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# The Effect of Different Coating Agents on the Microhardness, Water Sorption, and Solubility of EQUIA Forte® HT

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**Abstract:** Objective: This study aimed to assess the effect of four different surface-coating agents on the microhardness, water sorption, and solubility of the highly viscous glass ionomer cement EQUIA Forte® HT. Materials and methods: A total of 100 cylindrical EQUIA Forte® HT samples were examined, with 50 tested for Vickers hardness and the other 50 for water sorption and solubility. For each test, the specimens were divided into five groups (10 specimens/group) according to coating method: Group 1—no coating (control), Group 2—EQUIA Forte® Coat, Group 3—Single Bond™ Universal Adhesive, Group 4—ExciTE®F adhesive, and Group 5—petroleum jelly. Data were analyzed using the paired *t*-test, one-way analysis of variance, and Tukey's post hoc test for multiple comparisons. Statistical significance was set at $p < 0.05$. Results: The mean microhardness of the coated groups was significantly higher than that of the uncoated group. Moreover, a significant difference in the microhardness value was detected between the coated groups. Furthermore, EQUIA Forte® Coat had the highest mean hardness value. The mean water sorption at 7 days showed that EQUIA Forte® Coat had the lowest values. In terms of water solubility, a statistically significant difference was found between no coating and all groups except EQUIA Forte®, between Single Bond Universal Adhesive and petroleum jelly, between petroleum jelly and EQUIA Forte Coat, and between EQUIA Forte® Coat and ExciTE®F. Conclusions: The study revealed that all coating agents significantly increased the microhardness of EQUIA Forte® HT, with EQUIA Forte® Coat showing the highest hardness and the lowest water sorption values.

**Keywords:** high-viscosity glass ionomer cement; microhardness; water sorption; solubility; surface protection

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## 1. Introduction

In the late 1960s, Wilson and Kent invented glass ionomer cement (GIC), which McLean and Wilson introduced into dentistry in the 1970s [1]. Owing to the unique qualities of this bioactive substance, GIC has been used in restorative dentistry for many years. GIC is capable of chemically binding to the tooth structure, has a thermal expansion equivalent to that of dentin, and has a high capacity for remineralization [2–4]. In addition, GIC releases fluoride and acts as a reservoir for fluoride ions. However, the main disadvantages of GICs that restrict their use in high-stress areas are poor fracture resistance, poor mechanical characteristics, and moisture sensitivity during the long setting phase (first 24 h) [2,5]. Numerous efforts have been made to enhance the mechanical and physical properties of GICs without compromising their biological characteristics. This has been achieved by incorporating diverse filler materials, including montmorillonite clay, silver powder, silver amalgam alloy, zirconia, hydroxyapatite, glass fibers, phosphopeptide–amorphous calcium phosphate, bioactive glass particles, and prereacted glass ionomer particles. Furthermore, calcium phosphates have been integrated into GICs to enhance their bioactivity [6–11].

High-viscosity GIC (HVGIC) is a restorative material that has been introduced to the market in recent years owing to its improved mechanical and biomimetic features. The material was designed to compensate for the mechanical deficiencies of GICs by providing improved physical properties, such as flexural, compressive, and tensile strengths. In addition, HVGIC offers significant resistance to wear [12]. One example of HVGICs is the EQUIA® system, which was developed by GC Corporation and introduced to the market in 2009; EQUIA Forte®, the next generation of the system, was introduced in 2014. In clinical trials, both materials demonstrated excellent results in both permanent and primary teeth [13–19]. Numerous studies have evaluated the mechanical properties of the EQUIA® systems [20–23]. Moshaverinia et al. investigated the mechanical features of three GICs and discovered that EQUIA Forte® displayed the highest hardness in comparison to Fuji® IX GP and ChemFil™ Rock [20]. In another study, Savas et al. compared the water sorption and solubility characteristics of three GIC-based restorative materials (Ketac™ N100, GCP® GlassFill, and EQUIA®) and showed that EQUIA® exhibited a significantly lower water sorption capacity [22]. Aydın et al. evaluated the water sorption and solubility of different HVGIC and resin-modified GIC (RMGIC) restorations, with and without surface protection. RMGIC materials showed more water absorption than HVGICs, and EQUIA Forte® had the lowest water sorption value among the tested HVGICs. More importantly, the water absorption values of all the materials without surface protection were higher than those with surface protection [23]. The third generation, known as EQUIA Forte® HT, was released in 2019 and marketed as a strong biocompatible long-term bulk fill restorative system. These materials are enhanced by adding highly reactive particles to conventional fillers, which boost reactivity by increasing matrix crosslinking and improving the mechanical characteristics [5]. The manufacturer also introduced EQUIA Forte® Coat as an essential part of the EQUIA Forte® HT system to protect and maximize the physical properties of the restoration beneath it and consequently enhance the performance of restorative materials under oral settings.

