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Original research

Effects of surface coating on the flexural strength of fluoride-releasing restorative materials after water aging for one year

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Purpose
To evaluate the effects of surface coating and one-year water storage on the flexural strength of fluoride-releasing restorative materials.

Materials and Methods
Forty specimens were prepared from each material; 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; 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 strength was evaluated using three-point bending test according to the ISO 4049:2009 standard using a universal testing machine. After flexural strength test, a cross-section of the coated specimens was evaluated with scanning electron microscopy (SEM).

Results
A significant increase was observed on the flexural strength of Amalgomer CR, Zirconomer and Fuji IX GP after 24 h when G-Coat Plus was applied ($p<0.05$). This significant increase was observed on the flexural strength of only Amalgomer CR and Zirconomer after 1 year ($p<0.05$). The highest flexural strength was obtained with Beautifil II, Estelite Σ Quick and reliaFIL LC after 24 h and 1 year ($p<0.05$). After 1 year, there was decrease on the flexural strength of the other materials except Beautifil II, Estelite Σ Quick and reliaFIL LC.

Conclusion
The resin coating improved the flexural strength of some glass ionomer-based materials but the water aging decreased the same physical properties.

Keywords: *Flexural strength, Glass ionomer cement, Scanning electron microscopy, Surface coating, Water aging*

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 setting reactions may cause low mechanical properties of the GICs (4,5). During the setting process, water has an important role for proper maturation of GICs (5). The initial stage, which is the clinical setting reaction, occurs within the first 10 minute 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

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dehydration result in incomplete or inadequate maturation of GICs and thus to inferior mechanical properties (4).

When selecting a material to restore teeth, one of the main considerations is 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 mechanical properties for a long term (8,9). 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 (10). One of the recent developments in the fluoride-releasing restorative materials has been introduction of the 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 (11) and the mechanical properties of GICs by preventing the water contamination and dehydration (12-16). 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 (13,16). 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 (17). It has been additionally stated that the coating agent could improve the mechanical properties by filling the surface micro porosities of the materials (14). Reviewing the literature, there is little data on the mechanical properties of the recently developed fluoride-releasing materials and, no information is available regarding the effect of resin coating and water aging on the mechanical properties of these materials (3,10,12-16).

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

Materials and Methods

Restoratives

Five different fluoride-releasing restorative materials were tested in the present study. The restorative materials were a glass carbomer (GCP Glass Fill; GCP, Vianen, 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, Tonbridge, UK,) 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, Tokyo, Japan, Lot:1710031) was also tested.

Specimen preparation

The 25x2x2 mm bar-shaped forty specimens were prepared from each material. After the materials were inserted into the teflon mould, 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 below 1000 mW/cm^2. For GCP Glass Fill and Fuji IX GP, a capsule mixer (Silver Mix; Stomamed, Bratislava, Slovakia) was used for 10 seconds of mixing before application of the material. Amalgomer CR and Zirconomer were mixed within a total of 30 seconds according to the manufacturer's instructions (Table 2). After the light curing and setting cycle, the specimens were removed from the mould. In order to obtain flat surface, both side of the specimens were gently polished manually with a circular motion with 1000-grit and 1500-grit wet silicon carbide papers. Each specimen was brief rinsed in tap water between each grit. After the polishing procedure, the specimens were randomly divided into two groups according to coated with G-Coat Plus and uncoated. G-Coat Plus was applied using a micro-tip applicator, then gently air thinned for 5 seconds and light cured for 20 seconds with the LED light curing unit (Smartlite Focus; Dentsply, Milford, DE, USA, 1000 mW/cm^2) 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. The temperature and humidity were measured with a digital thermometer. Each group was subdivided into the two groups stored in distilled water at 37°C for 24 h and 1 year before testing. The ten specimens were tested in each subgroup (n=10).

Flexural strength

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).

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, Netherlands) with secondary electrons at ×500, ×1000 and ×2000 magnification by 20 kV.

