 ± 1 °C for seven days in a vertical position, with a minimum of 3 mm space in between. Four of the specimens in the uncoated EFF group showed various fractures after seven days of immersion in water. Therefore, the procedure was repeated for an extra four specimens without trimming the periphery. No fracture was observed for these specimens after immersion in water. The specimens were then removed, washed with water, air dried for 15 s and weighed on an electronic balance (GR-3000, A & D CL Toshiba, Tokyo, Japan) to an accuracy of 0.1 mg one minute after removal from water. This mass was recorded as m_1. Later, the specimens were placed in a desiccator (Labx Company, Ontario, Canada) containing freshly dried silica gel (SIGMA-ALDRICH, Taufkirchen, Germany) maintained at 37 ± 1 °C until a constant mass (m_2) was obtained.

The values for water sorption (W_{sp}) were calculated in µg/mm^3, using the following equation: $W_{sp} = (m_1-m_2)/V$; Where m_1 is the mass of the specimen after immersion in water for seven days; m_2 is the constant mass of the specimen obtained after removal from water (all in micrograms); and V is the volume of the specimen, in cubic millimeters.

2.2. Water solubility test

According to ISO 6876 [8], the filled moulds were placed in the cabinet maintained at 37 ± 1 °C with relative humidity not less than 95% for 50% longer than the setting time stated by the manufacturer.

The specimens of each material were removed from the mould and randomly divided into two groups of 16 coated and eight uncoated. For the coated group of each material, eight specimens were coated with G-CP and eight with EFC. All surfaces of the specimens were coated with the resin-based coatings in the "coated" groups. The mass of the specimens was determined using the same electronic balance to the nearest 0.1 mg. Two specimens were placed in a shallow petri dish "A" such that the surfaces did not touch the dish and they remained undisturbed. Distilled water (50 ± 1 ml) was added and the dish covered, the dish was then placed in the cabinet for 24 h. The contents of dish "A" (water together with the specimens) were poured into a fluted filter placed into a funnel 20 mm above the bottom of dish "B". The previously used dish

Table 1
Description of the materials.

Material	Manufacturer	LOT number	Type	Composition
riva self cure	SDI, Victoria, Australia	1081615F	CGIC	Fluoro-aluminosilicate glass/ Polyacrylic acid/Tartaric acid
riva light cure	SDI, Victoria Australia	1082160EG	RM-GIC	Fluoro-aluminosilicate glass/Polyacrylic acid
Fuji II LC	GC Japan	1607141	RM-GIC	Aluminium-fluoro-silicate glass/Poly-HEMA
Fuji Bulk	GC Australia	1511171	CGIC	Ultrafine highly reactive glass particles/Higher molecular weight Polyacrylic acid
Equia Forte Fil	GC Europe	1604041	CGIC	Fluoro-alumino-silicate glass/Polybasic carboxylic acid/Polyacrylic acid/Distilled water
G-Coat Plus	GC Europe	1311121	Light-cured resin coating	Urethane methacrylate/Methyl methacrylate/Camphorquinone/Silicon dioxide/Phosphoric ester monomer
Equia Forte Coat	GC Europe	1608021	Light-cured resin coating	Urethane methacrylate/Methyl methacrylate /Camphorquinone/Colloidal silica/Phosphoric ester monomer

"A" was later washed three times with 5 ml of water and the water poured into the fluted filter.

To evaporate water, dish "B", along with the collected water, was placed in an oven at (110 ± 2 °C). After which, the dish was placed in the desiccator and cooled at room temperature prior to weighing each to arrive at the constant mass. The difference between the original mass of the shallow dish "B" and its final constant mass was recorded to the nearest 0.1 mg as the amount of material removed from the specimens. The difference in mass, calculated as a percentage of the original combined mass of the two specimens, was recorded to the nearest 0.1% as water solubility.

2.3. Colour change test

A total of 360 specimens were prepared. For each material, 72 disc-shaped specimens of 10 ± 0.1 mm in diameter and 1.0 ± 0.1 mm thickness were prepared by filling a polyethelyn ring, as explained above. The specimens of each material were randomly divided into two groups, 48 coated and 24 uncoated. For the coated group of each material, 24 specimens were coated with G-CP, and 24 with EFC. All surfaces of the specimens were coated with the resin-based coatings in the "coated" groups.