In 1990, the American Dental Association (ADA) declared that varnishes or light-polymerized bonding agents are essential for traditional GIC restorations [24]. Applying a nano-filled resin layer to GIC is believed to decrease margin or surface imperfections while also producing a protective barrier against changes in moisture content, thereby increasing physical and mechanical qualities [14,25,26]. This recommendation is also applicable for HVGIC, which includes the recently launched EQUIA Forte® HT, which is provided with a new coating agent that has never been evaluated. Protection of the GIC surface during setting has been achieved mostly using resin-containing compounds, varnishes, cocoa butter, and petroleum gels [27–29]. A GIC surface can be fully sealed using a nano-filled resin coating with low viscosity and high hydrophilicity [24]. If the protective coat material maintains continuous contact with the GIC restoration, the probability of degradation of its mechanical properties diminishes [30]. Numerous in vitro studies have examined the effects of coating agents on the hardness of HVGICs. Other studies have evaluated the effects of coating agents on the water sorption and solubility of HVGICs [23,27,31–35]. The inconsistency of the data regarding the influence of coating agents on HVGIC and the lack of data on the newly introduced material EQUIA Forte® HT indicate the need for further investigation. Therefore, this study aimed to assess the effect of four different surface-coating agents on the microhardness, water sorption, and solubility of EQUIA Forte® HT.

## 2. Material and Methods

### 2.1. Sample Size

The sample size calculation was performed using the G*Power program (Version 3.1.9.4) at $\alpha = 0.05$ with an effect size of 0.6, and a total sample size of 50 specimens was required to achieve a power of 0.9 in each experiment.

The specimens were divided into five groups (10 specimens per group). Group 1: no coating (control), Group 2: EQUIA Forte® Coat (GC, Tokyo, Japan), Group 3: Single Bond™ Universal Adhesive (3M ESPE, St. Paul, MN, USA), Group 4: ExciTE®F (Ivoclar Vivadent, Schaan, Liechtenstein), and Group 5: petroleum jelly (Vaseline, Lever Ltd., Dubai, United Arab Emirates). The materials used in this study and their compositions are presented in Table 1.

**Table 1.** Material used in the study.

| Product | Manufacturer | Composition |
|---|---|---|
| EQUIA Forte® HT | GC, Tokyo, Japan | Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide<br>Liquid: polybasic carboxylic acid, water |
| EQUIA Forte Coat | GC, Tokyo, Japan | MMA * 25%–50%, photoinitiator 1%–5%, synergist 15%–5%, phosphoric acid ester monomer 15%–5%, BHT * <1% |
| Single Bond Universal Adhesive | 3M ESPE, St. Paul, MN, USA | MDP *, dimethacrylate resins, HEMA *, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane |
| ExciTE®F Adhesive | Ivoclar vivadent, Schaan, Liechtenstein | Phosphoric acid acrylate, HEMA *, Bis-GMA *, alcohol, di-methacrylates, silicon dioxide, initiators, stabilizers |
| Petroleum jelly | Vaseline, Unilever Ltd., Dubai, United Arab Emirates | Mineral oils, paraffin, microcrystalline waxes |

* MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, MMA: methyl methacrylate, BHT: butylated hydroxytoluene, Bis-GMA: bisphenol A-glycidyl dimethacrylate.

