Original Article

How Do the Surface Coating and One-Year Water Aging Affect the Properties of Fluoride-Releasing Restorative Materials?

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Abstract

Objective: To evaluate the effects of surface coating and 1-year water aging on flexural strength, compressive strength (CS) and surface roughness of fluoride-releasing restorative materials. **Materials and Methods:** The specimens were prepared from seven materials: GCP Glass Fill (GCP), Amalgomer CR (AHL), Zirconomer (Shofu), Fuji IX GP Capsule (GC), Beautifil II (Shofu), Estelite Σ Quick (Tokuyama), and reliaFIL LC (AHL). The specimens were randomly divided into two groups for each test: surface coated with G-Coat Plus (GC) and uncoated. Each group was subdivided into two groups stored in distilled water at 37°C for 24 h and 1 year before testing (n = 10). The flexural and CS were evaluated according to ISO standards on a universal testing machine. The surface roughness was assessed with AFM. After flexural strength test, a cross-section of the coated specimens was evaluated with SEM. Data were analyzed with one-way analysis of variance, Duncan and independent t-tests (P = 0.05). **Results:** After 24 h, a significant increase was observed on the flexural and CS of Amalgomer CR, Zirconomer, and Fuji IX GP by coating ($P < 0.05$). After 1 year, the coating increased the flexural strength of Amalgomer CR and Zirconomer, and CS of GCP Glass Fill ($P < 0.05$). The coating decreased the surface roughness of GCP Glass Fill, Amalgomer CR, and Zirconomer after 1 year ($P < 0.05$). The water aging decreased the mechanical properties of glass ionomer-based materials and increased their surface roughness ($P < 0.05$). **Conclusion:** The mechanical properties and surface roughness of glass ionomer-based materials were affected by coating and water aging.

Keywords: *Compressive strength, flexural strength, glass ionomer cement, surface coating, surface roughness*

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Introduction

The glass-ionomer cements (GICs) have been widely used in dentistry due to their beneficial properties, such as biological compatibility, chemical adhesion to tooth structure, and especially fluoride release which contribute to caries preventive character.[1,2] However, some characteristics of the GICs can limit their indications for clinical use.[3] The long setting reaction time and the water sensitivity during the setting reaction may cause low mechanical properties of the GICs.[4,5] During the setting process, water has an important role for the proper maturation of GICs.[5] The initial stage, which is the clinical setting reaction, occurs within the first 10 min after mixing. The second stage, involving the release of the calcium and aluminum ions within the matrix, is a slower continuation of the acid-base reaction that lasts 24 h.[4] The material is very sensitive to water uptake at the first reaction, while the material is very susceptible to dehydration during the second step. Both water contamination and dehydration result in incomplete or inadequate maturation of GICs and thus to inferior mechanical properties.[4]

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When selecting a material to restore teeth, one of the main considerations is the mechanical properties of the material.[6] The mechanical properties of a direct restorative material need to be strong enough to withstand the forces associated with mastication and other possible loading.[7] The materials must also maintain these properties for the long term.[8,9] In laboratory conditions, the flexural and compressive strength (CS) tests are commonly used to evaluate and compare the mechanical properties of dental materials.[10,11] Moreover, surface characteristic such as surface roughness also determines the clinical quality of restorative materials.[12] The GICs have been introduced in dental practice by Wilson and Kent in the early 1970s.[1] Since then, several researches have been done to enhance their mechanical properties and to expand their clinical applications. Consequently, fluoride-releasing and glass ionomer-based materials have been recently developed. Some of these materials are the high viscosity GIC, the ceramic reinforced GIC, the zirconia reinforced GIC, and the GIC containing calcium fluorapatite nanocrystals.[13] One of the recent developments in the fluoride-releasing restorative materials has been the introduction of giomer materials. The giomer is a hybridization material of GIC and composite resin, containing surface pre-reacted glass ionomer (S-PRG) filler particles within a resin matrix.[3]

In previous studies, the resin coating has been recommended for increasing the clinical performance of glass-ionomer restoration[14,15] and the mechanical properties of GICs by preventing water contamination and dehydration.[16-19] The coating agent acts as barriers to water, so the hardening and maturation processes of GIC can take place unaffected by water uptake and water loss.[16,19] It has been reported that the self-adhesive resin coating agent provided a seal of the GIC's surface through high hydrophilicity and low viscosity.[20] It has been additionally stated that the coating agent could improve the mechanical properties by filling the surface micro porosities of the materials.[17] Reviewing the literature, there is little data on the mechanical properties of recently developed fluoride-releasing materials, and no information is available regarding the effect of resin coating and water aging on the mechanical properties and surface roughness of these materials.

