

Influence of Nanocoats on the Physicomechanical Properties and Microleakage of Bulk-fill and Resin-modified Glass Ionomer Cements: An *In Vitro* Study

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ABSTRACT

Aim: To analyze the impact of two nanocoating materials, EQUIA Forte nanocoat and universal adhesive, on flexural strength, color changes, surface roughness, and microleakage of bulk-fill and resin-modified glass ionomer cements (RM-GICs).

Materials and methods: A total of 45 specimens were prepared for each group, bulk-fill (EQUIA Forte Fil) and RM-GI (Fuji II LC) cements, according to manufacturer's instructions for flexural strength, color change, and surface roughness tests. Each group was equally subdivided into three subgroups according to coating materials used; either without a coat (negative control) or covered with EQUIA Forte coat or universal adhesive.

For the flexural strength test, 15 bar-shaped specimens were prepared using a rectangular-split Teflon mold (25 × 2 × 2 mm), then the test was conducted using a universal testing machine. Thirty disk-shaped specimens were prepared for color change and surface roughness tests using cylindrical-split Teflon mold (10 mm diameter and 2 mm height). The color change was measured using a spectrophotometer after immersion in tea infusion for seven days at room temperature. Surface roughness was examined using a profilometer after exposure to 2400 brushing cycles.

Moreover, a microleakage test was conducted in 30 teeth restored with the same restorative protocols and evaluated using a stereomicroscope. Finally, the data were statistically analyzed.

Results: EQUIA Forte nanocoat subgroups exhibited the highest flexural strength in both tested GICs compared to other subgroups (91.07 ± 7.12 MPa for RM-GIC and 51.61 ± 4.42 MPa for bulk-fill GIC). For the color change, the lowest ΔE values for bulk-fill and RM-GICs were recorded in EQUIA Forte nanocoat subgroups (2.37 ± 0.25 and 2.97 ± 0.39, respectively) with no significant difference between both groups.

The surface roughness of both GICs was significantly decreased in the coated subgroups either with EQUIA Forte coat or universal adhesive, with no significant difference between both coating agents. Also, microleakage was significantly decreased in the coated subgroups with no significant difference between the coating materials.

Conclusion: Nanocoats, especially the EQUIA Forte nanocoat, positively impact the physicomechanical properties and adaptation of bulk-fill GICs and RM-GICs.

Clinical significance: The application of nanocoats on GI restorations is highly recommended.

Keywords: Bulk-fill, EQUIA Forte, Glass ionomer, *In vitro*, Microleakage, Nanocoats, Physicomechanical, RM-GIC, Universal adhesive.

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INTRODUCTION

Currently, the management of hard tooth tissue defects is directed toward the use of microinvasive or even noninvasive protocols. This emphasizes the role of adhesive restorative materials and dictates the continuous improvement of their shortcomings. One of the most important materials that showed excellent adhesive potentials to the tooth structure is the glass ionomer cements (GICs). Additionally, its biocompatibility and fluoride release enables it to gain significant popularity among dentists.^{1,2} However, it has poor mechanical properties, short working time, long setting time, and not outstanding esthetically. With its sensitivity to hydration and dehydration, a long setting time is hazardous and threatens all GI properties.^{3,4}

From this brief overview, continuous improvement in the GI materials is demanded. High viscosity with fast-set GI is one of the breakthroughs that has been done in the GI. It is also termed as glass hybrid bulk-fill GIC that is based on an increase in the powder/liquid ratio with incorporation of high-molecular-weight (Mw) acrylic acid molecules and more reactive silicate particles. These modifications increase the degree of matrix cross-linking with subsequent improvement of the mechanical properties.^{5,6} On the contrary,

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resin modification is another way for GI reinforcement where polymerizable monomers were added to the powder and/or liquid, and it undergoes acid-base reaction and photopolymerization. Compared to the conventional type, it has less moisture sensitivity, better handling, and physicomechanical properties.

