Effect of Acidulated Phosphate Fluoride Gel Application on the Surface Roughness of Resin Sealant

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Abstract. When resin sealant for teeth is exposed to acidulated phosphate fluoride (APF) gel, a commonly used fluoride application agent, its surface roughness can increase, potentially resulting in bacterial adhesion and secondary caries. Therefore, this study analyzed the effect of the application of APF gel on the surface roughness of resin sealants. Thirty-six resin sealant specimens were randomly divided into six groups, which received 1, 2 or 3 applications of APF gel or were soaked in distilled water (aquadest) 1, 2, or 3 times for 30 min. A Mitutoyo SJ201 surface roughness tester was used to measure the mean roughness. Surface roughness was significantly increased after a single gel application, but decreased significantly after two gel applications (p < 0.05). Thus, APF gel reduced resin sealant surface roughness after two or more applications.

1. Introduction

Pit and fissure sealant is applied as a physical barrier to protect the surface of teeth against bacteria and their acidic products. The sealant coats and protects the small pits and fissures in teeth, which are often not cleaned effectively by saliva [1,2]. It is indicated for fully developed posterior teeth with deep pit and fissure anatomy and without extensive restoration or active caries, and it is applied as early as possible. Pit and fissure sealant can be made from glass ionomer cement or resin, with resin being used more often because of its mechanical properties and its greater durability.

Fluoride is effective for remineralizing the surfaces of teeth [3]. It is applied to the smooth and root surfaces of the teeth of patients who are at a high risk of caries, who are undergoing orthodontic treatment, or who have a low saliva flow rate. Topical fluoride is applied every 4–6 months [4]. Acidulated phosphate fluoride (APF) gel 1.23% is the most commonly used fluoride application agent because of its stability and commercial availability. The gel contains acid phosphate, which etches the tooth enamel; fluoride ions then replace the hydroxyapatite mineral to form fluoroapatite, which is more stable and more resistant to acid [5].

A mouth tray is used to apply the APF gel to the teeth, which means that the surface of any restorative material on the teeth may also come into contact with the gel. Therefore, the effect of the application of APF gel on the surface of materials such as resin sealants is important [6]. The composition of resin sealants is similar to that of resin composites in general [7], with filler being one of the components [8]. Previous research has shown that the application of acidic APF gel 1.23% may degrade filler particles in resin composites; the released filler particles from the matrix can result in holes on the surface, increasing the surface roughness [9]. A surface roughness of 0.2 µm can increase the tendency...
for bacterial accumulation, which can result in secondary caries and treatment failure [10]. Therefore, the present study investigated the effect of the application of APF gel on the surface roughness of resin sealants.

2. Materials and methods
This experimental study was conducted in the Dental Materials Laboratory, Faculty of Dentistry, Universitas Indonesia, during August and September 2014. A total of 36 cylindrical resin sealant specimens were made from 3M ESPE Clinpro Sealant, USA using an 8 × 2 mm acrylic split ring mold and polymerization with a light curing unit for 20 s. The specimens were randomly divided into six treatment groups and specimens received 30-min applications of APF gel on one, two, or three successive days. Then, a Mitutoyo SJ201 surface roughness tester was used to measure the surface roughness of each specimen on three sides.

Data analysis was performed using a computing program. The Shapiro–Wilk test showed the data were not normally distributed; therefore, the non-parametric Kruskall–Wallis and Mann–Whitney U tests were used to test the differences between the average roughness of the resin sealant surface after the application of APF gel and after being soaked in aquadest, with a significance level of P < 0.05 and confidence level of 95%.

3. Results
The surface roughness of the resin sealant specimens decreased after three applications of APF gel, but increased after being soaked three times in aquadest (table 1). However, the mean surface roughness did not exceed the mean critical value of 0.2 µm in any of the groups.

Table 1. The mean ± surface roughness (in µm) of the resin sealant specimens before and after acidulated phosphate fluoride (APF) gel application or soaking in aquadest.

| Treatment   | Initial roughness | One application | Two applications | Three applications |
|-------------|-------------------|-----------------|------------------|-------------------|
| APF         | 0.059 ± 0.016     | 0.134 ± 0.025   | 0.115 ± 0.031    | 0.036 ± 0.012     |
| Aquadest    | 0.060 ± 0.019     | 0.070 ± 0.020   | 0.084 ± 0.018    | 0.088 ± 0.019     |

The surface roughness increased after the first APF gel application, then decreased after the second and third applications (figure 1).
The differences in roughness between all the gel-treated groups and the baseline value, and between the three treated groups, were all statistically significant (Mann–Whitney U test; table 2).

### Table 2. Significance of the differences in mean roughness between the APF gel treatment groups.

| Comparisons                              | P     |
|------------------------------------------|-------|
| Baseline and one APF gel application     | 0.000* | ↑  |
| Baseline and two APF gel applications    | 0.000* | ↑  |
| Baseline and three APF gel applications  | 0.000* | ↓  |
| One and two APF gel applications         | 0.047* | ↓  |
| One and three APF gel applications       | 0.000* | ↓  |
| Two and three APF gel applications        | 0.000* | ↓  |

Notes: ↑ increased roughness, ↓ decreased roughness, * P < 0.05

With the resin sealant specimens soaked in aquadest, the mean surface roughness increased with each soaking (figure 2).

### Figure 1. Mean surface roughness of the specimen groups treated one, two or three times with acidulated phosphate fluoride (APF) gel.