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	BISGMA, 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	Before activation shake the capsule or tap its side on a hard surface to loosen the powder. For activation 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 seconds with high frequency mixers. Remove the pin from the nozzle after mixing. Insert the capsule into the capsule gun and pull the lever 2 times (2 clicks) to prime. Within 15 seconds maximum after mixing, start to extrude the mixture directly into the preparation.
Amalgomer CR	Powder to liquid ratio 3.6g /1.0g (3.6:1.0 m/m) Use a glass block for best results and a stainless steel "Silicate" spatula. Incorporate half the powder into the liquid as quickly as possible (5-10 seconds) and then add the remainder and spatulate to a thick putty-like consistency. Total mixing time 30 seconds. Do not add powder in small increments.
Zirconomer	Powder to liquid ratio 3.6g /1.0g (3.6:1.0 m/m) Dispense two level scoops of powder with the measuring scoop provided onto a mixing pad. Then, 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 second with the plastic spatula Then, add the remaining half and mix until it reaches a thick putty-like consistency. Mixing must be completed within a total of 30 second.
Fujı IX GP	Before activation, shake the capsule or tap its side on a hard surface to loosen the powder. To activate the capsule, push the plunger until it is flush with the main body and hold it down for 2 seconds. Immediately set it into a mixer (or an amalgamator) and mix for 10 seconds (~ 4,000 RPM) Immediately remove the mixed capsule from the mixer and load it into the GC Capsule Applier. Make two clicks to prime the capsule then syringe. The working time is 2 minutes from start of mixing. Within 10 seconds 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 seconds (halogen lamp) or 10 seconds (high power LED light).
Estelite Σ Quick	Dispense the necessary amount of material from the syringe. Light cure for 20 seconds (halogen lamp) or 10 seconds (high power LED light).
reliaFIL LC	Dispense the necessary amount of material from the syringe. Light cure for 30 seconds (halogen lamp) or 10 seconds (high power LED light).

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 values of the material groups were compared using one-way ANOVA and Duncan post-hoc tests. An independent t test analyzed the differences in flexural strength values of the materials, evaluating the effect of coating and aging. The p-value less than 0.05 was considered statistically significant for all statistical analyses.

Results

The flexural strength values were shown in Table 3 and graphically presented in Figure 1. The higher flexural strength values were obtained with Beautifil II, Estelite Σ Quick and reliaFIL LC than other materials after 24 h and 1 year regardless of coating ($p<0.05$). A significant increase was observed on the flexural strength 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 significantly increased the flexural strength of Amalgomer CR and Zirconomer ($p<0.05$). 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 ($p<0.05$).

Table 3. *The mean flexural strength values (MPa) and standard deviations of the materials (n=10 for each subgroups)*

	Flexural Strength		
	24 hours	***1 year***	**p‡**
GCP Glass Fill	31.27±4.18^{a}	25.60±3.94^{a}	0.002
GCP Glass Fill Coated	30.54±4.06^{a}	26.46±3.91^{a}	0.013
p†	0.698	0.630	
Amalgomer CR	35.74±5.29^{ab}	31.86±4.65^{ab}	0.161
Amalgomer CR Coated	45.06±4.41^{cd}	41.95±5.09^{c}	0.008
p†	0.000	0.000	
Zirconomer	35.58±3.94^{ab}	33.96±3.81^{b}	0.008
Zirconomer Coated	44.12±4.81^{c}	41.90±5.33^{c}	0.321
p†	0.000	0.001	
Fuji IX GP	41.29±4.95^{bc}	45.27±4.46^{c}	0.122
Fuji IX GP Coated	51.82±5.48^{d}	48.27±3.46^{c}	0.014
p†	0.000	0.110	
Beautifil II	114.75±10.64^{e}	111.34±10.16^{d}	0.232
Beautifil II Coated	115.51±12.08^{e}	114.17±11.38^{de}	0.729
p†	0.884	0.564	
Estelite Σ Quick	121.04±11.34^{e}	119.10±10.00^{e}	0.185
Estelite Σ Quick Coated	122.58±11.44^{e}	120.23±10.64^{e}	0.437
p†	0.766	0.810	
reliaFIL LC	117.95±11.17^{e}	116.62±11.42^{de}	0.673
reliaFIL LC Coated	117.40±11.68^{e}	114.94±11.03^{de}	0.362
p†	0.916	0.742	

Same small superscript letter indicates no statistical difference in the column; **p†**: Significance levels of the uncoated and coated groups of each material; **p‡**: Significance levels of the 24 hours and 1-year groups.