All specimens were immersed at 37 °C distilled water for 24 h, then each group was divided into three subgroups of eight specimens (n = 8) and immersed in three FSS; either lactic acid, coffee and distilled water for another 24 h and for a further one week storage at 37 °C. Before each measurement, the specimens of lactic acid and coffee were rinsed with tap water for 10 s and blotted dry with tissue paper. A colour measurement was taken after each treatment for each specimen with a spectrophotometer (Spectroshade V; Vita, Bad Sackingen, Germany) against a white background. Baseline CIE values (L_0, a_0, b_0) were obtained after 24 -h immersion in distilled water. CIE values (L_i, a_i, b_i) were measured after 24 h and one week of immersion in the solutions. The differences in the lightness and chromaticity values (ΔL, Δa, Δb) were determined and the total colour change (ΔE) was calculated using the formula:

$$\Delta E_n = [(\Delta L_n)^2 + (\Delta a_n)^2 + (\Delta b_n^2]^{1/2}$$

Where n is the value calculated for ΔE measuring the differences between two treatment periods. ΔE_1 is the colour difference within the first 24 h; and ΔE_2 is the colour difference between 24 h and one-week immersion.

2.4. Statistical analyses

The data were analysed using SPSS, version 18 (SPSS Inc., Chicago, IL, USA). Kolmogorov-Smirnov test was employed for assessing normality assumption. For water sorption/solubility, two-way ANOVA was applied to investigate any interactions between materials and coatings. For colour change, three-way ANOVA was applied to find interactions between materials, coatings and FSS. For both tests, one-way ANOVA was used to compare different variables between the materials, and *post-hoc* Tukey's test was performed to show significant differences in subgroup comparisons. The level of significance was set at 0.05.

3. Results

3.1. Water sorption/solubility

The normality assumption was held in all cases. Two-way ANOVA revealed a significant interaction between the materials and coatings. The mean water sorption and solubility with standard deviations are shown in Tables 2 and 3, respectively.

Among the materials tested, FB showed the lowest water sorption compared to other GICs with or without coating, particularly when coated with EFC ($P < 0.001$). In the coated groups, water sorption of

Table 2
Mean ± SD (µg/mm^3) of water sorption of 5 glass ionomer restoratives coated with and without nanofilled resin-based coatings *(ISO: 4049)*.

Material	Coating		
	Equia Forte Coat	G-Coat Plus	Uncoated
riva light cure	99.1 ± 5.8[A,a]	79.9 ± 7.5[A,b]	105.0 ± 3.8[AB,a]
riva self cure	71. 9 ± 12.8[B,a]	59.2 ± 6.4[B,a]	61.7 ± 7.5[C,a]
Fuji II LC	82.3 ± 7.1[AB,ab]	73.1 ± 9.7[AB,b]	89.9 ± 9.0[B,a]
Fuji Bulk	35.6 ± 3.9[C,b]	54.6 ± 14.1[B,a]	59.6 ± 6.0[C,a]
Equia Forte Fil	79.1 ± 15.7[B,b]	72.7 ± 12.9[AB,b]	119.8 ± 17.9[A,a]

Different upper-case letters show significant difference between water sorption of materials in each coating (column).
Different lower case letters show significant difference between water sorption of coatings in each material (row).

Table 3
Mean ± SD (µg/mm^3) of water solubility of 5 glass ionomer restoratives coated with and without nanofilled resin-based coating *(ISO: 6876)*.

Material	Coating		
	Equia Forte Coat	G-Coat Plus	Uncoated
riva light cure	0.19 ± 0.01[B,b]	0.00 ± 0.00[C,c]	0.49 ± 0.07[B,a]
riva self cure	0.42 ± 0.05[A,a]	0.13 ± 0.04[A,b]	0.45 ± 0.18[B,a]
Fuji II LC	0.18 ± 0.06[BC,b]	0.18 ± 0.05[A,b]	0.36 ± 0.00[C,a]
Fuji Bulk	0.08 ± 0.02[C,b]	0.03 ± 0.00[BC,b]	0.23 ± 0.02[D,a]
Equia Forte Fil	0.24 ± 0.05[B,b]	0.12 ± 0.07[AB,c]	0.63 ± 0.00[A,a]

Different upper case letters show significant difference between water solubility of materials in each coating (column).
Different lower case letters show significant difference between water solubility of coatings in each material (row).