### 2.2. Specimen Preparation

The specimens were prepared using custom-made disc-shaped stainless-steel molds (10 mm diameter and 2 mm thickness). EQUIA Forte® HT was mixed in an automixer (Ultramat2, SDI, Bayswater, Australia) according to the manufacturer's instructions and then injected directly into the mold. Strip bands were placed on both sides of each disk and sandwiched between two glass plates. The specimens were allowed to set for 5 min. The samples were then removed from the molds, cleaned, and embedded in polyvinyl chloride (PVC) rings to facilitate handling during polishing. Subsequently, sequential silicon carbide papers (#400, 600, 800, and 1200 grit) were used with an automated polishing machine (Automata, Jeanwirtz, Dusseldorf, Germany) for surface standardization.

The surface-coating agents for Groups 2, 3, and 4 were applied to the samples as a single layer using a microbrush (Ultrabrush®, Microbrush, Grafton, WI, USA) according to the manufacturer's instructions. In Group 5, a thin layer of Vaseline was applied using a Q-tip.

### 2.3. Microhardness

The specimens were labeled, stored, and tested after being immersed for 24 h in distilled water at 37.0 °C. After the immersion process, the specimens of Groups 2, 3, and 4 were polished to obtain a flat polished surface of the material without any coating agents. The specimens were subjected to Vickers hardness testing (NOVA130, INNOVATEST, Maastricht, The Netherlands) with an indentation time of 15 s and a load of 300 g.

### 2.4. Water Sorption and Solubility

Volumes were calculated using digital calipers (Ultra Cal Mark III; Fowler Sylvac, Moutier, Switzerland). The volume (V) of each specimen was calculated in cubic millimeters, as follows:

$$V\ (\text{mm}^3) = \pi r^2\ h$$

r: mean sample radius (diameter/2), and h: mean sample thickness.

The samples were then placed in a desiccator for 24 h until a constant weight was obtained. After 24 h, the samples were weighed with a digital scale (AS 220/C/2, Radwag, Radom, Poland) with a sensitivity level of 0.0001 g and recorded as M1 (µg). After these operations, samples were kept in distilled water and incubated in an oven (JSGI-150T, JSR, Yongin City, Republic of Korea) for 24 h. Then, the samples were removed from the solution and dried with blotting paper (Kimwipes E-L, Kimberly-Clark, Dallas, TX, USA), and the weight measurements were repeated and recorded as M2a. Subsequently, the samples were kept in an oven, and the distilled water was changed every 24 h until a constant weight was obtained (7 days). At the end of the study period, weight measurements were repeated and recorded (M2b). The samples were then placed in a desiccator again and weight measurements were repeated after 24 h (M3). For each group, the means and standard deviations ($\mu g/mm^3$) of water sorption (WSO) and solubility (WSL) were calculated using the following formulas:

$$\text{WSO (a)}\ (\mu g/mm^3) = \frac{M2a - M3}{V} \quad \text{WSO (b)}\ (\mu g/mm^3) = \frac{M2b - M3}{V}$$

$$\text{WSL}\ (\mu g/mm^3) = \frac{M1 - M3}{V}$$

WSO (a): water sorption 24 h after immersion, WSO (b): water sorption 7 days after immersion, M1: specimen mass after desiccation (µg), M2a: specimen mass 24 h after immersion (µg), M2b: specimen mass 7 days after immersion (µg), M3: specimen mass after second desiccation (µg), and V: specimen volume before immersion ($mm^3$).

### 2.5. Statistical Analysis

Data were analyzed using SPSS version 24 (IBM Inc., Chicago, IL, USA). The Shapiro–Wilk test of normality was used first and showed a normal distribution of the data. One-way analysis of variance (ANOVA) with Tukey's post hoc test was used for multiple comparisons between groups, and a paired *t*-test was used for water sorption at 24 h and 7 days for the same group. Statistical significance was set at $p < 0.05$.