These hybrid materials showed excellent properties when they are coated with resin coating.⁷ Coating of the GI plays a vital role in protecting against hydration or dehydration during the initial setting reaction and sealing any surface defects. Moreover, it enhances its abrasion resistance, esthetic properties, and overall mechanical properties.⁸ Different coating materials are available, including varnish, petroleum jelly, and bonding agents. However, they have a transient superficial effect without deep penetration into any surface defects.

Recently, new resinous coating materials appear in the markets which are augmented by nanofillers.⁹ One of these nanocoats is known as EQUIA Forte coat which is a self-adhesive, light-cured coat that contains evenly dispersed nanofillers, 50% methyl methacrylate and 0.09% camphorquinone. Such low-viscosity nanofilled resin coating was claimed by the manufacturer to provide a tough coating layer that can evenly seal the GI surface, improving its physical and optical properties. Furthermore, the universal adhesive is used as a light polymerizable resinous coating material containing nanofillers and 10- methacryloyloxydecyl phosphate monomer.

The multifactorial oral environment represents a great challenge to all restorative materials in general and GI, precisely due to its solubility.¹⁰ Therefore, the current study aimed to evaluate the effect of using two nanocoats on color change and surface roughness of bulk-fill GIC and resin-modified GIC (RM-GIC) after being challenged with staining solution and toothbrush simulation. Also, the flexural strength and microleakage of these materials with two nanocoats were evaluated. This study's null hypothesis was that the nanocoat application, either EQUIA Forte coat or universal adhesive, does not affect the two investigated GICs' physicomaterial properties. Also, no significant difference between both nanocoating agents could be detected.

MATERIALS AND METHODS

This *in vitro* study was approved by the ethics committee of faculty of dentistry for research on human subjects or specimens (code PD-P-020-005, Al-Azhar University, Girls branch). The study was carried out at the Biomaterials and Conservative Dentistry Departments. The sample size calculation was performed using G*Power version 3.1.9.2 at $\alpha = 0.05$ and 80% power with an effect size of 1.16837 regarding the flexural strength according to Molina et al.,¹¹ 1.03719 for color change according to Ozkanoglu et al.,¹² 1.0309 for surface roughness according to Lopes et al.,¹³ and 1.169022 for

microleakage according to Tamhankar et al.¹⁴ calculations yielded a sample size of four specimens per group. However, five specimens per group were used to gain extra power for each test.

The commercial names of the materials used in this study with their specifications, compositions, and manufacturer are presented in Table 1.

SPECIMEN PREPARATION

Forty-five specimens were prepared for each GI material, bulk-fill GIC (EQUIA Forte Fil) and RM-GIC (Fuji II LC), for the flexural strength, color change, and surface roughness tests. Fifteen bar-shaped specimens were prepared for the flexural strength test using split Teflon mold having a central rectangular hole, 25 × 2 × 2 mm. For the color change and surface roughness tests, 30 disc-shaped specimens were prepared with the help of a cylindrical-split Teflon mold with a central hole (10 mm diameter and 2 mm height).

The capsules, either bulk-fill GIC or RM-GIC, were shaken to loosen the powder, the plunger was pressed to remove the diaphragm, then they were clicked once in the applicator and put in the amalgamator (Ultramat S, SDI, Australia) for 10 seconds. The mixed materials were immediately introduced into the mold supported by a glass slab and polyester strips were placed between the glass slab and the mold to prevent GI adhesion to the slab. A standardized weight (250 gm) was applied over the strip to confirm cohesive specimens without voids. According to the manufacturer's instructions, the specimens of bulk-fill GIC were left to set for 2.5 minutes, while the RM-GIC was light cured for 20 seconds from each surface using LED-curing light (Elipar™ S10, 3M ESPE, USA) with an intensity of 1200 mW/cm². Then, one surface of the specimens was polished with polishing discs (fine and superfine, Soflex; 3M ESPE, St. Paul, MN, USA) to obtain uniform surfaces. Specimens were then incubated at 37°C for 24 hours, numbered, and colored nail varnish was applied to the unfinished specimen's surface to act as a marker for the surfaces to be tested.