RMGIC was greater than that of the conventional GIC; the highest water sorption was observed for RLC followed by FLC, EFF, RSC and FB.

For the uncoated groups, the highest water sorption belonged to EFF (119.8 ± 17.9 µg/mm^3) followed by two RMGICs, with the least noted for FB (59.6 ± 6.0 µg/mm^3). Coating the specimens resulted in a decrease in the water sorption of almost all tested GICs, which was highly significant in the EFF group ($P = 0.001$). Between the two coating agents, G-CP generated less water sorption compared to EFC in almost all GICs. Only in the FB specimens, did EFC (35.6 ± 3.9 µg/mm^3) perform better than G-CP (54.6 ± 14.1 µg/mm^3) in reducing water sorption

The lowest water solubility was shown in FB among uncoated GICs and the highest in EFF. However, the solubility depended on the coating agents. Coating the specimens led to a significant decrease in solubility of all the tested materials ($P < 0.001$ in RLC, FB, and EFF; $P = 0.002$ in FLC) with the exception of RSC. G-CP resulted in less water solubility values compared to EFC in all tested GICs.

3.2. Colour change

For the colour change test, three-way ANOVA revealed a significant interaction among the materials, coatings and FSS. The colour change values of almost all coated and uncoated GICs showed an increase after 7 days of immersion in all three FSS. Only in the FB specimens coated with EFC immersed in DW, did the colour change value after 7 days (5.6 ± 2.1) remain similar to the colour measured after 24 h of immersion (5.9 ± 2.6). Due to the significant differences observed in the colour values after 7 days (ΔE_2) compared to the 24 h colour change (ΔE_1), the colour change of GICs in this study will be interpreted based on the colour alteration after 7 days of immersion (ΔE_2). However, all colour change values of the tested GICs for all the time intervals are presented in Tables 4–6. The colour change values in different solutions were observed to be material-dependent (Table 7).

Table 4

The Mean (SD) colour change (ΔE) of all materials with and without coating in Distilled Water for all time intervals.

Material	ΔE_1	ΔE_2	Δl_1	Δl_2	Δa_1	Δa_2	Δb_1	Δb_2
riva self cure	9.3 (3.4) [a,A]	12.5 (3.4) [a,A]	4.8	3.8	−1.4	−3.0	−3.8	−9.0
riva self cure [+GC]	3.1 (1.1) [b]	5.7 (2.1) [b]	1.6	2.6	−1.0	−1.6	−2.8	−3.4
riva self cure [+EQ]	4.5 (1.4) [b]	11.3 (3.2) [a]	1.8	2.3	−0.6	−2.7	−2.4	−10.5
riva light cure	12.1 (4.1) [a,A]	13.3 (4.6) [a,A]	6.2	4.0	−1.6	−2.1	−9.3	−11.8
riva light cure [+GC]	3.7 (1.5) [a]	9.4 (3.9) [a]	0.1	−0.1	−1.2	−1.2	−2.6	−5.8
riva light cure [+EQ]	5.9 (2.4) [a]	10.1 (2.4) [a]	4.1	2.7	−0.9	−1.8	−2.3	−9.4
Fuji ll LC	4.5 (1.3) [a,A]	8.0 (2.5) [a,A]	2.0	7.1	−1.2	−0.9	0.8	2.9
Fuji ll LC [+GC]	3.5 (2.0) [a]	5.6 (2.3) [a]	9.0	4.2	−0.9	−1.6	−1.0	−0.2
Fuji II LC [+EQ]	4.5 (1.3) [a]	5.2 (1.2) [a]	5.4	2.5	−0.5	−2.2	−0.4	−1.2
Fuji Bulk	5.9 (2.6) [a,A]	8.1 (2.1) [a,A]	5.0	2.4	1.6	0.2	0.5	0.6
Fuji Bulk [+GC]	2.2 (0.8) [a]	6.3 (1.9) [a]	8.5	6.1	1.0	0.1	−0.1	1.0
Fuji Bulk [+EQ]	5.9 (2.6) [a]	5.6 (2.1) [a]	7.3	2.3	1.7	−1.7	3.5	−3.3
Equia Forte Fil	7.5 (3.6) [a,A]	8.3 (2.3) [a,A]	6.2	−1.6	0.4	−0.4	−3.7	−2.0
Equia Forte Fil [+GC]	5.9 (1.9) [a]	9.9 (3.4) [a]	7.9	6.2	0.8	−0.8	−0.5	−3.7
Equia Forte Fil [+EQ]	6.2 (2.5) [a]	7.7 (2.2) [a]	5.4	4.3	0.7	−0.7	−0.3	−5.0