## 3. Results

### 3.1. Microhardness

The mean and standard deviation of the microhardness values of the experimental groups are presented in Table 2. EQUIA Forte® Coat demonstrated the highest mean microhardness value (108.3 ± 1.2), followed by ExciTE®F (99.4 ± 2.2), Single Bond™ Universal Adhesive (94.8 ± 1.0), and petroleum jelly (89.0 ± 1.2), and the lowest was observed with no coating (85.6 ± 0.2). A statistically significant difference was detected between the groups ($p < 0.001$) (Figure 1).

**Table 2.** Comparison of the mean microhardness values with different coating agents.

| | **Mean ± Std. Deviation (HV)** | **Minimum** | **Maximum** | ***p* Value** |
|---|---|---|---|---|
| No Coating | 85.6 $^a$ ± 0.2 | 85.3 | 85.9 | <0.001 * |
| EQUIA Forte Coat | 108.3 $^a$ ± 1.2 | 106.5 | 110.5 | |
| Single Bond Universal Adhesive | 94.7 $^a$ ± 1.0 | 93.3 | 96.3 | |
| ExciTE®F | 99.4 $^a$ ± 2.2 | 97.1 | 104.2 | |
| Petroleum Jelly | 89.0 $^a$ ± 1.2 | 87.5 | 90.6 | |

* ANOVA test showed statistically significant differences between the groups. The Tukey post hoc showed statistically significant differences between groups with similar small letters.

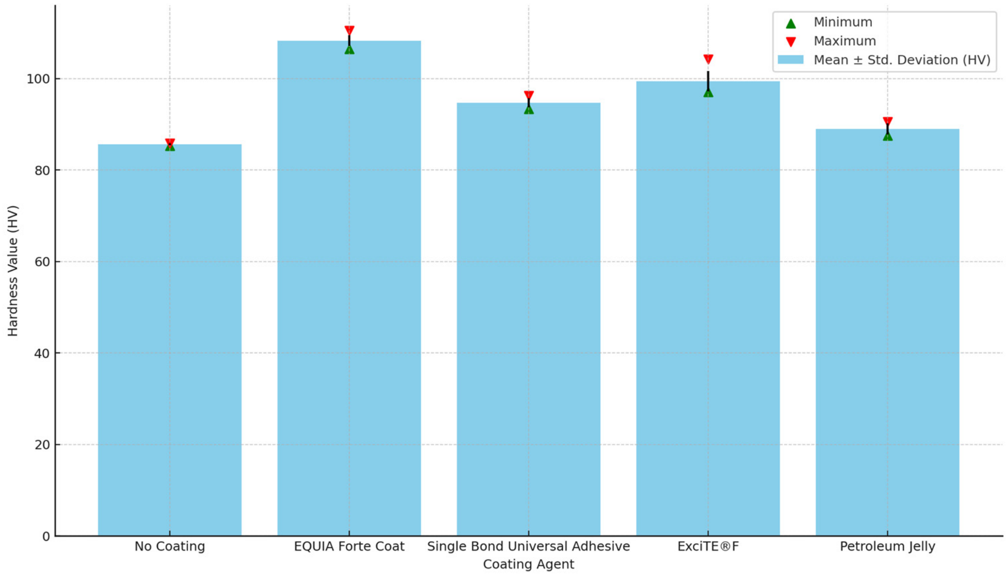

**Figure 1.** Mean microhardness values with different coating agents.

### *3.2. Water Sorption*

The means and standard deviations of the water sorption values are presented in Table 3. After 24 h, the EQUIA Forte Coat group showed the least water sorption, followed by ExciTE®F, petroleum jelly, and Single Bond Universal Adhesive. The uncoated group had the highest mean water sorption value, which was significantly higher than that of EQUIA Forte® Coat ($p < 0.05$). The mean water sorption at 7 days showed that EQUIA Forte® Coat had the lowest value followed by petroleum jelly, no coating, ExciTE®F, and Single Bond Universal Adhesive. A statistically significant difference was observed only between the Single Bond Universal Adhesive and EQUIA Forte Coat ($p < 0.05$). The differences in mean water sorption values between 24 h and 7 days for all groups were statistically significant ($p < 0.001$), except for petroleum jelly ($p = 0.9$). The water sorption values were higher at 7 days with Single Bond Universal Adhesive, EQUIA Forte® Coat, and ExciTE®F. On the other hand, the water sorption values were higher at 24 h with no coating and petroleum jelly (Figure 2).