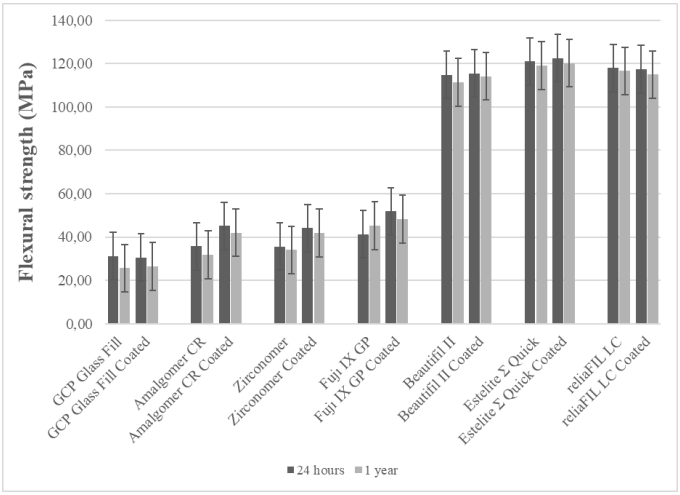

Figure 1. *The mean flexural strength values of the materials after 24 hours and 1 year.*

The SEM micrographs were presented in Figure 2. The SEM micrographs showed that there was a micro-mechanical interlocking between the materials and the coating agent after 24 h and 1 year.

Discussion

This study evaluated the flexural strength of the fluoride-releasing restorative materials and the composite resins which were commonly used as restorative materials. The effects of surface coating and one-year water aging on the flexural strength of the materials were investigated in the present study. The flexural strength test is commonly used to evaluate and compare the mechanical properties of dental materials in laboratory conditions (18-20). The flexural strength has been defined as the maximum stress that a material subjected to a bending load can resist before failure (20). It is regarded as the most important measure of strength for dental materials because considerable flexural stresses occur during the complex mastication process (18,20). The restorative materials must have high flexural strength to enhance the longevity of the restorations. (13,18). The minimum requirement of flexural strength for occlusal restorations is 80 MPa according to ISO 4049 (18). In the present study, GCP Glass Fill, Amalgomer CR, Zirconomer and Fuji IX GP did not meet the minimum requirement of ISO 4049 for occlusal restorations. The resin coating and water aging influenced the flexural strength of some fluoride-releasing materials. Therefore, the null hypothesis, that the resin coating and water aging would not affect the flexural strength of the materials, was partially rejected.

The setting process of GICs generally is characterized by interaction between a polyacid liquid and a glass powder in form of 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 a proper maturation (5,12). The setting process of GCP Glass Fill, Amalgomer CR, Zirconomer and Fuji IX GP occur in form of acid-base reac-