Different upper case letters show significant difference between uncoated materials (in a column).
Different lower case letters show significant difference between coatings in each material (in a column).
GC refers to the specimens coated with G-Coat Plus, and EQ refers to the specimens coated with Equia Forte Coat.

3.3. Comparison of ΔE_2 values among uncoated materials

No significant difference was found between uncoated GICs immersed in DW and LA in terms of ΔE_2 ($P = 0.403$ and $P = 0.465$). However, there was a significant difference between ΔE_2 values of uncoated GICs immersed in coffee ($P < 0.001$). Among the materials immersed in coffee, ΔE_2 of RMGICs was greater than the conventional GICs. The highest ΔE_2 in coffee was observed for FLC (16.5 ± 5.2), followed by RLC (12.6 ± 0.9), EFF (9.0 ± 1.3), FB (5.1 ± 2.9), and RSC (5.0 ± 1.5).

3.4. Comparison of ΔE_2 values among coatings

In almost all GICs kept in DW, there were no significant differences between coatings in terms of the 7 days colour change ($P = 0.635$, 0.139, 0.428, and 0.843 in RLC, FLC, FB, and EFF respectively). Only in RSC, the application of G-CP led to a significant reduction of ΔE_2 compared to EFC ($P = 0.013$) and uncoated RSC ($P = 0.003$).

Among conventional GICs immersed in coffee, no significant difference was observed between different coatings with regard to ΔE_2 ($P = 0.126$, 0.277, and 0.352 in RLC, FB, and EFF respectively). However, in the RMGICs immersed in coffee, G-CP led to a significant reduction of ΔE_2 compared to EFC (P = 0.037 and P = 0.012 in RLC and FLC, respectively). There was no significant difference among the

coatings for the specimens immersed in LA ($P = 0.665$, 0.179, 0.255, 0.124 in RLC, FLC, FB, and EFF respectively), except for RSC, in which the application of EFC led to a significant increase in ΔE_2 values compared to that of G-CP ($P = 0.045$) and the uncoated specimens ($P = 0.019$).

4. Discussion

The water sorption/solubility, surface reactivity and setting reaction are considered the effective factors on dental materials' colour susceptibility [20,21]. A large number of studies have been conducted to assess the protection ability of surface coating agents [10,15,22,23] and have recommended the application of coating agents over GIC and RMGIC restorations.

The lack of specific ISO standards for GIC is an important issue currently. These materials present quite particular characteristics. Therefore, the use of most ISO standards, which deal with polymer-based materials may not be suitable for testing GICs. However, in this study two different ISOs were employed to examine water sorption (ISO 4049) and solubility (ISO 6876), which do not hamper acid-base dental cements. ISO 4049 could not be used for glass ionomer cements' solubility measurement as it requires the primary weight of the specimens in the solubility formula. This primary mass is obtained through placing the specimens in desiccators maintained for 24 h until a constant mass

Table 5

The Mean (SD) colour change (ΔE) of all materials with and without coating in Coffee for all time intervals.

