Topical fluoride applications on human enamel; a combined in vivo - in vitro study
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The aims of this investigation were:
1. to follow longitudinally in vivo the amounts of fluoride on the enamel surface as well as the acquired fluoride in the enamel after topical F applications.
2. to determine in vitro the amounts of fluoride in and on the enamel after various topical fluoride applications (F_in and F_on, resp.).
3. to investigate the acid etching of human enamel (F_in).

The thickness of enamel layers removed by etching with acids is of considerable practical interest because the layer thickness removed influences the fluoride content given in ppm F directly and consequently the fluoride profiles in enamel.

In the first chapter the subject of topical fluoride applications on enamel is introduced.

In chapter 2 two methods are compared for estimating the amount of enamel removed by etching with 0.1 M HClO_4 solutions in various time intervals:

i) a measurement of the layer depth based on the amount of calcium removed during an etch period (d_Ca) and
ii) a direct measurement of the layer depth on tooth sections by an optical depth determination (d_DP).

Both techniques were applied to the same sample. The results show in most cases a statistical significant difference between the two values of interest d_Ca and d_DP; d_DP is greater than d_Ca. Increased exposure times result in larger differences between d_DP and d_Ca. For long exposure times d_DP is considerably greater than d_Ca; the difference can be as much as 50%. The reason for the discrepancy between optical and chemical depth determination is most likely due to the enamel prism structure. SEM pictures on 0.1 M HClO_4 etched specimen strongly indicate an increasing surface roughness with increased etching periods.

In chapter 3 we compare the rate of etching and the relative surface roughness caused by HClO_4 solutions of different concentrations. The average thickness of the enamel layer removed is a linear function of the total etching
period (except for very short periods). The rate of etching (β) with HClO₄ solutions in μm·sec⁻¹ is strongly influenced by acid strength and stirring rate during etching; β is ≈ 0.02, 0.06, 0.18 and 0.74 μm·sec⁻¹ for 0.02, 0.1, 0.5 and 2.0 HClO₄ solutions, resp.

β is a linear function of the acid strength. SEM pictures of the specimens show that acid strength, etching time and chemical composition of the enamel are the determining factors for surface morphology of etched enamel. A low acid concentration gives a sharp etch; increased acid strength causes increased surface roughness.

In chapter 4 the accuracy and reproducibility of the fluoride determination with solid state fluoride electrodes using the acid-etch procedure is described. Different fluoride electrodes show a comparable behaviour. The fluoride determined as described in this thesis is accurate and reproducible within ± 2% (range 0.04–0.6 ppm F).

In chapter 5 a comparison is presented of the amount of calcium fluoride deposited on the enamel surface and the acquired amount of fluoride in the enamel by topical applications in vitro. A single topical treatment with three different fluoride-containing systems on human enamel resulted both in an enrichment of the F content in the enamel and an accumulation of F reaction products on the enamel surface. The aim of this study was to quantify the fluoridation of an APF gel and two F-containing lacquers and to measure the amounts of KOH-soluble fluoride and acquired fluoride separately. Blocks of intact enamel were treated with the topical agents and subsequently exposed to 1 M KOH for 24 h. No measurable amount of fluoride was dissolved from the control specimens of intact enamel by this treatment. Results are presented from experiments with the three different fluorides applied to natural surfaces of the same tooth. The amount of fluoride \( F_{\text{on}} \) for the APF gel and Duraphat was comparable (≈ 20 μg·cm⁻²); Fluor Protector deposited more than twice this amount. The layer thickness of CaF₂ based on KOH-soluble fluoride is calculated. The acquired amount of fluoride in the enamel after exposure to KOH was not related to the pH value of the fluoridating agent nor to the total amount of fluoride deposited on or in the enamel. The total amount of acquired fluoride in the enamel—a 30 μm thick layer—was 4, 9 and 10 μg·cm⁻², for APF gel, Duraphat and Fluor Protector, resp. The total amount \( F_{\text{on}} \) and \( F_{\text{in}} \) in the specimens was 28, 38 and 60 μg·cm⁻², resp. The CONTACT TIME plays a dominant role in the fluoridating effect in deeper layers (5–30 μm) in the enamel surfaces for agents with the same pH.