Therefore, the objective of this study was to evaluate the effect of resin coating and 1-year water aging on the flexural strength, CS, and surface roughness of the fluoride-releasing materials. The null hypothesis tested was the resin coating and water aging would not affect the flexural strength, CS, and surface roughness of the materials.

Materials and Methods

Five different fluoride-releasing restorative materials were tested in this present study. The restorative materials were a glass carbomer (GCP Glass Fill; GCP, Vianen, the Netherlands), a ceramic reinforced GIC (Amalgomer CR; Advanced Healthcare Ltd, Tonbridge, UK), a zirconia reinforced GIC (Zirconomer; Shofu, Kyoto, Japan), a high-viscosity GIC (Fuji IX GP Capsule; GC, Tokyo, Japan), and a giomer (Beautifil II; Shofu, Kyoto, Japan). As control, a nano-filled composite resin (Estelite Σ Quick; Tokuyama, Tokyo, Japan) and a nano-hybrid composite resin (reliaFIL LC; Advanced Healthcare Ltd) were used. The materials are listed in Table 1 with the composition, manufacturer, and lot number. A nano-filled surface sealant agent (G-Coat Plus; GC, Lot: 1710031) were also tested.

Specimen preparation

The 40 specimens were prepared from each material for each test. The dimensions of bar-shaped specimens for flexural strength test were 25 × 2 × 2 mm. The specimens for CS test were 4 mm in diameter and 6 mm in height. The specimens for surface roughness test were 5 mm in diameter and 2-mm thickness. After the material was inserted into the teflon molds, the polyester strips (Mylar strip; SS White Co., Philadelphia, PA, USA) were pressed onto the mould surfaces with glass plates to extrude excess material and obtain a flat surface. The giomer and composite resins were polymerized through the glass plate using a LED light-curing unit (Smartlite Focus; Dentsply, Milford, DE, USA) according to the manufacturer's instructions [Table 2]. The intensity of the curing light (Smartlite Focus; Dentsply, Milford, DE, USA) was measured before and after application and the light output was never <1,000 mW/cm^2. The giomer and composite resins were placed incrementally into the mould for CS test and each 2-mm thick layer was light-cured. For GCP Glass Fill and Fuji IX GP, a capsule mixer (Silver Mix; Stomamed, Bratislava, Slovakia) was used for 10 s of mixing before application of the material. Amalgomer CR and Zirconomer were mixed within a total of 30 s according to the manufacturer's instructions [Table 2]. After the light curing and setting cycle, the specimens were removed from the molds. In order to obtain flat surface, both side of the specimens were gently polished manually with a circular motion with 1,000-grit and 1,500-grit wet silicon carbide papers. Each specimen was brief rinsed in tap water between each grit. The specimens were randomly divided into two groups for each test according to coated with G-Coat Plus and uncoated. G-Coat Plus was applied using a micro-tip applicator, and then

gently air thinned for 5 s and light cured for 20 s with the LED light curing unit according to manufacturer's instructions. Only one surface of the specimens was coated as in a clinical application. All the specimens were prepared at room temperature (21 ± 1°C) in 55% relative humidity. Each group was subdivided into two groups stored in distilled water at 37°C for 24 h and 1 year before testing. The 10 specimens were tested in each subgroup (n = 10).

Mechanical properties

The flexural strength was evaluated using three-point bending test according to the ISO 4049:2009 standard with a 20-mm span at a crosshead speed of 1 mm/min on a universal testing machine (Autograph AGS-X; Shimadzu, Kyoto, Japan). Before testing, the specimen dimensions were measured using a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). The flexural strength (FS) of the material was calculated by $FS = 3P_{max}L/(2bh^2)$, where P_{max} is the maximum load (N) on the load-displacement curve, L is the span length (mm), b is the width of the specimen (mm), and h is the thickness of the specimen (mm).

The CS was evaluated using the test method according to the ISO 9917-1:2007 standard. Before testing, the specimen dimensions were measured using the digital caliper. The test was carried out using the universal testing machine that had a crosshead speed of 1.0 mm/min. The specimens were placed with their flat ends between the plates of the testing machine, so the progressively increasing compressive load was applied along the long axis of the specimens. The maximum load applied to fracture the specimens was recorded. The CS of the material was calculated by $CS = 4P_{max}/\pi d^2$, where P_{max} is the maximum applied load (N), $\pi = 3.14$, and d is the diameter of the specimen (mm).