Material	ΔE_1	ΔE_2	Δl_1	Δl_2	Δa_1	Δa_2	Δb_1	Δb_2
riva self cure	3.9 (1.4) [a,AB]	5.0 (1.5) [a,C]	−1.6	3.1	−0.3	−2.4	−0.7	−2.4
riva self cure [+GC]	2.7 (1.2) [a]	4.4 (1.4) [a]	−1.5	−2.4	−0.3	−2.3	1.1	−1.7
riva self cure [+EQ]	4.1 (1.0) [a]	9.8 (4.0) [a]	−1.9	−2.2	−1.2	−4.3	1.7	−4.7
riva light cure	9.8 (3.7) [a,AB]	12.6 (0.9) [a,AB]	1.3	−7.0	0.1	1.0	9.4	9.1
riva light cure [+GC]	2.4 (0.8) [b]	3.9 (1.4) [c]	0.3	−1.8	−0.5	−0.4	0.7	1.8
riva light cure [+EQ]	5.6 (2.5) [ab]	7.3 (2.4) [b]	0.3	−2.4	−0.1	−0.9	2.7	1.3
Fuji ll LC	10.8 (4.8) [a,A]	16.5 (5.2) [a,A]	−5.3	−8.5	−1.6	−1.7	6.1	9.7
Fuji ll LC [+GC]	4.5 (1.8) [a]	8.1 (1.4) [b]	−5.5	−5.8	−1.3	−2.8	1.3	3.4
Fuji II LC [+EQ]	9.9 (3.5) [a]	16.7 (3.0) [a]	−4.1	−12.7	−0.8	−2.5	6.5	3.1
Fuji Bulk	2.8 (1.1) [a,B]	5.1 (2.9) [a,C]	−1.7	−0.6	−0.7	0.9	0.9	3.3
Fuji Bulk [+GC]	2.0 (0.7) [a]	5.2 (2.1) [a]	−2.5	−1.6	−0.4	−1.1	4.8	0.6
Fuji Bulk [+EQ]	3.0 (1.4) [a]	3.3 (1.0) [a]	−0.9	0.3	−1.0	−2.4	−0.3	−1.4
Equia Forte Fil	5.4 (2.2) [a,AB]	9.0 (1.3) [a,BC]	−2.7	−1.8	−1.2	−1.3	0.1	0.4
Equia Forte Fil [+GC]	4.6 (1.8) [a]	7.9 (2.5) [a]	−1.7	−3.1	−1.1	−3.5	1.3	−3.8
Equia Forte Fil [+EQ]	5.8 (1.5) [a]	7.4 (2.2) [a]	−2.5	−3.0	−1.0	−3.4	0.1	−3.9

Different upper case letters show significant difference between uncoated materials (in a column).
Different lower case letters show significant difference between coatings in each material (in a column).
GC refers to the specimens coated with G- Coat Plus, and EQ refers to the specimens coated with Equia Forte Coat.

Table 6
The Mean (SD) colour change (ΔE) of all materials with and without coating in Lactic Acid for all time intervals.

Material	ΔE_1	ΔE_2	Δl_1	Δl_2	Δa_1	Δa_2	Δb_1	Δb_2
riva self cure	2.0 (0.3)[b,A]	3.4 (1.5)[b,A]	−1.2	0.3	−0.3	−1.6	−0.7	−2.0
riva self cure [+GC]	1.5 (0.0)[b]	4.7 (2.5)[b]	−0.6	−1.0	−1.0	−1.9	−0.4	−2.5
riva self cure [+EQ]	5.8 (2.8)[a]	12.0 (3.1)[a]	−2.0	−3.0	−1.0	−4.5	−4.7	−10.4
riva light cure	4.7 (1.5)[a,A]	6.6 (2.7)[a,A]	0.6	0.9	−0.1	−0.1	−0.7	1.4
riva light cure [+GC]	3.5 (0.8)[a]	5.9 (2.1)[a]	1.9	3.3	−0.9	−1.4	0.4	0.5
riva light cure [+EQ]	4.5 (1.2)[a]	5.1 (2.6)[a]	−0.3	0.7	−1.2	−2.0	−2.1	−2.6
Fuji ll LC	6.3 (2.9)[a,A]	8.9 (1.8)[a,A]	−0.1	−0.5	−0.6	−1.7	−1.1	−1.7
Fuji ll LC [+GC]	4.2 (1.8)[a]	8.6 (3.4)[a]	−3.0	−1.5	−1.2	−3.2	−3.7	−4.7
Fuji II LC [+EQ]	7.3 (1.8)[a]	8.0 (3.3)[a]	−5.1	0.9	−1.0	−2.3	−8.7	−2.3
Fuji Bulk	3.9 (1.9)[a,A]	4.7 (1.2)[a,A]	−0.2	−0.6	−1.6	−1.6	−2.0	−2.5
Fuji Bulk [+GC]	3.8 (1.0)[a]	10.6 (4.7)[a]	−2.7	−4.5	−2.4	−3.2	−5.4	−8.9
Fuji Bulk [+EQ]	8.4 (2.5)[a]	8.5 (2.7)[a]	−3.4	−2.0	−2.0	−1.7	−6.5	−7.4
Equia Forte Fil	4.5 (1.1)[a,A]	6.3 (2.8)[a,A]	−1.5	−1.6	−1.3	−2.8	−1.4	−5.2
Equia Forte Fil [+GC]	2.9 (1.8)[a]	6.1 (2.2)[a]	0.3	−0.5	0.8	−1.9	−0.7	−4.8
Equia Forte Fil [+EQ]	4.8 (2.9)[a]	5.9 (2.8)[a]	−2.8	−1.4	−3.4	−4.2	−4.0	−8.5