**Table 3.** Comparison of the mean water sorption values between 24 h and 7 days.

| | **24 h** | **7 days** | |
|---|---|---|---|
| | **Mean ± Std. Deviation (μg/mm$^3$)** | **Mean ± Std. Deviation (μg/mm$^3$)** | ***p* Value *** |
| No Coating | 108.5 ± 6.8 [a] | 95.9 ± 6.8 | <0.001 * |
| EQUIA Forte Coat | 74.4 ± 11.8 [a] | 91.5 ± 8.1 [b] | <0.001 * |
| Single Bond Universal Adhesive | 98.8 ± 16.8 | 109.2 ± 15.6 [b] | <0.001 * |
| ExciTE®F | 95.3 ± 7.8 | 103.6 ± 9.3 | <0.001 * |
| Petroleum Jelly | 95.5 ± 41.1 | 93.3 ± 21.0 | 0.9 * |
| *p* value ** | 0.015 ** | 0.024 ** | |

* Measured by paired *t*-test. ** Measured by ANOVA test. The Tukey post hoc showed statistically significant differences between groups with similar small letters.

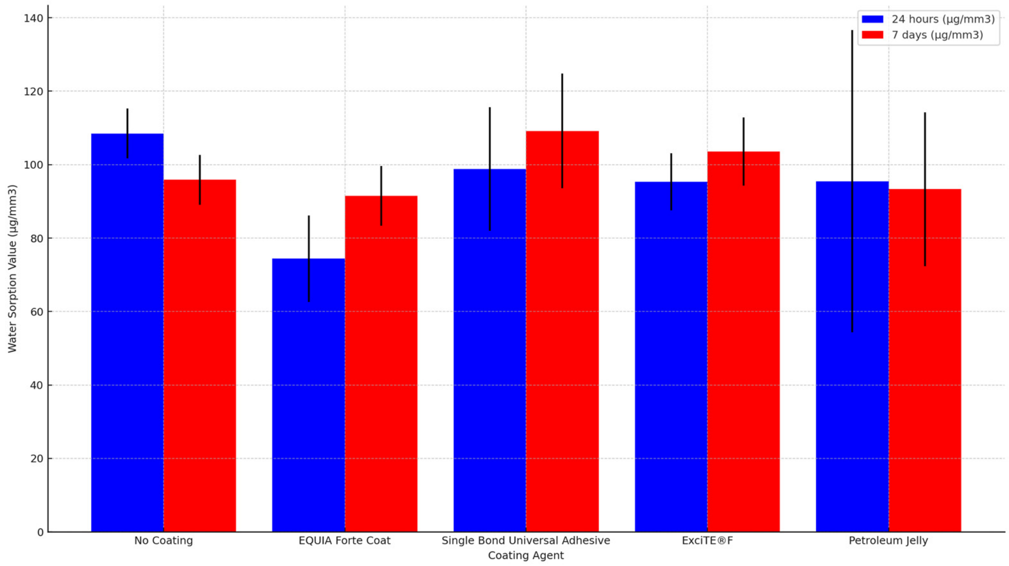

**Figure 2.** Mean water sorption values between 24 h and 7 days.

### 3.3. Water Solubility

The means and standard deviations of the water solubility values are shown in Table 4. A statistically significant difference was found between no coating and Single Bond Universal Adhesive, no coating and petroleum jelly, no coating and ExciTE®F, Single Bond Universal Adhesive and petroleum jelly, petroleum jelly and EQUIA Forte® Coat, and EQUIA Forte® Coat and ExciTE®F ($p < 0.001$).