Surface roughness

Atomic force microscopy (AFM, ezAFM, NanoMagnetics Instruments, Ankara, Turkey) was used to determine the mean surface roughness values (Ra) of the specimens. The mean surface roughness was assessed using a Si_3N_4 tip with frequency of 1 Hz in contact mode. Three different areas were randomly selected with a scan area of 5 × 5 μm and resolution 512 × 512 pixels to obtain surface roughness values. The analysis of surface roughness values was done by NMI ezAFM v4.8.2.3 control software and the mean roughness value was determined for each specimen. Then, three-dimensional images were also acquired.

SEM analysis

After flexural strength test, a cross section of a specimen was randomly selected in each coated group for SEM analysis. All specimens were adhered with conductive carbon tape to aluminum stubs and observed under SEM (Quanta Feg 250, FEI, the Netherlands) with secondary electrons at × 500, ×1,000, and ×2,000 magnification by 20 kV.

Statistical analysis

Statistical analyses were performed with the SPSS Program, version 20.0 (Statistical Package for the Social Sciences; SPSS, Chicago, IL, USA). The Kolmogorov–Smirnov test was applied to verify if the data were normally distributed. The mean flexural strength, CS and surface roughness values of the material groups were compared using one-way analysis of variance and Duncan post-hoc tests. An independent t-test analyzed the differences in flexural strength, CS, and surface roughness values of the materials, evaluating the effect of coating and aging. Pearson correlation test was performed to investigate a possible correlation between the mechanical properties and surface roughness. The P value < 0.05 was considered statistically significant for all statistical analyses.

Results

The flexural strength and CS values were presented in Table 3. The highest flexural strength and CS were obtained with Beautifil II, Estelite Σ Quick and reliaFIL LC ($P < 0.05$). A significant increase was observed on the flexural and CS of Amalgomer CR, Zirconomer, and Fuji IX GP after 24 h when G-Coat Plus was applied ($P < 0.05$). After 1 year, the coating increased the flexural strength of Amalgomer CR and Zirconomer and the CS of GCP Glass Fill ($P < 0.05$). The water aging significantly decreased the flexural strength of GCP Glass Fill, GCP Glass Fill Coated, Amalgomer CR Coated, Zirconomer, Fuji IX GP Coated groups ($P < 0.05$). The water aging significantly decreased the CS of GCP Glass Fill and Fuji IX GP Coated groups ($P < 0.05$).

The surface roughness values were revealed in Table 4. Some superficial images obtained via the contact mode of the AFM were shown in Figure 1a-1f. There were statistically differences between the surface roughness of all materials after 24 h and 1 year ($P < 0.05$). After 24 h, the coating did not affect the surface roughness of any materials ($P > 0.05$). After 1 year, a significant difference was observed on the surface roughness of GCP Glass Fill, Amalgomer CR, and Zirconomer between coated and uncoated groups ($P > 0.05$). The water aging significantly increased the surface roughness of all materials except the composite resins ($P < 0.05$). After 1 year, the highest surface roughness was obtained with GCP Glass Fill group ($P < 0.05$).

Table 1: The composition of the materials according to the manufacturers' data

Materials	Type	Composition	Manufacturer	Lot
GCP Glass Fill	Glass carbomer	Fluoroaluminosilicate glass, nano fluoro/hydroxyapatite, polyacids	GCP, Vianen, Netherlands	71702144
Amalgomer CR	Ceramic reinforced GIC	Powder: Fluoroaluminosilicate glass, polyacrylic acid powder, tartaric acid powder, ceramic reinforcing powder. Liquid: Polyacrylic acid, distilled water	Advanced Healthcare Ltd, Tonbridge, UK	011804-81
Zirconomer	Zirconia reinforced GIC	Powder: Fluoroaluminosilicate glass, zirconium oxide, pigments Liquid: Polyacrylic acid solution, tartaric acid	Shofu, Kyoto, Japan	02160281
Fujı IX GP	High viscosity GIC	Polyacrylic acid, fluoroaluminosilicate glass, polybasic carboxylic acid	GC, Tokyo, Japan	180110A
Beautifil II	Giomer	BIS-GMA, TEGDMA, inorganic glass filler, aluminium oxide, silica, prereacted glass ionomer filler, camphoroquinone	Shofu, Kyoto, Japan	111787
Estelite Σ Quick	Nano-filled composite resin	Bis-GMA, TEGDMA, silica zirconia fillers, silica-titania fillers, photoinitiators	Tokuyama, Tokyo, Japan	E699
reliaFIL LC	Nano-hybrid composite resin	Bis-GMA, TEGDMA, fluoroboroaluminosilicate glass fillers, photoinitiators	Advanced Healthcare Ltd, Tonbridge, UK	021722-8

