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Etching effect of acidic fluorides on human tooth enamel in vitro

Carl Hjortsjö [a,*], Grazyna Jonski [b], Alix Young [c], Erik Saxegaard [a]

[a] Department of Prosthetic Dentistry and Oral Function, Faculty of Dentistry, University of Oslo, P.O. Box 1109, Blindern, 0317 Oslo, Norway

[b] Oral Research Laboratory, Faculty of Dentistry, University of Oslo, P.O. Box 1109, Blindern, 0317 Oslo, Norway

[c] Department of Cariology and Gerodontology, Faculty of Dentistry, University of Oslo, P.O. Box 1109, Blindern, 0317 Oslo, Norway

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ABSTRACT

Objective: This in vitro study aimed to examine the etching effect of acidic fluoride solutions on enamel.

Materials and methods: 24 human teeth divided into 48 enamel-specimens were partly isolated with impression material. Specimens were exposed for 10 min to 20 ml of the following solutions: 1.6% TiF_4, 3.9% SnF_2, 0.2% HF and 1.8% citric acid (CA). The isolation was removed and 24 specimens analysed by profilometry (Δheight: exposed/isolated enamel surfaces, surface roughness parameters). For the remaining 24 specimens [Ca^{2+}] in the test solutions was analysed by atomic absorption spectroscopy.

Results: Median Δheights (μm) after exposure were: TiF_4 0.07, SnF_2 −0.03, HF −0.14 and CA −5.92. TiF_4-exposed surfaces showed both deposits and etched areas and exhibited statistically significant different surface roughness parameters compared to the HF- and SnF_2-exposed surfaces. Median [Ca^{2+}] values (ppm): TiF_4 1.88, SnF_2 0.11, HF 0.10 and CA 2.17.

Conclusion: At the [F] tested in this study it can be concluded that SnF_2- and HF solutions had negligible erosive effects on enamel. TiF_4 solution resulted in an incomplete surface deposition associated with calcium dissolution suggesting that TiF_4 applied as solution may not be advisable.

1. Introduction

Dental erosion lesions are caused by the action of extrinsic- or intrinsic acids usually in combination with abrasion and or attrition.[1,2] Citric acid is a common extrinsic acid often found in beverages and food. Treatment of enamel with acidic fluoride solutions such as titanium tetrafluoride (TiF_4), stannous fluoride (SnF_2) and hydrogen fluoride (HF) has in vitro and in situ shown to provide some protection against dissolution upon exposure to such acids.[3–8] It has been suggested that the erosion-inhibiting effects of TiF_4 and SnF_2 are related to the formation of a coating or a glaze layer that prevents further erosion or due to the formation of

* *Corresponding author at*: Department of Prosthetic Dentistry and Oral Function, Institute of Clinical Dentistry, University of Oslo, P.O. Box 1109, Blindern, 0317 Oslo, Norway. Tel.: +47 22852368; fax: +47 22852344.
E-mail address: carl.hjortsjo@odont.uio.no (C. Hjortsjö).

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acid-resistant calcium fluoride precipitates on the enamel surface.[3,6,9–11] Some authors have reported the coating/glaze layer to be incomplete or have cracks,[6,9] and have attributed this observed lack of a complete glaze coating to microscopy preparation. However, if the formation of an incomplete coating/glaze layer is a true effect, it may influence the protective effect against erosion seen by TiF_4 and SnF_2. It has also been demonstrated that in vitro solutions containing stannous ions (Sn^{2+}) may lead to the incorporation of Sn in the outer enamel surface.[12,13] The higher the amount of incorporated Sn in the enamel, the less enamel tissue was shown to be lost when the surface was exposed to citric acid.[12]

Acidic fluoride solutions below pH 3 contain fluoride largely in the form of undissociated HF^0 molecules, in addition to smaller amounts of free fluoride anions. HF^0 has the ability to diffuse into intercrystalline spaces of enamel[14] and penetrate the hydroxyapatite lattice in the root-dentine surface.[15] Recently it has been suggested that HF^0 may result in the formation of a subsurface layer of acid-resistant calcium fluoride-like material.[5] However, concerns regarding the potential of acidic fluorides to remove calcium (and phosphate ions) from the enamel surface have been raised. In a study by Skartveit and co-workers where tooth root surfaces were treated with 4% TiF_4, a partly demineralised zone with a depth of 8–10 μm was demonstrated after 1 min. After 4 min this zone was 5–27 μm deep.[16] Different dental hard tissues react differently to erosive demineralization as demonstrated by Ganss and co-workers.[17] Although one cannot assume that the above-mentioned changes seen in root dentine can be directly extrapolated to dental enamel, the changes may serve as a predictor of possible reactions.