In chapter 6 a comparison of the fluoride uptake by human enamel from APF gels
with different fluoride concentrations is presented. The experimental data of this experiment indicate that:
i) no statistically significant difference exists between the amount of fluoride acquired on and in the enamel for the gel preparations used, even not for the lowest concentration of 0.11% F;
ii) a statistically significant increase of the acquired F in the surface enamel (layer I) was observable only for 1.23% F gels.

Therefore, it is questionable whether there is any significant advantage in the application of high-concentration F gels.

In chapter 7 the fluoride deposited as $F_{\text{on}}$ and $F_{\text{in}}$ by topical applications in vitro with stannous fluoride on human enamel is described. A 4% aqueous solution of $\text{SnF}_2$ was applied for 5 min and 30 min, resp., and the amounts of KOH-soluble fluoride and acquired fluoride were measured separately. The amount of fluoride in the KOH solutions after 5 and 30 min $\text{SnF}_2$ application are comparable: 6.2 and 7.4 $\mu$g.cm$^{-2}$, resp. A single topical treatment of $\text{SnF}_2$ of 5 and 30 min did NOT produce a significant enrichment of fluoride IN the enamel. The considerable accumulation of fluoride reaction products on the outer enamel surface was only 1/3 of the amount deposited by APF gels.

In chapter 8, in an IN VIVO study the amount of fluoride present on and in human enamel was followed longitudinally for a period of 3 months. The fluoridating agents were an APF gel and the fluoridating lacquers Duraphat and Fluor Protector. The fluoride on the enamel ($F_{\text{on}}$, mainly as $\text{CaF}_2$) was determined by the Caslavska method. The fluoride in the enamel ($F_{\text{in}}$) was measured in 5 enamel layers removed by acid etching. 12 patients did wear the treated enamel specimens and controls for periods of 1, 4 and 12 weeks before $F_{\text{on}}$ and $F_{\text{in}}$ determinations were made. The $\text{CaF}_2$ ($F_{\text{on}}$) is lost in vivo in all 3 treatments with a rate of about 20 $\mu$g.cm$^{-2}$ in the FIRST WEEK. Consequently, APF gel and Duraphat treated specimen lost nearly all $\text{CaF}_2$ in vivo in this period. The amount of $\text{CaF}_2$ present in Fluor Protector was noticeable up to 1 month. A second conclusion is that the amount of acquired $F^-$ ($F_{\text{in}}$ in a layer of 30 $\mu$m) is after an APF gel or Duraphat treatment negligible after 1 week in vivo. A Fluor Protector treatment introduced an amount of acquired fluoride of 11, 14 and 15 $\mu$g.cm$^{-2}$ after 1, 4 or 12 weeks, resp.

This study shows conclusively that if sufficient $\text{CaF}_2$ is deposited on the enamel in vivo for a sufficiently long time, the amount of $F^-$ in the enamel can be increased significantly.

The role of calcium fluoride in topical fluoridation of human enamel is discussed in chapter 9. In case of the acidic local fluoride agents, the $F_{\text{on}}$
consists most likely of amorphous CaF$_2$. The removal in vivo of the $F_{on}$ after local application is due to a local dissolution of the CaF$_2$ and subsequent leaching away of Ca$^{++}$ and $F^-$ through the pellicle. Only a fraction of the amount of $F$ applied by the topical agents is used in the fluoridation process; for APF gel, Duraphat and Fluor Protector applications about 5, 1 and 44% of the F actually available participates in the fluoridation process.

From this study one has to conclude that:
1) large amounts of CaF$_2$ are formed after topical fluoride application on the enamel surface,
2) nearly all CaF$_2$ in vivo is removed fast, despite the presence of the pellicle,
3) CaF$_2$ leaching away in vivo does hardly or not fluoridate enamel of adjacent teeth,
4) CaF$_2$, present as $F_{on}$, introduces minor amounts of fluoride (as $F_{in}$) in the enamel,
5) the $F_{in}$ acquired from the CaF$_2$ sufficiently long in contact with the teeth can penetrate deep (30 μm) into sound enamel.