**Table 4.** Comparison of the mean water solubility values with different coating agents.

| | **Mean (µg/mm$^3$)** | **Std. Deviation** | **Minimum** | **Maximum** |
|---|---|---|---|---|
| No Coating | $-43.0$ $^{a,b,c}$ | 21.5 | $-100.6$ | $-19.7$ |
| Single Bond Universal Adhesive | $-12.2$ $^{a,d}$ | 6.3 | $-28.0$ | $-5.7$ |
| Petroleum Jelly | 18.4 $^{b,d,e}$ | 12.1 | 7.0 | 43.9 |
| EQUIA Forte Coat | $-20.6$ $^{e,f}$ | 40.0 | $-133.1$ | 2.5 |
| ExciTE®F | 9.0 $^{c,f}$ | 5.3 | 0.0 | 17.2 |
| $p$ value | <0.001 * | | | |

* Measured by ANOVA test. The Tukey post hoc showed statistically significant differences between groups with similar small letters.

## 4. Discussion

The hardness of a material depends on its resistance to permanent surface indentations and penetrating forces. Additionally, hardness affects the ease of cutting, finishing, and polishing of a material as well as its resistance to in-service scratching [36]. In general, two hardness tests are performed for dental materials: the Vickers test for small, rounded sections and the Knoop test for small, elongated sections, which are also utilized to study the structure of enamel and dentine [37]. A significant issue for the Knoop test is the necessity for a highly polished flat surface, which requires prolonged time and attention compared to other tests. The Vickers hardness test was used in this study to determine the hardness. This process involved applying a square base at an angle of 136° between opposing sides with a certain force to the surface of the material sample, forming a trace. The hardness values derived from the materials exhibited an inverse relationship with the size at this point [38].

Several studies have investigated the effects of coating agents on the hardness of HVGIC. Brito et al. compared the effects of different coating agents on the hardness of HVGIC and concluded that petroleum jelly was a superior coating agent [27]. Dulsamphan et al. discovered that coating agents such as Ketac Glaze and G-Coat Plus significantly increased the hardness of various HVGICs [39]. Handoko et al. determined that nano-filled resin coatings significantly improved the hardness of EQUIA® Forte surfaces [32]. Faraji et al. and Ryu et al. established that surface coatings with nano-filled resins decreased the surface hardness compared with uncoated specimens, contradicting the manufacturer's statement [33,34]. In this study, all the coating agents resulted in increased microhardness compared to that exhibited by the uncoated samples, which is consistent with the ADA recommendations and previous studies [24,27,32,39]. However, studies by Faraji et al. and Ryu et al. contradicted these findings, indicating that the microhardness of uncoated materials was higher than that of coated materials [33,34]. This can be attributed to the variable thickness of the coating layer, which is often softer than the underlying material. Therefore, in this study, the specimens were subjected to a polishing procedure after immersion in distilled water for 24 h to ensure that the surfaces of the materials were tested.

In this study, applying EQUIA® Forte Coat resulted in the highest hardness compared to that produced by other groups. This result is consistent with those of the previous study by Handoko et al. who reported that applying the nano-filled coating improved the hardness of EQUIA® Forte [32]. The mechanical properties of the restorative materials were enhanced by increasing the filler content [40,41]. Thus, the elevated hardness levels detected in the EQUIA Coat group may be related to this phenomenon.

ExciTE®F, characterized as a fluoride-releasing adhesive, demonstrated superior performance in this study compared to those of the Single Bond™ and petroleum jelly, both of which are frequently employed as coating agents in dental practices. The primary advantage of GIC lies in its ability to release and recharge fluoride, which is crucial for preventing caries' recurrence and demineralization of the tooth structure. Notably, previous research has demonstrated that the application of coating agents decreases the amount of fluoride released and recharged [42,43]. Even though ExciTE®F displayed excellent results in this study, it shares the inherent limitations of coating agents and serves as a barrier against fluoride release from GIC [44]. Petroleum jelly is used as a coating in the atraumatic restorative treatment (ART) approach due to its ease of application [45]. However, this study established that EQUIA® Forte HT coated with petroleum jelly exhibited lower microhardness values than those displayed by other coating agents, although it might still be a viable alternative compared to no coating.