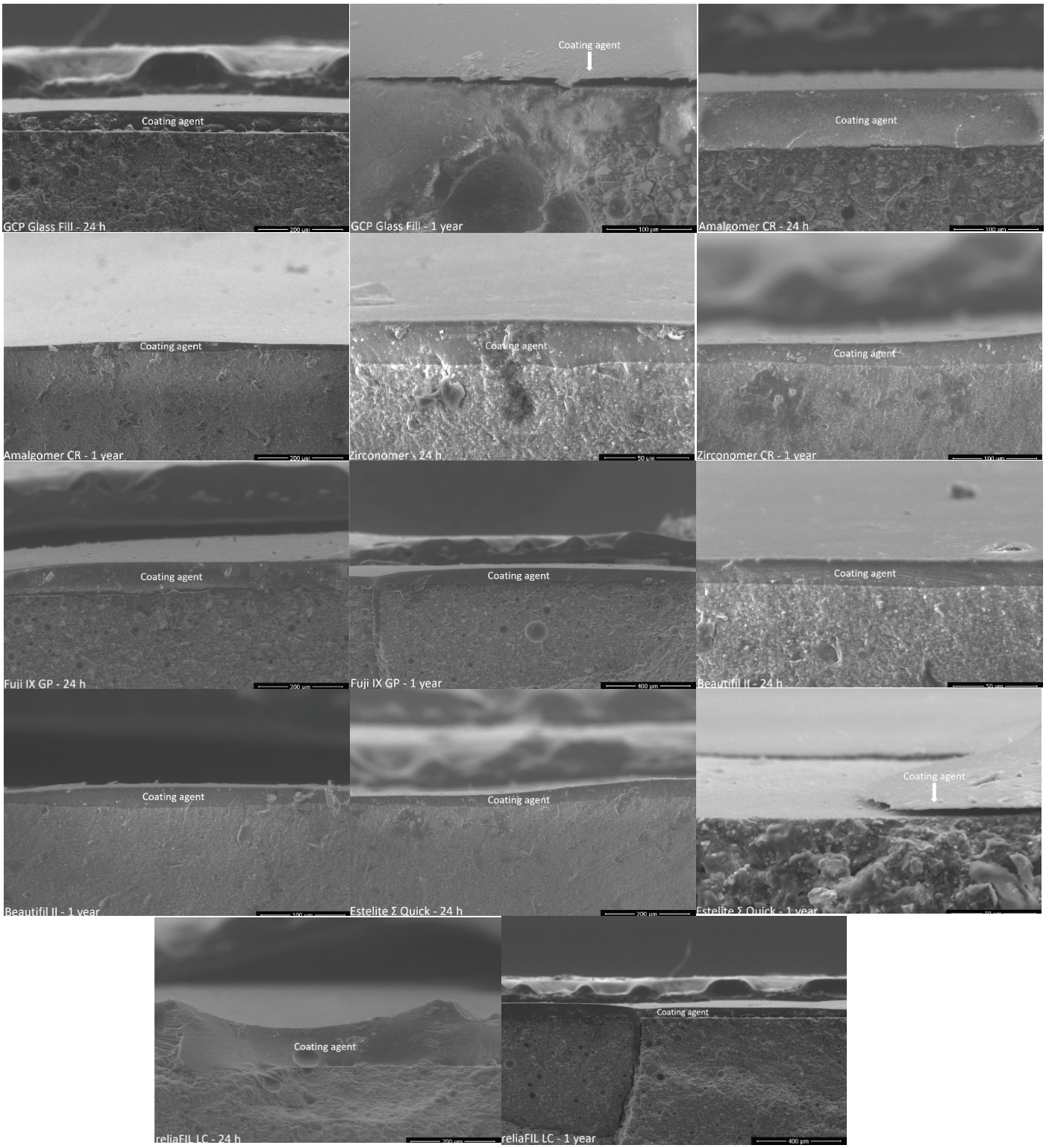

Figure 2. *SEM photomicrograph of the cross-section of the coated specimens after 24 h and 1 year. The SEM micrographs of all the materials showed that there was a micro-mechanical interlocking between the materials and the coating agent after 24 h and 1 year.*

tion like a conventional GIC. In the present study, the surface coating significantly increased the flexural strength of Amalgomer CR, Zirconomer and Fuji IX GP after 24 h. As reported in previous studies, the increase could be due to that the coating agent exerted control on the setting process of the materials within 24 h (12-16).

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 (16). Previous studies concluded that significant improvement of wear resistance (13), shear punch strength (16), and flexural strength (13-15) 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 (12). In this study, the surface coating did not affect the flexural strength 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. 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 (16).

In this study, the glass ionomer-based materials GCP Glass Fill, Amalgomer CR, Zirconomer and Fuji IX GP showed lower flexural strength 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 (14,23-25). In the present study, the coating did not influence the flexural strength of Beautifil II and the composite resins regardless of water storage. This result can be due to the high flexural strength of the materials. It has been stated that during the three-point bending test, the crack starts from within the specimen not from the surface, therefore the coating does not play a role on materials which are more resistant to flexural stresses (13).

