

Fluoride rinse effect on retention of CaF_2 formed on enamel/dentine by fluoride application

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Abstract: Calcium fluoride-like materials (“ CaF_2 ”) formed on dental surfaces after professional fluoride application are unstable in the oral environment but can be retained longer with a daily NaF mouthrinse. We tested the effect of twice daily 0.05% NaF rinses on the retention of “ CaF_2 ” formed on enamel and dentine after applying acidulated phosphate fluoride (APF). “ CaF_2 ” formed on enamel/dentine by APF application significantly decreased after exposure to artificial saliva and the 0.05% NaF rinse was ineffective to avoid this reduction. These findings suggest that the combination of APF and 0.05% NaF is not clinically relevant, either for caries or dental hypersensitivity.

Keywords: Calcium Fluoride; Dentin; Fluorides, Topical.

Introduction

The anticaries benefit of acidulated phosphate fluoride (APF) application is well established.¹ It results in the formation of a high concentration of calcium fluoride-like materials (“ CaF_2 ”) on enamel and dentine surfaces, which has been considered responsible for the anticaries effect of professionally applied topical fluoride treatments.²

Nevertheless, 80%-90% of “ CaF_2 ” formed is dissolved in the oral environment during the first week because saliva is undersaturated with respect to “ CaF_2 ”.³ Therefore, retaining “ CaF_2 ” on enamel or root dentine for a prolonged period of time is desirable, mainly for patients with a high risk for caries.⁴

The dissolution of “ CaF_2 ” formed on enamel and dentine after applying APF could be decreased by the daily use of fluoride products, such as a 0.05% NaF mouthrinse. A 0.05% NaF mouthrinse may compensate for the dissolution of “ CaF_2 ” because it contains 225 ppm F, which forms a small amount of “ CaF_2 ” on enamel-dentine; however, this subject has not been explored.

Therefore, we evaluated if twice daily exposure to 0.05% NaF solution *in vitro* (simulating F mouthrinse) would prolong the retention of “ CaF_2 ” formed on enamel and dentine after applying APF.

Methodology

Experimental design

An *in vitro* 2×3 factorial study was conducted, including the following factors: 1) APF application at two levels, *i.e.*, applied (APF⁺) or not (APF⁻)



and 2) 0.05% NaF treatment at three levels, *i.e.*, not treated (NT), treated with NaF during saliva immersion (NaF⁺), or treated with purified water (NaF⁻) during saliva immersion.

Enamel (48) and dentine (48) slabs were randomly divided into six groups of eight slabs each, according to the factorial design: APF⁻/NT; APF⁻/NaF⁻; APF⁻/NaF⁺; APF⁺/NT; APF⁺/NaF⁻, and APF⁺/NaF⁺. The APF⁺ and APF⁻ groups were pre-treated with 0.5 M NaF in 0.1 M H₃PO₄, pH 3.5 (APF⁺) or 0.1 M H₃PO₄, pH 3.5 (APF⁻) solutions, respectively. The alkali-soluble fluoride (“CaF₂”) concentration formed in groups APF⁻/NT and APF⁺/NT was immediately determined after treatment. Slabs from the NaF⁺ and NaF⁻ groups were individually immersed in artificial saliva following pre-treatment and treated twice daily with 0.05% NaF solution (NaF⁺) or purified water (NaF⁻), respectively. “CaF₂” concentration was determined in the slabs after 7 days.

Preparation of enamel and dentine slabs

The enamel and dentine slabs (3 × 3 × 2 mm) were obtained from sound bovine incisors, and their external surfaces were polished flat. Then, the slabs were isolated with wax; thus, only the external surface of the enamel or dentine was exposed to the treatment.

Treatments

The enamel and dentine slabs were immersed into either the control or APF solution for 4 min under agitation (120 rpm). The slabs were washed for 30 s with a stream of purified water and gently dried with absorbent paper. The slabs in the APF⁻/NT and APF⁺/NT groups were immediately analyzed for alkali-soluble F concentration. The remaining slabs were individually immersed in artificial saliva⁵ (1.7 mL/mm²) and maintained at 37°C for 7 days. The slabs from both groups were exposed to purified water or a 0.05% NaF solution for 1 min under agitation (120 rpm) twice daily at 7 am and 5 pm. Following each treatment, the slabs were flushed with purified water (10 s), dried, and immersed again in artificial saliva. The artificial saliva was changed every morning after treatment.

Determination of “CaF₂”

Slabs were individually immersed in 1 M KOH (0.4 mL/block) for 24 h under agitation. An additional 6-h extraction with 1 M KOH (0.3 mL/block) was performed to deplete all alkali-soluble fluoride. The extraction solution was buffered with TISAB II containing 1 M HCl. Fluoride was measured with an ion-selective electrode (Orion 96-09; Thermo Scientific, Inc.) and an ion analyzer (Orion EA-940, Thermo Scientific) against standards prepared as the samples, and the alkali-soluble fluoride concentration (sum of the two alkali extractions) was expressed as µg F/cm².