Microsoft Excel random generator software was used to randomly divide each material's specimens into three subgroups (five specimens/subgroup) and allocate the coating agents to each subgroup. To implement the allocation sequence, numbered, sealed envelopes were used, and the sequence was concealed till the day of the intervention and chosen by an independent coworker. The operator was blinded and unaware of the type of the tested materials. The nanocoating agents were dispensed in serially

Table 1: Commercial names, specification, composition, and manufacturer of all materials used in this study

Commercial name	Specifications	Composition	Manufacturer
EQUIA Forte Fil	Glass hybrid, self-cure bulk-fill glass ionomer cement	Powder: 95% strontium fluoroalumino-silicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid Shade: A3	GC Corporation, Tokyo, Japan
Fuji II LC	Light-cured, resin-modified glass ionomer cement	Powder 100% alumino-fluoro-silicate glass Liquid: 20–22% polyacrylic acid, 30–40% 2-hydroxyethyl methacrylate (HEMA), 5–7% trimethyl hexamethylene dicarbonate, 4–6% triethylene glycol dimethacrylate, <10% urethane dimethacrylate, initiator, <1% camphorquinone Shade: A3	
EQUIA Forte Coat LC	Nanofilled self-adhesive light-cured protective coating	40–50% methyl methacrylate, 10–15% colloidal silica, 0.09% camphorquinone, 30–40% urethane methacrylate, 1–5% phosphoric ester monomer	
Single bond universal	Nanofilled universal adhesive	10-methacryloyloxydecyl phosphate monomer, HEMA, BisGMA, DMA resins, nanofillers, photoinitiator, polyacrylic acid copolymer, itaconic acid, silane, water, ethanol	3M, ESPE, St Paul, MN, USA

numbered dappen dishes by the same independent coworker and presented to the operator.

Surface coating protocols were as follows; the first subgroup was left without coating to act as a negative control group; the second subgroup was coated with EQUIA Forte coat, and the last subgroup was coated with single-bond universal. Double coats of the EQUIA Forte coat were actively applied by a microtip applicator, left undisturbed for 10 seconds, and then light cured for 20 seconds. The same protocol was followed for the universal adhesive.

FLEXURAL STRENGTH

This test was conducted in reference to the ISO standards, ISO 9917-2.¹⁵ After 24 hours of storage of the specimens in distilled water at room temperature, the specimens' height and width were verified using a digital caliper. Three-point bending test was done using the Instron universal testing machine (model 3354, Instron Instruments, England) at a crosshead speed of 1.0 mm/minute. Flexural strength was calculated using the following equation: $FS = 3Fl/2wh^2$ where F is the force magnitude at the failure point, l is the distance between the supports (20.0 mm), w is the width of the specimen, and h is its height. Data were recorded using a computer software program (Bluehill. 3 software version 3.3).

COLOR MEASUREMENTS

Baseline specimen color was evaluated with the aid of a spectrophotometer (UV-Shimadzu 3101 PC-spectrophotometer) using a standard white background (VITA Easyshade, VITA Zahnfabrik, Germany) using the Commission Internationale de l'Eclairage $L^* a^* b^*$ (CIE-LAB) system. Measurements were made under standard lighting conditions D65, and the device was calibrated according to the manufacturer's instructions before each measurement. The color is evaluated through three axes, L^* (the degree of lightness within the specimen ranging from black (0) to white (100)), a^* (the degree of green/red color), and b^* (the degree of blue/yellow color). Each measurement was repeated three times and the mean L^* , a^* , and b^* values were recorded.