Bis-GMA=bisphenol A diglycidyl methacrylate; TEGDMA=triethylene glycole dimethacrylate

Table 2: The application procedures of the materials according to manufacturer instructions

Materials	Application procedure
GCP Glass Fill	Shake the capsule or tap its side on a hard surface to loosen the powder and push the plunger on a plane surface to the end of the capsule.
	Insert the capsule into a universal capsule gun and click once to standardize.
	Insert the capsule into a mixer and mix the capsule for 10-15 ss with high-frequency mixers.
	Within 15 s maximum after mixing, start to extrude the mixture directly into the preparation.
Amalgomer CR	Powder to liquid ratio 3.6/1.0 g (3.6:1.0 m/m)
	Incorporate half the powder into the liquid as quickly as possible (5-10 s) and then add the remainder and spatulate to a thick putty-like consistency.
	Total mixing time 30 s.
	Do not add powder in small increments.
Zirconomer	Powder to liquid ratio 3.6/1.0 g (3.6:1.0 m/m)
	Dispense two level scoops of powder with the measuring scoop provided onto a mixing pad, and dispense one drop of liquid separately.
	Divide the dispensed powder into 2 equal portions; introduce the first half to the dispensed liquid and mix for 5-10 s with the plastic spatula and then, add the remaining half and mix until it reaches a thick putty-like consistency.
	Mixing must be completed within a total of 30 s.
Fujı IX GP	Shake the capsule or tap its side on a hard surface to loosen the powder, and push the plunger until it is flush with the main body and hold it down for 2 s.
	Immediately set it into a mixer (or an amalgamator) and mix for 10 s (~4,000 RPM)
	The working time is 2 min from start of mixing.
	Within 10 s maximum after mixing, start to extrude the mixture directly into the preparation.
Beautifil II	Dispense the necessary amount of material from the syringe.
	Light cure for 20 s (halogen lamp) or 10 s (high-power LED light).
Estelite Σ Quick	Dispense the necessary amount of material from the syringe.
	Light cure for 20 s (halogen lamp) or 10 s (high-power LED light).
reliaFIL LC	Dispense the necessary amount of material from the syringe.
	Light cure for 30 s (halogen lamp) or 10 s (high-power LED light).

The statistically significant positive correlation was observed between flexural strength (FS) and CS of the materials regardless of coating after 24 h and 1 year, whereas the statistically significant negative correlation was found between the mechanical properties and surface roughness (Ra) of the materials. The Pearson's