Water sorption is a process regulated by diffusion and may lead to chemical breakdown of the material. This breakdown often occurs because of the breakage of the connection between the inorganic filler and organic matrix or the release of residual monomers. This results in a decline in the mechanical qualities of the material, leading to a decrease in the lifespan of the restoration. However, these compounds are released from the restoration via the solubility phenomenon, which gives rise to more alarming biological effects [46].

In this study, significant differences were observed in the water sorption results across experimental groups after 24 h and 7 days. Notably, specimens coated with EQUIA Forte® Coat exhibited the lowest water sorption values, indicating superior resistance to water absorption compared to the other coating agents. This finding is consistent with that of a previous study conducted by Yilmaz et al. who reported that the specimens coated with EQUIA Forte® Coat had the lowest water sorption values. This indicated that the nano-filled resin-based coating efficiently sealed the material surface, decreased water infiltration, and improved its longevity [35]. The hygroscopic expansion of resin-based materials is affected by the characteristics of the organic matrix, particularly the chemical composition of the monomers and their hydrophilicity [47]. Hydrophobic adhesives act as membranes with low permeability; however, as permeable membranes, polymerized hydrophilic adhesives have the potential to allow fluid to flow both outward and inward [48]. The composition of Single Bond Universal and ExciTE®F adhesives comprises an organic matrix consisting of hydrophilic monomers such as bisphenol A-glycidyl dimethacrylate (Bis-GMA) and 2-hydroxyethyl methacrylate (HEMA). The observed increase in the water sorption values may be ascribed to the hydrophilic properties of the matrix [44].

After 7 days, the performance of petroleum jelly in this investigation was found to be superior to that of both the Single Bond Universal Adhesive™ and ExciTE®F. Similar findings were observed by Yilmaz et al., in which petroleum jelly demonstrated superior performance to the Single Bond Universal Adhesive™ [35]. The simplicity of petroleum jelly administration makes it a suitable coating for atraumatic restorative treatment (ART) [45]. The gradual increase in water sorption over time for some coating agents indicates possible degradation or weakening of the coating layer, resulting in elevated water infiltration into the underlying material. This phenomenon emphasizes the need for durable and reliable coating agents to guarantee prolonged protection against water infiltration and consequent degradation of the materials. The significant differences observed between the coating agents highlight the importance of selecting appropriate surface-coating materials based on their specific effects on water sorption and solubility. Nano-filled coatings such as EQUIA Forte Coat have shown potential for improving the water resistance and longevity of HVGIC restorations.

The negative solubility values obtained in this study suggest that these products exhibit a higher affinity for water sorption, perhaps leading to concealment of their true solubility. This does not prove that there was no solubility but rather that the amount of water absorbed was greater than the amount of soluble material. This may be related to the increased hydrophilicity of the materials [49,50]. Numerous studies have shown that negative solubility results in the use of many different types of restorative materials [35,50–53].

However, it is important to acknowledge the limitations of this study. This experiment was limited by its short duration in the laboratory, which does not accurately reproduce clinical conditions including dynamic variables such as masticatory and brushing forces, temperature changes, and pH variations. These elements have the potential to compromise the adhesion between the coating agent and material surface. Further investigations are required to examine the additional mechanical and biological characteristics of EQUIA Forte® HT restorations in different clinical situations to provide a more thorough evaluation.

## 5. Conclusions

Overall, the findings of this in vitro study contribute to the growing body of knowledge on the influence of surface-coating agents on some of the physical properties of EQUIA Forte® HT materials. The study found that all coating agents significantly increased the microhardness of EQUIA Forte® HT, with the application of EQUIA Forte® Coat significantly increasing the hardness of EQUIA Forte® HT compared to the other agents. The application of EQUIA Forte® Coat showed the lowest water sorption values in comparison to the other coating agents.

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