After determining the baseline color value, specimens were stored in a tea infusion at 37°C for 7 days. The infusion was prepared by adding 25 gm tea powder (Yellow Label Tea, Lipton, London) in 500 ml of distilled water and heated till boiling for 15 minutes. Then, filtration with a piece of gauze was done to remove any remnants from the infusion. The infusion was refreshed daily. After 7 days, the specimens were removed from the infusion, washed for 5 minutes in distilled water, and blotted dry with an absorbent paper. Then, secondary color measurements were conducted using the same spectrophotometer as previously described. Color difference (ΔE) between 7 days' immersion in tea infusion and baseline measurements was calculated using the following equation: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. It is worth mentioning that the values of $\Delta E \geq 3.3$ were documented to detect the color change clinically.¹⁶

SURFACE ROUGHNESS TEST

Surface roughness baseline values were assessed before the toothbrush challenge. It was measured using a profilometer (Taylor Hobson Ltd, Leicester, England) with an attached diamond needle (2 μ m radius). Measurements were conducted from the center of the specimen, using a tracing length of 4 mm, a cutoff value of 0.8 mm, and a measuring speed of 0.5 mm/s. Surface roughness

was recorded as Ra (μ m), which is the arithmetic average of the valleys and peaks of the specimen's surface. Measurements were taken three times from each specimen at three different locations and the mean Ra was calculated and used as a baseline value for each specimen.

After that, toothbrushing challenge was done with the following protocol; the uncoated surfaces of the specimens were exposed and fixed in a custom-made holder device, having a central hole with the same diameter of the specimen ± 0.5 mm, 1 mm deep to stabilize the specimen and 1 mm above the hole height. The used brush was an electric toothbrush (Oral-B, TriZone 1000, 40,000 pulsations/minute and 8800 sweeps/minute), which had extra soft nylon bristles. Its head was aligned parallel to the specimen's surface. Constant brushing force was achieved by a standardized load that was attached to the brush head.¹⁷ The followed protocol dictated the application of distilled waterdrops during the brushing time, approximately 12 minutes to achieve around 100,000 sweeps. For each subgroup, a new toothbrush was used. Roughness was recalculated after toothbrushing (2400 cycles), and the differences between before and after readings were evaluated. All the used devices were checked regularly for calibration.

MICROLEAKAGE TEST

Thirty freshly extracted human third molars were selected after being examined with magnification loupes ($\times 3.5$) to be free from caries, cracks, developmental defects, or fractures. Teeth were cleaned with a hand scaler to remove any hard or soft tissue deposits and were stored in distilled water at 4°C for a maximum of 2 weeks. Class V cavities (4 mm width \times 2 mm height \times 1.5 mm depth) were prepared on the cervical third of the buccal and lingual surfaces of the teeth with the coronal margins located in the enamel and the cervical margins located 1 mm below the cemento-enamel junction. All cavities were prepared using a round bur (Mani Dia/No. BR-41) in a high-speed handpiece under a water coolant. All cavosurface margins were kept at 90°, and the burs were changed after every five preparations. For standardization, all cavities were prepared by only one operator and the cavity dimensions were checked with a graduated periodontal probe. After cavity preparation, the teeth were randomly distributed into three subgroups for each material (five teeth/subgroup) according to the surface coating used.

Cavities were conditioned with a GC cavity conditioner for 20 seconds using a cotton pellet, rinsed, and air-dried. Then, the teeth were restored in the assigned GIC according to the manufacturer's instructions. Restorations were finished and polished using descending orders of Sof-Lex discs (3M ESPE, St. Paul, MN, USA). This was followed by applying different coats using a microtip applicator and light curing for 20 seconds as mentioned before. Restored teeth were then subjected to thermocycling at 5°C and 55°C for 500 cycles, with a dwell time of 30 seconds in each bath and a transfer time of 10 seconds.