### Figure 2. Mean surface roughness of the specimen groups soaked one, two, or three times in aquadest.
Table 3 shows the statistical significances for the differences in mean roughness between each group and the baseline value and between the groups.

| Comparisons                                    | P       |
|------------------------------------------------|---------|
| Baseline and one soaking in aquadest           | 0.181   |
| Baseline and two soakings in aquadest          | 0.010*  |
| Baseline and three soakings in aquadest        | 0.000*  |
| One and two soakings in aquadest               | 0.033*  |
| One and three soakings in aquadest             | 0.006*  |
| Two and three soakings in aquadest             | 0.520   |

Note: * P < 0.05

4. Discussion
In this study, APF gel was applied to resin sealant one, two, or three times for 30 min. This mimicked the use of topical fluoride up to three times a year on the assumption that the patient does not eat, drink, or rinse for 30 min after the application. The mean surface roughness of the resin sealant increased after the first application and then decreased after the second and third applications. These changes would be influenced by the pH of the APF gel (pH 2). In contrast, with the specimens soaked in aquadest, the mean surface roughness increased significantly after the second soaking only.

Changes in surface roughness of resin sealant can be caused by the chemical degradation of the matrix and filler [11]. Degradation is caused by the release or the loss of chemical structure of the resin composite, which is influenced by the composition and type of chemical bonds of the matrix polymer, the degree of acidity of the soaking medium, and the ability of the matrix to absorb the water [12].

Under acidic conditions, an organic ester derivative in the matrix, methyl methacrylate, undergoes hydrolysis. The water content can hydrolyze the bond between the matrix and filler on the coupling agent [13]. Fluoride ions also play a role in the depolymerization reaction of the coupling agent [14]. This mechanism can weaken the interface between the filler and matrix, resulting in the release of the filler particles, causing the filler particles to project out and creating holes on the surfaces, thereby increasing surface roughness. In addition, this can reduce the weight and hardness of resin composites [15,16].

The matrix of methyl methacrylate-based resin sealants belongs to the organosilicon ester group. Under acidic conditions, the ester group undergoes a hydrolysis reaction, which is accelerated by acid catalyst (H⁺). This reaction produces carboxylic acid and alcohol. The solubility of carboxylic acid in water is high; even carboxylic acid with 1–4 carbon atoms can fully dissolve in water. Filler particle projections and holes form on the surface of the resin sealant because of the degradation of the matrix.

Filler particles may be released from the matrix because of the weakening of the siloxane bond. Siloxane bonds in the coupling agent play a role in bonding the filler particles to the matrix. The siloxane bonds can be weakened by autocatalytic hydrolysis. OH⁻ ions from the water diffuse into the matrix and break the siloxane bond into silanol and Si–O. The Si–O compounds react with water, forming silanol and OH⁻. As long as the resin sealant is soaked in the water, this reaction continues. Diffusion of the water into the matrix is also influenced by the network density of the matrix polymer. Filler particles can be released from the matrix because of the weakened siloxane bond, creating holes on the surface [17].

In the present study, the surface roughness of the resin sealant after the first application of APF gel increased from 0.059 µm to 0.134 µm. This was due to the acidic nature of the APF gel (pH 2), which accelerated the continuation of the hydrolysis reaction of the resin sealant matrix, so the matrix dissolved
in water after the first application of APF gel and created projections of filler particles on the surface. This notion is supported by previous research that reported that APF gel can etch restorative material with high glass or quartz content, such as GIC, RMGIC and resin composites [18,19]. Projections that formed on the surface increased the surface roughness of the resin sealant. This is in accordance with the research by Dionysopoulos et al., who reported that, clinically, the topical application of APF gel could accelerate the degradation of the surface of restorative material and increase its surface roughness [20]. This contrasted with the research by Jung et al., which found that the surface roughness of nanofill resin composite did not increase significantly [13]. This difference in results was probably due to a difference in the size of the filler in the resin composite. Smaller sized fillers are released to a lesser extent by APF gels [9,21].

In the present study, the surface roughness decreased after the second application of APF gel. This was because the material had reached saturation point after the first application. The acid catalyst hydrolysis that changed the ester groups into carboxylate acid and alcohol started to decrease. In addition, the release of the siloxane bond on the coupling agent released the projections of filler particles located on the surface after the first application. The release of these filler particles made the surface of the resin sealant flatter and smoother. After the third application of APF gel, the surface of the resin sealant was less rough than the initial value. A decrease in the surface roughness of microhybrid and microfilled resin composites has been observed in previous studies [22,23]. Smoothing of the surface may result from the degradation of larger filler particles, leaving smaller filler particles on the surface. In addition, the amount of APF gel degradation on the surface of the resin composite may also have been influenced by the existence of a resin-rich layer on the surface of the resin composites [22].

According to Mair et al., the acid content of APF gel can cause erosion on the surface of restorative materials [24]. This has been supported by Papagiannoulis et al., who reported that the acid content of APF gel could dissolve resin composite filler [25], and by Soeno et al., who reported that the application of APF gel could improve surface roughness and reduce the wear-resistance of resin composites [11]. However, the condition of the restorative material after the clinical application of APF gel may differ from that found in an in vitro study because there are other factors in vivo that can affect the acidity, such as the pellicle, the saliva protection status, the degree of buffering, and oral hygiene [22].

5. Conclusion
APF gel, a topical fluoride with an acidic content, increased the surface roughness of resin sealant after one application, although without exceeding the critical value of 0.2 µm at which there is a tendency for increased cariogenic bacteria colonization. Three applications of APF gel lowered the surface roughness of the resin sealant, leaving it smoother than before the first application.

6. References
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