Statistical analysis

A factorial 2 × 3 analysis was conducted. Enamel and dentine data were independently analyzed. The alkali-soluble fluoride concentrations in the enamel and dentine slabs were transformed to square root and log₁₀, respectively. Tukey test was used for the post-analysis of variance comparisons. The data were analyzed using SAS software version 8.01 (SAS Institute, Cary, USA), and a p-value of < 0.05 was considered significant.

Results

APF resulted in significantly higher “CaF₂” concentrations in the enamel (Table 1) and dentine (Table 2) slabs (p < 0.05) than the control. “CaF₂” concentrations significantly decreased by 85 and 90% in the enamel and dentine after exposure to saliva and purified water, respectively (p < 0.05). The “CaF₂” concentration retained in the enamel and dentine after treatment with 0.05% NaF did not differ from the control group treated with water (p > 0.05).

Discussion

Our results confirmed that loosely-bound fluoride (“CaF₂”) was formed after APF was applied to enamel,² but also showed that the same occurs with dentine. The “CaF₂” concentration formed on dentine was 7-fold higher than that on enamel, which may be explained by the smaller hydroxyapatite crystals in dentine resulting in a larger surface area to crystallite volume ratio

Table 1. Alkali-soluble fluoride ("CaF₂") (μg F/cm²) formed on enamel by APF or control pre-treatments and retained after 7 days under saliva exposure and daily treatment with 0.05% NaF or the control (mean ± SD; n = 8).

| Pre-treatment groups | Formed (not treated) | Retained after saliva exposure | |
|---|----------------------|--------------------------------|------------------------------|
| | | NaF (purified water) | NaF ⁺ (0.05% NaF) |
| APF (0.1 M H ₃ PO ₄ , pH 3.5) | 0.50 ± 0.25 A,a | 0.25 ± 0.04 A,b | 0.44 ± 0.12 A,a |
| APF ⁺ (0.5 M NaF in 0.1 M H ₃ PO ₄ , pH 3.5) | 17.82 ± 11.56 B,a | 3.21 ± 1.91 B,b | 2.57 ± 1.13 B,b |

APF: acidulated phosphate fluoride.

Differences between rows are indicated by capital letters, and among columns by lower-case letters.

Table 2. Alkali-soluble fluoride ("CaF₂") (μg F/cm²) formed on dentine by APF or control pre-treatments and retained after 7 days under saliva exposure and daily treatment with 0.05% NaF or the control (mean ± SD; n = 8).

| Pre-treatment groups | Formed (not treated) | Retained after saliva exposure | |
|---|----------------------|--------------------------------|------------------------------|
| | | NaF (purified water) | NaF ⁺ (0.05% NaF) |
| APF (0.1 M H ₃ PO ₄ , pH 3.5) | 0.46 ± 0.24 A,a | 0.56 ± 0.17 A,a | 1.31 ± 0.77 A,b |
| APF ⁺ (0.5 M NaF in 0.1 M H ₃ PO ₄ , pH 3.5) | 127.19 ± 20.11 B,a | 13.34 ± 5.56 B,b | 10.27 ± 2.82 B,b |

APF: acidulated phosphate fluoride.

Differences between rows are indicated by capital letters, and among columns by lower-case letters.

and therefore a more reactive mineral phase.⁶ Additionally, dentine is more acid-soluble than enamel,⁷ resulting in more calcium being released by the APF treatment, which reacts with fluoride and precipitates as "CaF₂". Nevertheless, our data confirmed that "CaF₂" formed on the enamel and dentine was not stable in a saliva-like solution.³ "CaF₂" concentration decreased similarly (85%-90%) in enamel and dentine after 7 days of exposure to artificial saliva, and the 0.05% NaF twice daily treatment did not prevent this decrease. However, the amount of "CaF₂" remaining was higher than the concentration found in slabs pre-treated with a non-fluoridated control solution (APF⁻). The findings for enamel show that these reservoirs may last for a significant period of time, as previously shown *in vitro*³ and *in situ*⁸ and the same is expected for dentine.

The finding that the 0.05% NaF rinse did not prevent the dissolution of "CaF₂" formed by APF may be explained by the very small amount of fluoride deposited on sound dental tissues from a mouthrinse.⁹ In fact, the twice daily NaF treatment significantly

increased alkali-soluble fluoride concentrations on the dentine slabs that were not pre-treated with fluoride, but the concentration after 7 days was only approximately 1% of that resulting from the APF treatment (Table 2).

The present results are also relevant when APF is recommended as a dentine hypersensitivity treatment.¹⁰ Although "CaF₂" may block dentinal tubules and decrease sensitivity for some time, the daily use of a fluoride mouthrinse may not be suitable to prolong this effect.

Conclusion

Our findings suggest that 0.05% NaF treatment twice daily does not prevent oral dissolution of "CaF₂" formed on enamel or dentine by APF application.

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