The apical foramina of teeth were sealed with an acrylic resin and the entire tooth surface was covered with two coats of nail polish except for restoration and 1 mm around the restoration margin. The coated teeth were then immersed in 2% methylene blue dye solution (Ranbaxy Fine Chemicals Ltd, India) and incubated at 37°C for 24 hours. After removal from the dye, the teeth were thoroughly washed under tap water, dried, and mounted horizontally on an acrylic block for ease of handling on the microtome. The teeth were sectioned longitudinally through the restorations' center in a buccolingual direction with a diamond saw

Table 2: Mean values, standard deviation, standard error, confidence intervals, and level of significance for the flexural strength (MPa) results for each material and different subgroups

Materials	Mean ± SD	Std. error	95% confidence interval for mean		P-value
			Lower bound	Upper bound	
EQUIA (E)	22.9 ^f ± 2.47	1.105	19.833	25.971	0.0001*
E & EQUIA Forte nanocoat	51.61 ^d ± 4.42	1.978	46.121	57.106	
E & universal adhesive	41.65 ^e ± 3.76	1.685	36.976	46.332	
Fuji II LC (F)	71.44 ^c ± 3.17	1.418	67.505	75.377	
F & EQUIA Forte nanocoat	91.07 ^a ± 7.12	3.186	82.228	99.921	
F & universal adhesive	81.96 ^b ± 4.92	2.200	75.858	88.076	

Different small letters indicate a significant difference within the same column. * indicates a significant change in strength at $p \leq 0.05$

Table 3: Mean values, standard deviation, standard error, confidence intervals, and level of significance for the color change results (ΔE) for each material and different subgroups

Materials	Mean ± SD	Std. error	95% confidence interval of the difference		P-value
			Lower bound	Upper Bound	
EQUIA (E)	3.07 ^{a,b} ± 0.32	0.14	2.68	3.46	0.003*
E & EQUIA Forte nanocoat	2.37 ^b ± 0.25	0.11	2.05	2.69	
E & universal adhesive	2.76 ^{ab} ± 0.38	0.17	2.29	3.23	
Fuji II LC (F)	3.57 ^a ± 0.45	0.20	3.02	4.12	
F & EQUIA Forte nanocoat	2.97 ^{ab} ± 0.39	0.17	2.49	3.45	
F & universal adhesive	3.22 ^a ± 0.61	0.27	2.46	3.98	

Different small letters indicate a significant difference within the same column. * indicates a significant change in color at $P \leq 0.05$

of the hard tissue microtome under water coolant. The degree of dye penetration was examined at x40 original magnifications under the stereomicroscope (Leica MZ16FA, Leica, Wetzlar, Germany). Leakage values were determined in millimeters along the occlusal and cervical wall and then the mean value was calculated for each tooth using image analysis software.¹⁸

STATISTICAL ANALYSIS

Statistical analysis was computed using SPSS (statistical package for social sciences, IBM SPSS Statistics for Mac, version 24 software, Armonk, NY: IBM Corp, USA). Data were presented as mean and standard deviations. One-way analysis of variance was used to explore different materials' effect on the physical and mechanical property results. Post hoc comparisons using Tukey's test was used to investigate the differences between groups. The significance level was set at $p \leq 0.05$.

RESULTS

Flexural Strength Results

The analyzed results are presented in Table 2. Results showed significant differences between the tested groups at $p < 0.0001$. Regarding the coating materials, results showed that the EQUIA Forte nanocoat subgroups in both tested GICs exhibited the highest flexural strength in comparison to other tested subgroups (91.07 ± 7.12 MPa for RM-GIC and 51.61 ± 4.42 MPa for bulk-fill GIC). However, the lowest flexural strength values were recorded in the uncoated subgroups, either RM (71.44 ± 3.17 MPa) or bulk-fill GICs (22.9 ± 2.47 MPa). Regarding the type of GIC, results revealed higher flexural strength values in RM-GIC than in bulk-fill GIC, either coated or uncoated.