The 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 materials (8). It has been stated that the storage agent had a low effect on the mechanical properties, furthermore the storage time was more important factor (4,15). The water aging can cause detrimental effect on GICs, 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, therefore 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 water sorption. Therefore, the long-term storage in water can influence mechanical properties of the composite resins (29). Furthermore, it has been also reported that 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 water aging (14). 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 stated that the storage time was an effective factor in the flexural strength of either uncoated and coated GICs (14). In the present study, the 1-year water aging did not affect the flexural strength of Beautifil II and the composite resins regardless of coating; however, it decreased the flexural strength values on GCP Glass Fill, Amalgomer CR, Zirconomer and Fuji IX GP. As stated in a previous study, the decrease could attribute to water uptake of the materials (7). The decrease of flexural strength was not observed on Zirconomer coated group. It could be due to that the coating can reduce 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, in this study, the water uptake was not evaluated.

In the present 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 was stated that the masticatory forces could cause debonding the coating agent over time in oral environment (12). The *in vitro* researches cannot exactly reflect the actual status of the oral cavity since oral environment is dynamic and different from laboratory conditions. But the laboratory studies simulating most clinical conditions are very useful to assess behavior of biomaterials (14,16). The longevity is one of the most important considerations of restorations (32). Therefore, the restorative materials are evaluated with *in vitro* studies to determine if they are susceptible to degradation during long-term using. 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 a valuable support for some of the glass ionomer-based materials, since it led to significant improvements in flexural strength of the materials. The giomer and composite resins had higher mechanical properties than the glass ionomer-based materials regardless of coating and water aging. The one-year water aging decreased the flexural strength of the glass ionomer-based materials while it did not affect the flexural strength of the giomer and composite resins.

Türkçe Özet: *Yüzey örtülemenin bir yıl suda yaşlandırmadan sonra florid salan restoratif materyallerin eğilme dayanımına etkisi. Amaç: Yüzey örtüleme ve bir yıl suda yaşlandırmanın florid salan restoratif materyallerin eğilme dayanımı üzerindeki etkilerini değerlendirmek. Gereç ve Yöntem: Her materyalden kırk örnek hazırlandı; GCP Glass Fill (GCP), Amalgomer CR (AHL), Zirconomer (Shofu), Fuji IX GP Kapsül (GC), Beautifil II (Shofu), Estelite Σ Quick (Tokuyama) ve reliaFIL LC (AHL). Örnekler, G-Coat Plus (GC) ile yüzey örtülenmiş ve örtülenmemiş olarak rastgele iki gruba ayrıldı. Her grup testten önce 37 °C'de distile su içinde 24 saat ve 1 yıl saklanan iki alt gruba ayrıldı (n = 10). Eğilme dayanımı, universal test cihazında ISO 4049:2009 standardına göre üç nokta eğilme testi kullanılarak değerlendirildi. Eğilme dayanımı testinden sonra, yüzeyi örtülenmiş örneklerin bir kesiti taramalı elektron mikroskobu ile değerlendirildi. Bulgular: Amalgomer CR, Zirconomer ve Fuji IX GP'nin 24 saat sonundaki eğilme dayanımında, G-Coat Plus uygulandığında önemli bir artış gözlendi (p <0.05). Bu artış, 1 yıl sonunda sadece Amalgomer CR ve Zirconomer 'in eğilme dayanımında gözlenmiştir (p <0.05). En yüksek eğilme dayanımı değerleri 24 saat ve 1 yıl sonunda Beautifil II, Estelite Σ Quick ve reliaFIL LC ile elde edildi (p <0.05). Beautifil II, Estelite Σ Quick ve reliaFIL LC hariç diğer materyallerin eğilme dayanımı 1 yıl sonunda azalmıştır (p> 0.05). Sonuç: Rezin örtüleme, bazı cam iyonomer bazlı materyallerin eğilme dayanımında artışlar sağlamıştır. Suda yaşlandırma, cam iyonomer bazlı materyallerin eğilme dayanımını azaltmıştır. Anahtar kelimeler: Cam iyonomer siman, Eğilme dayanımı, Suda yaşlandırma, Taramalı elektron mikroskobu, Yüzey örtüleme*

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Conflict of Interest: The author had no conflict of interest to declare.

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