Different upper case letters show significant difference between uncoated materials (in a column).
Different lower case letters show significant difference between coatings in each material (in a column).
GC refers to the specimens coated with G- Coat Plus, and EQ refers to the specimens coated with Equia Forte Coat.

Table 7
Levels of significance (p values) of difference in colour changes in three different FSS.

Material	Coating	solution	Solution (24 h)			Solution (7d)		
			DW	Coffee	Lactic Acid	DW	Coffee	Lactic Acid
riva self cure	G- Coat Plus	DW	–	.762	.066	–	.634	.764
		Coffee	.762	–	.224	.634	–	.974
		Lactic Acid	.066	.224	–	.764	.974	–
	Equia Forte Coat	DW	–	.960	.731	–	.922	.978
		Coffee	.960	–	.568	.922	–	.830
		Lactic Acid	.731	.568	–	.978	.830	–
	Uncoated	DW	–	.011	.001	–	.000	.000
		Coffee	.011	–	.470	.000	–	.566
		Lactic Acid	.001	.470	–	.000	.566	–
riva light cure	G- Coat Plus	DW	–	.106	.921	–	.096	.350
		Coffee	.106	–	.202	.096	–	.703
		Lactic Acid	.921	.202	–	.350	.703	–
	Equia Forte Coat	DW	–	.985	.639	–	.233	.018
		Coffee	.985	–	.737	.233	–	.364
		Lactic Acid	.639	.737	–	.018	.364	–
	Uncoated	DW	–	.802	.138	–	.983	.208
		Coffee	.802	–	.367	.983	–	.272
		Lactic Acid	.138	.367	–	.208	.272	–
Fuji II LC	G- Coat Plus	DW	–	.745	.570	–	.449	.303
		Coffee	.745	–	.954	.449	–	.953
		Lactic Acid	.570	.954	–	.303	.953	–
	Equia Forte Coat	DW	–	.128	.093	–	.000	.175
		Coffee	.128	–	.982	.000	–	.000
		Lactic Acid	.093	.982	–	.175	.000	–
	Uncoated	DW	–	.104	.686	–	.012	.478
		Coffee	.104	–	.387	.012	–	.001
		Lactic Acid	.686	.387	–	.478	.001	–
Fuji Bulk	G- Coat Plus	DW	–	.000	.013	–	.929	.353
		Coffee	.000	–	.240	.929	–	.205
		Lactic Acid	.013	.240	–	.353	.205	–
	Equia Forte Coat	DW	–	.015	.575	–	.419	.251
		Coffee	.015	–	.103	.419	–	.025
		Lactic Acid	.575	.103	–	.251	.025	–
	Uncoated	DW	–	.004	.013	–	.386	.304
		Coffee	.004	–	.847	.386	–	.984
		Lactic Acid	.013	.847	–	.304	.984	–
Equia Forte Fil	G- Coat Plus	DW	–	.018	.001	–	.868	.609
		Coffee	.018	–	.287	.868	–	.892
		Lactic Acid	.001	.287	–	.609	.892	–
	Equia Forte Coat	DW	–	.978	.958	–	.992	.640
		Coffee	.978	–	.880	.992	–	.568
		Lactic Acid	.958	.880	–	.640	.568	–
	Uncoated	DW	–	.028	.013	–	.204	.677
		Coffee	.028	–	.926	.204	–	.622
		Lactic Acid	.013	.926	–	.677	.622	–

is obtained, whereas GIC specimens need to be placed in an aqueous environment immediately following preparation in order to prevent surface crazing. Therefore, ISO 6876 was used for glass ionomers' solubility due to its compatibility with acid-base materials.