Table 4: Mean values, standard deviation, standard error, confidence intervals, and level of significance for the surface roughness (μm) results before and after brushing for each material and different subgroups

Materials	Surface roughness		P-value
	Before brushing	After brushing	
	Mean ± SD	Mean ± SD	
EQUIA (E)	0.40 ^a ± 0.13	1.16 ^a ± 0.29	0.008
E & EQUIA Forte nanocoat	0.33 ^a ± 0.10	0.17 ^b ± 0.05	0.035
E & universal adhesive	0.45 ^a ± 0.23	0.22 ^b ± 0.05	0.074
Fuji II LC (F)	0.49 ^a ± 0.20	1.2 ^a ± 0.44	0.022
F & EQUIA Forte nanocoat	0.26 ^a ± 0.10	0.2 ^b ± 0.03	0.322
F & universal adhesive	0.52 ^a ± 0.26	0.14 ^b ± 0.06	0.020
P-value	0.269	0.0001	

Different small letters indicate a significant difference within the same column for every brushing action (before brushing and after brushing)

Color Change Results

Color change results are presented in Table 3. Results showed significant differences between the tested groups at $p < 0.003$. The lowest ΔE values for the bulk-fill and RM-GICs were recorded in the EQUIA Forte nanocoat subgroups (2.37 ± 0.25 and 2.97 ± 0.39, respectively) with no significant difference between these two groups. Meanwhile, higher ΔE values were recorded in the universal adhesive-coated subgroups followed by the uncoated subgroups with no significant difference between the uncoated GICs.

Surface Roughness Results

Surface roughness results are presented in Table 4. Results revealed nonstatistical significant differences between all the tested groups

Table 5: Mean values, standard deviation, standard error, confidence intervals, and level of significance for the microleakage (mm) results of each material and different subgroups

Material	Mean \pm SD	Std. error	95% confidence interval for mean		P-value
			Lower bound	Upper bound	
EQUIA (E)	1.58 ^a \pm 0.16	0.073	1.379	1.786	0.0001*
E & EQUIA Forte nanocoat	0.98 ^b \pm 0.17	0.076	0.764	1.189	
E & universal adhesive	1.19 ^b \pm 0.12	0.054	1.043	1.344	
Fuji II LC (F)	1.46 ^a \pm 0.15	0.071	1.263	1.655	
F & EQUIA Forte nanocoat	0.59 ^c \pm 0.09	0.044	0.473	0.716	
F & universal adhesive	0.71 ^c \pm 0.04	0.020	0.659	0.769	

Different small letters indicate a significant difference within the same column. * indicates significant microleakage at $P \leq 0.05$

before brushing ($P < 0.269$). However, after brushing, the highest surface roughness value was recorded in the uncoated subgroups of both GICs with no significant difference between them. Meanwhile, coating with either EQUIA Forte coat or universal adhesive resulted in a significant decrease in the surface roughness of both GICs with no significant difference between both coating agents. Comparing before and after brushing for the same subgroups, results revealed a significant increase in surface roughness in both uncoated subgroups. However, a significant decrease in roughness value was recorded in bulk-fill GIC coated with EQUIA Forte nanocoat ($P = 0.035$) subgroup and in RM-GIC coated with universal adhesive ($P = 0.02$) while no significant difference was found between all other tested subgroups.

Microleakage Results

Microleakage results are presented in Table 5. Results showed significant differences between the tested groups at $P < 0.0001$. The highest leakage values were recorded in the uncoated subgroup of both materials, bulk-fill (1.58 ± 0.164 mm) and RM subgroups (1.46 ± 0.158 mm), with no significant difference between both subgroups. However, the application of coats, either EQUIA Forte coat or universal adhesive, on both types of GICs resulted in a significant decrease in microleakage with no significant difference between the two coating materials. Regardless of the coating, the RM-GIC exhibited a significantly lower microleakage value than the bulk-fill GIC.