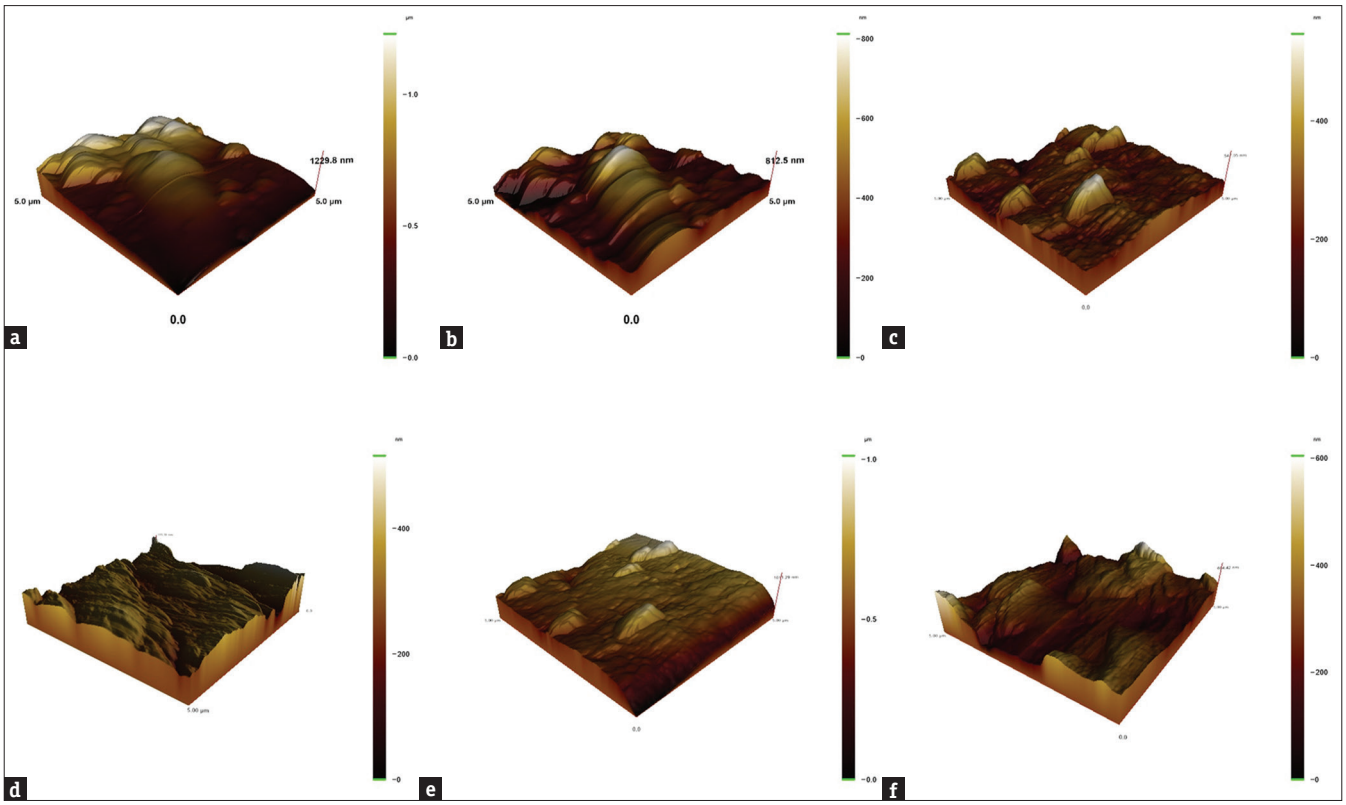

Figure 1: a. AFM image of the specimen in GCP Glass Fill Uncoated group after 1 year. b. AFM image of the specimen in GCP Glass Fill Coated group after 1 year. c. AFM image of the specimen in Amalgomer CR Uncoated group after 1 year. d. AFM image of the specimen in Amalgomer CR Coated group after 1 year. e. AFM image of the specimen in Zirconomer Uncoated group after 1 year. f. AFM image of the specimen in Zirconomer Coated group after 1 year. The topographical AFM 3D-images acquired in the contact mode from 5 × 5 mm area.

Table 3: The mean flexural and compressive strength values (MPa) of the materials with standard deviations

	Flexural strength			Compressive strength		
	24 h	1 year	$P^{\ddagger}$	24 h	1 year	$P^{\ddagger}$
GCP Glass Fill	31.27±4.18^{a}	25.60±3.94^{a}	0.002	130.82±16.03^{a}	106.90±16.42^{a}	0.008
GCP Glass Fill Coated	30.54±4.06^{a}	26.46±3.91^{a}	0.013	133.50±17.23^{a}	124.78±16.85^{b}	0.272
$P^{\dagger}$	0.698	0.630		0.723	0.027	
Amalgomer CR	35.74±5.29^{ab}	31.86±4.65^{ab}	0.161	141.58±17.40^{ab}	148.67±17.24^{c}	0.329
Amalgomer CR Coated	45.06±4.41^{cd}	41.95±5.09^{c}	0.008	158.14±17.61^{b}	147.63±17.26^{c}	0.249
$P^{\dagger}$	0.000	0.000		0.049	0.894	
Zirconomer	35.58±3.94^{ab}	33.96±3.81^{b}	0.008	140.55±17.75^{ab}	147.73±17.51^{c}	0.458
Zirconomer Coated	44.12±4.81^{c}	41.90±5.33^{c}	0.321	157.64±17.71^{b}	148.33±17.46^{c}	0.263
$P^{\dagger}$	0.000	0.001		0.045	0.940	
Fujı IX GP	41.29±4.95^{bc}	45.27±4.46^{c}	0.122	154.45±18.57^{b}	156.68±17.17^{c}	0.662
Fujı IX GP Coated	51.82±5.48^{d}	48.27±3.46^{c}	0.014	179.20±18.98^{c}	158.88±17.31^{c}	0.004
$P^{\dagger}$	0.000	0.110		0.009	0.778	
Beautifil II	114.75±10.64^{e}	111.34±10.16^{d}	0.232	248.36±22.00^{d}	245.60±21.86^{d}	0.744
Beautifil II Coated	115.51±12.08^{e}	114.17±11.38^{de}	0.729	250.14±21.44^{d}	247.36±21.48^{d}	0.795
$P^{\dagger}$	0.884	0.564		0.856	0.858	
Estelite Σ Quick	121.04±11.34^{e}	119.10±10.00^{e}	0.185	258.90±22.84^{d}	252.55±20.20^{d}	0.518
Estelite Σ Quick Coated	122.58±11.44^{e}	120.23±10.64^{e}	0.437	259.39±21.18^{d}	254.41±21.12^{d}	0.552
$P^{\dagger}$	0.766	0.810		0.961	0.843	
reliaFIL LC	117.95±11.17^{e}	116.62±11.42^{de}	0.673	250.65±21.64^{d}	248.76±21.43^{d}	0.360
reliaFIL LC Coated	117.40±11.68^{e}	114.94±11.03^{de}	0.362	251.57±21.29^{d}	248.42±19.05^{d}	0.334
$P^{\dagger}$	0.916	0.742		0.925	0.971	