Based on the results of the present study, conventional GICs exhibited lower water sorption values compared to RMGICs with or without coating except for EFF. This result is in agreement with the findings of previous studies, which have also demonstrated that the water sorption of RMGIC is higher than that of CGIC [5,24,25]. A possible justification could be the hydrophilic nature of the poly-hydroxy ethyl methacrylate (HEMA), a significant resin component in RMGICs, which absorbs water [26]. In addition, the lower rates of water sorption by FB and RSC could be due to the variations in the structure of the hardened materials. The low rate of water sorption in RSC could be attributed to the presence of tartaric acid in its composition. This dicarboxylic acid with two carboxylate anions (R-COO-) creates a large number of crosslinks, resulting in reduced empty spaces, and consequently water influx into the material [27]. Moreover, among conventional GICs, FB showed the least water solubility values compared to others. The wide differences in formulations among various brands may be the justification for this finding.

Coating the specimens resulted in a decrease in water sorption of all tested GICs with a significant reduction in the EFF group (p = 0.001). Moreover, covering the specimens using nanofilled resin-based coatings led to a significant decrease in solubility of almost all tested materials (p < 0.05). In a study by Jevnikar et al. [28], magnetic resonance micro-imaging was applied to study the effect of surface coating on water migration into RMGICs. It was reported that the application of a surface coating (Fuji Coat LC; GC) protected the cement from water diffusion from the surface of the restoration for 48 h or longer. Moreover, the results of the present study are in line with Hankins et al.'s [19] findings in which the effect of a nanofilled resin-based coating was investigated in water absorption by teeth restored with Fuji IX GP Extra through measuring cuspal flexure. The authors proposed that the application of G-CP results in the reduction of water exchange in and out of the glass ionomer materials.

It has been reported that both water sorption and solubility of restorative materials can weaken their physical and mechanical properties [5,20,29]. The application of protecting materials on the surface of GICs seems to maintain the water balance and improve GIC's properties [10,28]. The infiltration of coating gives internal protection against crack initiation and fills the porosities, both of which may decrease water sorption and solubility. On the other hand, the self-adhesive coating bonds to GIC and provides a lamination effect, which may decrease the materials' surface energy and consequently leads to less water adsorption. Therefore, its protective effect from extrinsic water may allow complete maturation of the GIC reaction with delayed water exposure, resulting in less water sorption.

G-CP and ECF were used as the coating materials in this study. The coatings were selected from the same manufacturer to investigate the difference between their protective functions and to examine whether they are both efficient in preventing water contamination. Furthermore, the number of studies that have investigated the efficiency of ECF application on GICs is very limited. The type of coating protection used in this research showed a significant effect on water sorption and solubility of the tested materials. The differences observed between the two coatings can be attributed to their different composition, which is not disclosed in detail by the manufacturer. Based on the good performance of G-CP, it is recommended that it should be routinely placed over both CGIC and RMGIC restorations. EFC had a positive effect on reducing water sorption and solubility when used with FB.

In evaluating colour change, Standard Commission Internationale de L'Eclairage (CIE L*a*b*) was used for the three-dimensional colour measurement of the specimens. Total colour difference (ΔE) represents colour changes that an observer can recognize with the naked eye and

occurs at differences above 3.3 [11]. The results of the present study demonstrated values above 3.3 for most of the tested specimens showing their susceptibility to staining in various FSS after one or 7 days of immersion. Data presented in Tables 4–6 revealed that all uncoated materials had a visible colour change after 7 days of immersion in DW, with the highest change for RLC (13.3 ± 4.6) and the lowest for FLC (8 ± 2.5). However, immersion in coffee demonstrated less colour change on the uncoated conventional GICs compared to that of RMGICs; the least change was related to RSC (5.0 ± 1.5) and the most to FLC (16.5 ± 5.2).