DISCUSSION

Water plays a crucial role in proper setting of GIC and development of its optimal properties. However, its proportion should be adequately adjusted during the initial setting time to avoid excessive hydration or dehydration.¹⁹ Hydration leads to increased GIC solubility and subsequent development of weak cement with soft, easily abraded surfaces. On the contrary, dehydration leads to cracked surfaces and terrible adaptation with the cavity walls and margins. For this reason, manufacturers highly recommended application of protective covering over GIC during this sensitive setting time period. Such coating materials include petroleum jelly, cocoa butter, waterproof varnishes, and even nail varnishes. However, these types are not properly bonded to the GIC, and they are susceptible to being lost by the masticatory force or brushing process. Solving this problem is achieved by using light-cured adhesive or glazing agents which form a protective bonded coat over GIC and they cannot be easily peeled off.²⁰

Recent nanotechnology has paved the way for the fabrication of materials in the range of 0.1 to 100 nanometers (nm), which

show great reactivity.²¹ Following this technology, nanocoats were introduced in the market either in the form of universal adhesive or a specially designed coat for GIC. They are composed of nanosized colloidal silica fillers and polymer matrix. These nanocoats are properly bonded to the GIC, forming a very thin uniform layer of ~ 40 to $70 \mu\text{m}$.²² In this study, the effect of nanocoats on the physicomechanical properties and microleakage of bulk-fill and RM-GICs was evaluated.

This study revealed that the flexural strength of GICs coated with nanocoatings, either EQUIA Forte coat or universal adhesive, was significantly higher than the uncoated materials (Table 2). This could be explained by the fact that GIC is a water-based restorative material sensitive to water gain or loss, which affects the polyacid salt matrix formation and maturation. Coating the GIC protects it against this water imbalance and thus increases its flexural strength.²³ Moreover, coats can fill the voids or cracks that might arise during material preparation and the finishing step of the GIC and hence improve the resistance to fracture.⁸ Regarding the nanocoatings, especially the EQUIA Forte coat, a previous study reported that nanofillers' addition in the protective coating agent improves its flow and surface wettability and produces a uniform, tough laminated layer. This layer perfectly seals any surface voids and disperses the falling mechanical stresses, which increases the materials' flexural strength.²⁴ Regarding the universal adhesive, it affords intermediate protection and this might be attributed to the HEMA (hydroxyethyl methacrylate) content, which has great affinity toward water absorption and subsequent adverse effect on the flexural strength.

The current study also revealed that RM-GIC had a higher flexural strength than bulk-fill GIC (Table 2). This finding was in agreement with that obtained by Thongbai-on et al.²³ The authors attributed their results to the difference in viscosities between the two investigated materials. Bulk-fill GIC's high viscosity increases the air entrapped into the material even if mechanically mixed, leading to a high internal porosity that negatively affects the flexural strength. Furthermore, the dual curing nature of RM-GIC with dual cross-linking and polymerized resin network that allow plastic deformation might be other reasons for flexural strength improvement.²⁴

On the contrary, Moshaverinia et al.²⁵ showed a significant increase in the flexural strength of bulk-fill GIC. The authors attributed this result to the optimized Mw of the polyacrylic acid, which increased the polysalt bridge formation and cross-linking in the structure of the cement after setting. This structure provided a more active carboxylic group for the acid-base reaction, allowing a complete setting reaction to occur. However, this study compared different bulk-fill GICs, not including RM-GIC.

In the current study, the color change was evaluated using the CIE $L^*a^*b^*$ system, which proved its efficiency in determining minute color changes, besides its sensitivity and reproducibility.²⁶ Previous study²⁷ showed that most of the staining took place during the first-week immersion in staining solution. Thus, our specimens were immersed in a tea infusion for one week and refreshed daily and then the color change was evaluated. The clinically acceptable color change was reported to be ≤ 3.3 .¹⁶ Accordingly, all tested subgroups of the current study were found to be within the acceptable range clinically, except for the uncoated RM-GIC (3.57 ± 0.45) (Table 3).