Same small superscript letter indicates no statistical difference in the column. $P^{\dagger}$: Significance levels of the uncoated and coated groups of each material. $P^{\ddagger}$: Significance levels of the 24 h and 1-year groups

Table 4: The mean surface roughness values (ηm) and standard deviations of the materials

	Surface roughness		
	24 h	1 year	$P^{\ddagger}$
GCP Glass Fill	95.65±23.80^{a}	167.81±29.35^{a}	0.000
GCP Glass Fill Coated	87.23±22.10^{ab}	113.84±24.81^{bc}	0.020
$P^{\dagger}$	0.423	0.000	
Amalgomer CR	86.85±21.34^{ab}	124.23±28.49^{b}	0.018
Amalgomer CR Coated	70.15±19.19^{bc}	94.53±22.38^{cd}	0.030
$P^{\dagger}$	0.082	0.019	
Zirconomer	87.23±22.10^{ab}	125.34±28.25^{b}	0.019
Zirconomer Coated	71.93±19.78^{bc}	96.45±20.97^{cd}	0.045
$P^{\dagger}$	0.120	0.018	
Fujı IX GP	67.53±17.89^{c}	106.89±27.68^{bcd}	0.004
Fujı IX GP Coated	63.65±18.07^{c}	87.77±21.71^{de}	0.026
$P^{\dagger}$	0.635	0.103	
Beautifil II	63.55±18.25^{c}	70.33±18.82^{ef}	0.009
Beautifil II Coated	59.37±13.37^{c}	64.85±17.12^{f}	0.388
$P^{\dagger}$	0.566	0.505	
Estelite Σ Quick	59.15±15.07^{c}	63.75±16.85^{f}	0.168
Estelite Σ Quick Coated	56.33±14.15^{c}	60.55±15.67^{f}	0.509
$P^{\dagger}$	0.671	0.665	
reliaFIL LC	64.66±15.02^{cc}	67.23±16.04^{ef}	0.757
reliaFIL LC Coated	59.01±13.74^{c}	65.06±14.80^{f}	0.193
$P^{\dagger}$	0.391	0.757	

Same small superscript letter indicates no statistical difference in the column. $P^{\dagger}$: Significance levels of the uncoated and coated groups of each material. $P^{\ddagger}$: Significance levels of the 24 h and 1-year groups

correlation coefficient values were stated in Figure 2. Some SEM micrographs were presented in Figure 3a-3d. The SEM micrographs showed that there was a micro-mechanical interlocking between all the materials and the coating agent.

Discussion

In this study, the flexural strength, CS and surface roughness of the fluoride-releasing restorative materials were evaluated. The effects of surface coating and 1-year water aging on these properties of the materials were investigated. Based on the results, the surface coating and water aging influenced the properties of some fluoride-releasing materials. Therefore, the null hypothesis, that the resin coating and water aging would not affect the flexural strength, CSm and surface roughness of the materials, was partially rejected.

The setting process of GICs generally is characterized by the interaction between a polyacid liquid and a glass powder in the form of an acid-base reaction. This reaction continues by a stepwise rather long-lasting setting.[21] The changes in mechanical properties of GICs occur within the first 24 h and, the changes can be observed over several weeks or months.[5] The coating is recommended during the initial setting stage of conventional GICs for proper maturation.[5,15] The setting process of GCP Glass Fill, Amalgomer CR, Zirconomer, and Fuji IX GP occur in form of an acid–base reaction like a conventional GIC. In the present study, the surface coating significantly increased the flexural and CS of Amalgomer CR, Zirconomer, and Fuji IX GP after 24 h. As reported in the previous studies, it could be due to the fact that the coating agent exerted control on the setting process of the materials within 24 h.[15,19]