Colour change values (ΔE) varied depending on the materials and FSS, in general, RMGICs revealed more colour change than the conventional glass ionomer cements. The same results have been reported by Lim et al. who investigated the degree of colour stability of conventional GICs, RMGICs and polyacid modified resin composites (PMRCs) in various environmental solutions. The authors reported that RMGICs were more susceptible to staining than the conventional GICs in 75% ethanol and 10% hydrogen peroxide solution [30]. The observed differences in staining susceptibility among various glass ionomers could be attributable to the variation in their compositions and setting reaction, which affects both water sorption and susceptibility to staining of GICs [31]. Due to their hydrophilic nature, the RMGICs tested in this experiment showed higher water sorption values, consequently resulting in a greater colour change.

As a previous study has shown [32], DW can have similar effects to artificial saliva when GICs are coated; thus, it was used as a storage medium to be compared with other FSS in our study. Specimens coated with G-CP showed more remarkable reductions in the colour change after one or seven days of immersion in DW and coffee, compared to EFC in most of the materials. It was also observed that in some materials such as RSC and FLC, after seven days of immersion in coffee, coating the specimens with EFC was not as effective as 24-hs immersion. Moreover, both coating agents did not significantly decrease the colour change of the materials after seven days of immersion in LA. A possible explanation could be the acidic effect of LA, which may lead to the dissolution of the coating materials. Similar effects of acidic beverages on coatings have been reported by Poggio et al. [33] where they evaluated the influence of acidic drinks on the bacterial adhesion of GICs. The results of this research also revealed that while the use of coating reduced adhesion in the control group (not immersed in acid), the application of acids altered its surface very quickly, raising the values of adhesion to the amount of the uncoated materials. Therefore, one may assume that coating GIC restorations with a protective resin-based coating may be beneficial in the reduction of water sorption and consequently staining susceptibility only in non-acidic media. Hence, clinicians should warn patients who are heavy drinkers of acidic beverages.

Among the coated and uncoated RSC and RLC immersed in DW, the greatest change in the colour parameters was attributed to Δb which was more negative (bluish), while for the remainder of the GICs stored in DW with or without coating, the significantly changed parameter was reported as ΔL being more positive (brighter). Between GICs immersed in coffee, the change of Δb was more significant for the coated and uncoated RLC and FLC, resulting in more positive (yellowish) chroma. However, for the other GICs tested, regardless of coating, the alteration of ΔL was more significant, causing more negative (darker) values. The major colour change parameter among the coated and uncoated GICs stored in LA was found to be the chroma (Δb) rather than the value (L) leading to more negative (bluish) results. The only exception was the G-CP coated RLC specimens, in which the alteration of ΔL was more significant.

The reason why seven days of immersion in FSS was chosen in this study was based on the calculations done by Guler et al. [34]. They considered the average time for consumption of one cup of coffee is 15 min, and among coffee drinkers, the average consumption is 3.2 cups per day. Thus, 7 days of storage simulate consumption of the drink

over 6 months.

The present study was an *in vitro* study, therefore one of the limitations is that it does not simulate clinical conditions precisely. The specimens were stored for 60 min in an incubator prior to being coated in order to follow ISO instructions. This does not exactly reproduce the clinical condition in which the GIC is coated immediately after the initial setting. Moreover, only short-term evaluation of the effect of the coatings was conducted in this research. Given the fact that a coating may be worn out and might not offer protection for long periods of time *in vivo*, further long-term studies are required.

5. Conclusion

Within the limitations of this study, the following conclusions were drawn: Coating the GIC restorations decreases water sorption and solubility of almost all materials with a significant reduction in most. G-CP is advantageous if used with all tested GICs except for FB, which performed better when using EFC. All materials were susceptible to staining by all the tested beverages, especially distilled water. Combinations of staining solution, the material, and the type of coating determine the susceptibility of GICs to staining, which was reduced following the application of G-CP.

Clinical significance

G-Coat Plus is recommended to be applied on GIC restorations, reducing water sorption/solubility and staining susceptibility, particularly in the first 24 h of application.

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Declaration of Competing Interest

None.

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