Results showed that the EQUIA Forte coated subgroups exhibited the significantly lowest ΔE value in both investigated GICs. This might be referred to as high degradation resistance of EQUIA Forte coat at low pH values as proved by Kurinji et al.²⁸ after immersion of the EQUIA Forte Fil GIC in tea and coffee for 28 days. However, in the universal adhesive-coated subgroups, a higher ΔE value was recorded, and this could be related to the adhesive chemical composition that contains hydrophilic monomer HEMA, facilitating water sorption, hence affecting the color stability. Results also revealed a high ΔE value in the uncoated GIC subgroups (Table 3). This could be attributed to the degradation and hydrolysis of the cement surface after immersion in the solution, resulting in higher surface roughness and color change.²⁹

Another important property evaluated in this study was the surface roughness. From the clinical perspective, patient can detect roughness in the range of 0.25–0.5 μm by his/her tongue tip.³⁰ The increase in surface roughness of the restorative material leads to stains and plaque accumulation that affects negatively the optical properties as well as the durability of restorative materials.³¹ Results of the present study showed that nanocoats significantly decreased the surface roughness after brushing in both tested materials compared to the corresponding control group with no significant difference between both nanocoating agents (Table 4). This could be considered one of the advantages of incorporating nanofillers in the materials as it allows high filler loading with minimal spaces between the particles, which provides better protection to the softer matrix. In turn, it prevents filler loss and improves the hardness and abrasion resistance of the materials.^{24,32} In addition to abrading off the fillers and matrix by the same rate, leaving a relatively smooth surface.³³ This clarification was in agreement with Bagis et al.³⁴ who found a significant decrease in the restorative materials' surface roughness after applying nanofilled protective resin coating.

Microleakage is an important phenomenon that is indirectly related to the degree of adaptation of restorative materials and it is defined as microscopic passage of fluids at the tooth-restoration interface.³⁵ Also, it could be caused by polymerization shrinkage or difference in the coefficient of thermal expansion and contraction between the tooth and the restorative material, leading to secondary caries and restoration failure.²⁴

Our findings revealed a significant decrease in microleakage after nanocoating application compared to the uncoated subgroups with no difference between the two coating materials (Table 5). This might be related to the nanocoats' protective effect against moisture contamination during the initial setting besides perfect sealing of any surface or interfacial defects, thus decreasing the microleakage.²⁴ This was in agreement with the results obtained by Arthilakshmi³⁶ who reported a total absence of microleakage after application of a nanocoat on RM-GIC compared to the uncoated one.

Regarding the GICs, results showed that the RM-GIC exhibited a significantly lower microleakage value than the bulk-fill GIC (Table 5). This could be related to the dual setting reactions of RM-GIC, acid-base reaction and light polymerization reaction, that affect this cement's microstructure. The matrix is formed of metal polyacrylate salts and polymeric matrix which renders the material less moisture sensitive and thereby decreases the microleakage.³⁷ Additionally, the twofold bonding mechanism of RM-GIC could be another reason where chemical ionic bonding can occur between the carboxylic group of polyalkenoic acid and calcium ions of the tooth structure. Secondly, micromechanical bonding where the polyalkenoic acid removes the smear layer and exposes the collagen fibrils up to 1 μm depth.³⁸ This was opposed by onefold bonding mechanism; chemical bonding of the bulk-fill GIC.

Finally, the use of protective nanocoats should be emphasized and tried on a wide scale through clinical trials to obtain a strong evidence about their use.

Based on the current study results, the null hypothesis that the nanocoat application does not affect the investigated GICs' physicochemical properties was rejected, while the second hypothesis can be partly rejected as a significant difference between coating agents was detected regarding flexural strength and color change.

CONCLUSION

Following the current study circumstances, the following conclusion is evident; nanocoats, especially EQUIA Forte nanocoat, positively impact the physicochemical properties and adaptation of bulk-fill GICs and RM-GICs.

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