The protective effect of the coating from extrinsic water may allow complete maturation of the GIC reaction with delayed water exposure, thus possibly creating a stronger material while it may not reinforce the surface of the material.[19] Previous studies concluded that significant improvement of wear resistance,[16] shear punch strength,[19] and flexural strength[16-18] of Fuji IX GP after coating with G Coat Plus before water contamination. It has been also reported that the strength increases in coated GIC resulted from that the protective coating contributes to the GIC strength by improving the maturation process and not by the inherent strength of the coating layer.[15] In this study, the surface coating did not affect the flexural and CS of GCP Glass Fill after 24 h. It could result from different moisture sensitivity of GCP Glass Fill. According to the manufacturer, heat application is recommended for GCP Glass Fill during the setting reaction to increase its mechanical properties. But it has been concluded that the gloss and heat application with LED curing unit did not influence the flexural strength of GCP Glass Fill.[22] This result has been attributed to different chemical composition and moisture sensitivity of the material.[22] After 1 year, the coating increased the flexural strength of Amalgomer CR and Zirconomer, and the CS of GCP Glass Fill. As reported in a previous study, it could be due to that the coating agent reduced the surface porosity and crack propagation on the GICs.[19]

In this study, the glass ionomer-based materials GCP Glass Fill, Amalgomer CR, Zirconomer, and Fuji IX GP showed lower mechanical properties than Beautifil II and the composite resins regardless of coating and water aging. It has been previously reported that the giomer and composite resins had higher mechanical properties than GICs.[17,23-25] In this study, the coating did not influence the mechanical properties of the giomer and composite resins. This result can be due to the high flexural and CS of the materials. It has been stated that the coating did not play a role in materials which were more resistant to flexural stresses.[16]

Water aging is one of the most widely used procedures in experimental studies to evaluate the performance of materials and simulate the physiological aging of

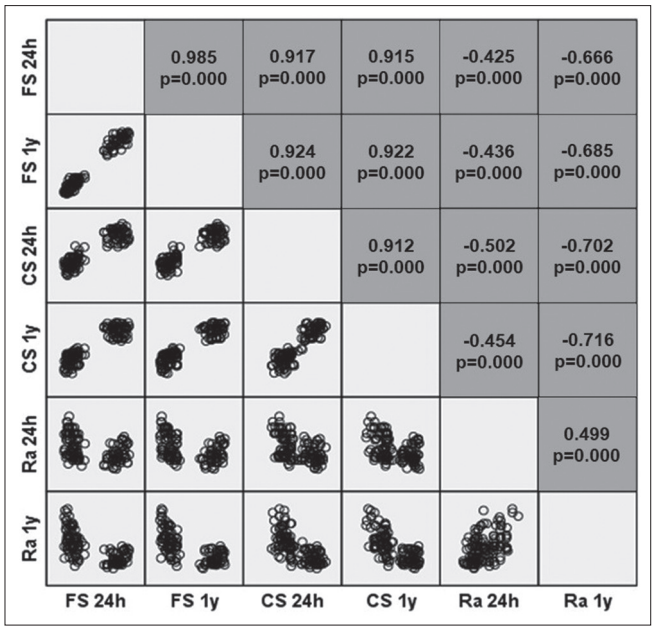

Figure 2: Pearson's correlation coefficient values for the properties of the materials. All of the correlation coefficient values displayed have statistical significance at $P < 0.01$ level. The statistically significant positive correlation was observed between flexural strength (FS) and compressive strength (CS) of the materials regardless of coating after 24 h and 1 year while the statistically significant negative correlation was found between the mechanical properties and surface roughness (Ra) of the materials

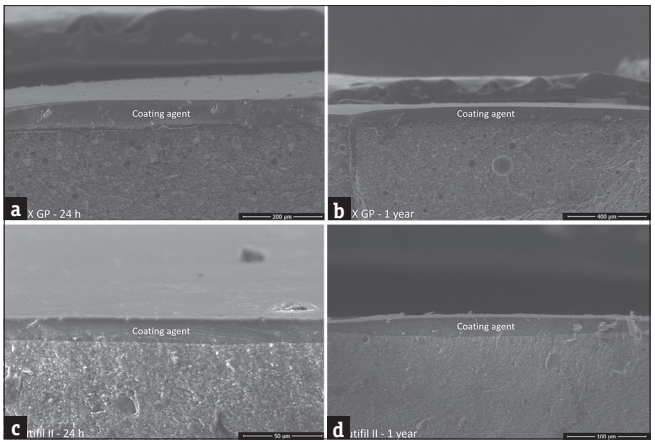

Figure 3: a. SEM photomicrograph of the cross-section of the specimen in Fuji IX GP Coated group after 24 h. b. SEM photomicrograph of the cross-section of the specimen in Fuji IX GP Coated group after 1 year. c. SEM photomicrograph of the cross-section of the specimen in Beautifil II Coated group after 24 h. d. SEM photomicrograph of the cross-section of the specimen in Beautifil II Coated group after 1 year. The photomicrographs were obtained secondary electrons mode at 20 kV. The SEM micrographs showed that there was a micro-mechanical interlocking between the material and the coating agent after 24 h and 1 year.