The aim of this in vitro study was therefore to investigate possible surface changes on human enamel specimens following prolonged exposure to different acidic fluoride solutions (TiF_4, SnF_2 and HF) using a simplified model not including a salivary pellicle. The null hypothesis to be tested was that these acidic fluoride solutions do not result in any etching of enamel surfaces as measured by profilometric- and spectrometric analyses.

2. Materials and methods

2.1. Tooth samples, specimen preparation and isolation

Twenty-four intact, surgically extracted human third molar teeth were collected from private dental practitioners in Oslo and surrounding areas, or from the Department of Oral Surgery and Oral Medicine, Institute of Clinical Dentistry, Faculty of Dentistry, University of Oslo. All tooth donors gave their consent to the use of their teeth for this research project. It was not known whether the teeth crowns (or parts of the crowns) had been exposed to the oral cavity prior to extraction. Teeth were cleaned and stored in a moist thymol environment (99.5% thymol, Sigma–Aldrich, St. Louis, MO, USA).

Prior to the experiment, the teeth were prepared using a saw with water cooling (Exakt-apparatgebau, Norderstedt, Germany) and were cut vertically into two halves thus providing 48 enamel specimens. The teeth that were to be analysed by profilometry also had their roots removed by use of the same saw with water cooling. All specimens were then glued to a plastic object plate (Technovit 7210 VLC; Heraeus Kulzer GmbH, Wehrheim, Germany). A flat enamel surface was ground wet on each specimen with an abrasive paper to grit 2500 (P2500, silicon carbide grinding paper, Buehler, Düsseldorf, Germany) (Exakt-apparatebau, Norderstedt, Germany) until a surface area of approximately 9 mm^2 was exposed. The size of this area was dependent on the original size, position and form of each tooth, and approximately 200 μm of the surface enamel was removed during this process.

One half of the enamel specimens ($n = 24$) were prepared for profilometric analysis. Half of the ground enamel surface area of each specimen was isolated using a thin layer of light body polyvinyl siloxane (PVS) impression material (Express II light body, 3M-ESPE, Seefeld, Germany). The border between the isolated and exposed area of enamel was situated at the centre of the ground enamel surface. After approximately 6 min the impression material was set and the specimens were ready for exposure to one of the test solutions.

The other half of the specimens ($n = 24$) were prepared for calcium analysis. Specimens were completely covered with a thin layer of the same light body PVS impression material. After setting a custom-made punching instrument with a circular cutting edge (diameter 2.5 mm) was used to carefully punch a hole through the impression material on each specimen, exposing a 4.9 mm^2 circular area at the centre of the ground enamel surface.

2.2. Test solutions and treatment of the specimens

The following fluoride solutions and citric acid solution were included in the study: TiF_4 solution (1.6% w/v, 0.5 M F^-, pH 1.42) was prepared from titanium tetrafluoride (Sigma–Aldrich Chemie, Steinheim, Germany), SnF_2 solution (3.9% w/v, 0.5 M F^-, pH 2.65) was prepared from tin II fluoride (Sigma–Aldrich Chemie, Steinheim, Germany), and HF solution (0.20% w/v, 0.1 M F^-, pH 3.02) was prepared from 40% hydrogen fluoride (Rectapur, Prolabo, Paris, France). Citric acid solution (1.8%, pH 2.27) was included as a positive control (Sigma–Aldrich Chemie, Steinheim, Germany). Deionised water was used in the preparation of all solutions. The choice of concentration for the fluoride solutions was based on previous studies reporting the beneficial effects of these fluoride solutions.[4,5,18,19]

The HF solution had a lower fluoride concentration than the TiF_4 and SnF_2 solutions since previous in vivo and in vitro studies have demonstrated HF toxicity at higher concentrations.[20] The pH of the solutions was measured at room temperature (pH-meter Orion Star, Thermo Electron Corporation, Beverly, MA, USA).

Following the isolation procedures, pellicle-free enamel specimens were exposed for 10 min to one of the test solutions ($n = 6$ specimens/solution) by submerging each specimen in separate plastic containers (Coulter, Kartell spa, Noviglio, Italy) each containing 20 ml of the solutions under constant, gentle agitation (100 rpm, IKA KS 260 Basic, Stuafen, Germany). The large volume of solution was used in order to avoid the possibility of re-precipitation of minerals onto the