materials.[8] It has been stated that the storage agent had a low effect on the mechanical properties; furthermore, the storage time was a more important factor.[4,18] The water aging can cause detrimental effect on GICs based on water sorption, as it erodes the surface of the material and induces hydrolysis and dissolution of GICs' components.[26,27] The water uptake in conventional GIC is rapid due to the hydrogel structure and large micropores on the surface; thus, a substantial decrease in strength and elasticity of the material may occur.[28] The water aging can also cause plasticization of the resin component in the composite resins due to the water sorption; therefore, the long-term storage in water can influence water sorption and consequently mechanical properties of the materials.[29] However, the effects of water aging could be related to the composition of composite resins and GICs.[7,29]

A previous study has concluded that the flexural strength of Fuji IX GP showed an increase up to 3 months and then decreased after 6 months of water aging.[17] The improvement in the strength up to 3 months has been attributed to the acid–base reaction that proceeds slowly until final maturation completion which may take a few months.[30] It has been also stated that the storage time was an effective factor in the flexural strength of either uncoated or coated GICs.[17] In this study, the decrease in mechanical properties of GCP Glass Fill, Amalgomer CR, Zirconomer, and Fuji IX GP were observed after 1 year, but the water aging did not affect the mechanical properties of Beautifil II and the composite resins. As stated in a previous study, the result could be attributed to the water sorption of the materials.[7] The decrease of flexural strength on Zirconomer coated group and the CS on GCP Glass Fill coated group was not observed. It could be due to that the coating reduced water uptake. It has been reported that the coating with G Coat Plus could be beneficial for reducing water absorption of GIC.[31] But, in this study, the coating did not show the same effect for each glass ionomer-based material. The differences could result from different chemical composition and water uptake of the materials. Unfortunately, the water sorption was not evaluated in this study.

The surface roughness of restorative materials has a major effect on the discoloration and initial bacterial adhesion.[32] It has been stated that the increased surface roughness might be a predisposing factor to microbial colonization and cause a decrease in mechanical properties of the materials.[6,9,32] In this study, the negative correlation was also found between the mechanical properties and surface roughness of the materials regardless of coating and water aging. In this study, the coating did not affect the surface roughness of any materials after 24 h. The result is in agreement with a study, which concluded that the coated Fuji IX GP with G-Coat Plus showed a surface roughness similar to uncoated Fuji IX GP after 1 week.[20] In this study, the surface roughness of GCP Glass Fill, Amalgomer CR, Zirconomer, Fuji IX GP, and Beautifil II increased after

1 year. The result could be due to the water uptake of the materials. After 1 year of water aging, the microcracks could be formed on the surface of the materials. The formation of microcracks can alter the surface roughness of materials.[33] In a previous study, the microcracks on GIC surfaces were observed after aging, which could be attributed to the water uptake of glass ionomers.[8] After 1 year, the surface roughness of GCP Glass Fill, Amalgomer CR, and Zirconomer decreased by coating. As stated in a previous study, it could be due to that the coating agent reduced the surface porosity on the GICs.[19]

In this study, the SEM micrographs showed that there was still a micro-mechanical interlocking between the materials and the coating agent after 1 year, but it has been stated that the masticatory forces could cause debonding the coating agent over time in the oral environment.[15] The *in vitro* researches cannot exactly reflect the actual status of the oral cavity since the oral environment is dynamic and different from laboratory conditions. Besides the *in vitro* studies, further clinical studies are also needed to investigate the performance of the fluoride-releasing materials and the effects of resin coating.

Conclusion

Within the limitations of this study, the resin coating provided valuable support for the glass ionomer-based materials, since it led to significant improvements in flexural or CS of the materials. The giomer and composite resins had higher mechanical properties than the glass ionomer-based materials. The surface roughness of any materials was not affected by the coating after 24 h, but the coating decreased the surface roughness of some of the glass ionomer-based materials after 1 year. For the glass ionomer-based materials, the 1-year water aging caused a decrease in the mechanical properties and an increase in the surface roughness.

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Conflicts of interest

There are no conflicts of interest.

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