

ABSTRACT

X-Ray Photoelectron Spectroscopy (XPS) was used to compare effects of commercial dentifrices on pellicle thickness/composition on treated enamel surfaces. Pellicle-like surfaces were prepared in triplicate on polished bovine enamel samples by 16 hour immersion in pooled, reconstituted human saliva. Dentifrice slurries [Crest® Regular (control - CR); Mentadent® (M - Baking Soda/Peroxide); Crest Gum Care (CGC - SnF₂); Colgate® Total (CoT - triclosan/Gantrez Acid Polymer); Arm and Hammer® Tartar Control (AHTC - soluble pyrophosphate PPI); Crest Tartar Control (CTC - soluble PPI); Crest Multicare (CM - soluble PPI); and Blend-a-Med® Synergy (BAMS - soluble polyphosphate - e.g. polypyrophosphate)] were prepared 25 % w/w paste/water and samples were treated by either brushing (30 second direct brushing of samples with slurry using medium brush) or dipping (chemical treatment only - 30 seconds). XPS analysis was performed on treated samples as described by Busscher et al. (Surf. Interf. Anal. 15, 344, 1990) - where pellicle surface thickness was estimated by an overlay model based upon the attenuation of the Ca signal. Brushing (combined chemical and abrasive effects) produced substantial reductions in pellicle content on teeth ranging from 65-85 %. Dipping produced substantially more variability between pastes - with relative pellicle dispersion vs. water measuring: Cr: 62 %; M: -11.5 % (thicker coating); CGC: 20 %; CoT: 43 %; AHTC: 66 %; CTC: 69 %; CM: 70 %; BAMS: 78%. **It is clear that dentifrice treatment results in significant variations in pellicle quantity, particularly in non-brushed areas. Polyphosphate and PPI dentifrice demonstrated superior chemical dispersion of pellicle components.**

MATERIALS AND METHODS**Treatments**

The dentifrices used in this study are compiled in Table 1 together with available information on the cleaning system ingredients which might be expected to strongly influence conditioning films. For treatments, slurries of dentifrices were prepared by thorough homogenization of 25 % (w/w) dentifrice water mixtures.

Conditioning Films

Pellicle conditioning films were formed by immersion of bovine enamel specimens for 16 hours in human saliva which is reconstituted to buffer media. Whole human saliva was collected as described previously (Bos et al., 1996). The saliva of approximately 20 volunteers was stimulated by the chewing of parafilm and collected in ice-chilled beakers. The saliva was pooled and centrifuged (10,000 g, 5 min, 10°C) with phenylmethylsulfonyl fluoride added to a final concentration of 1 mM as a protease inhibitor. The saliva was again centrifuged, dialysed (MW cutoff 6,000-8,000, Spectra/Por, Spectrum Medical Industries Inc., Los Angeles, CA, USA) for 2 days at 4°C against Millipore filtered water, then freeze-dried for storage. For experiments, 1.5 g/L freeze-dried saliva was reconstituted in buffer (2 mM potassium phosphate, 1 mM CaCl₂, 50 mM KCl, pH 6.8).

Enamel Preparation and Treatment

Bovine enamel blocks were sectioned and mounted in methacrylate. Surfaces were serially polished to 0.3 mm to a mirror finish prior to testing. Triplicate specimens were used for each treatment. Prior to dentifrice treatments, conditioning films were prepared by 16 hour immersion of specimens in reconstituted saliva. Treatments included: 1) 30 seconds vigorous brushing of the tooth surface with dentifrice slurry applied onto a soft bristle toothbrush or 2) 30 seconds dipping in dentifrice slurry without brushing. Specimens were separated into control (no treatment) and post-treatment groups (as XPS analysis modifies the surfaces - see below).

Analysis

Surfaces were analyzed for thickness and composition of thickening films by XPS as described by Busscher et al., (1990) where the stationary charge voltage of dental enamel specimens due to photoelectron ejection is related to the adsorbed layer thickness of specimens.

Tested Dentifrices

Dentifrice	Cleaning System
Crest Regular	Silica, surfactants
Mentadent	Baking soda, peroxide, surfactants, poloaxamer, silica abrasive
Crest Gum Care	Silica, surfactants, stannous fluoride
Colgate Total	Silica, surfactants, PMA-MVE copolymer
Arm & Hammer Tartar Control	Baking soda, silica, pyrophosphate
Crest Tartar Control	Silica, surfactants, pyrophosphate
Crest Multicare	Silica, surfactants, pyrophosphate
Blend-a-Med Synergy	Silica, surfactants, poly-pyrophosphate

RESULTS

Pellicle Conditioning Film Thickness - Post Treatment

Dentifrice	% Film Removed vs. Non Treated Control Brushing	% Film Removed vs. Water Control Dipping Only
Water Brush	--	--
Crest Regular	+77	+62
Mentadent	+76	-12
Crest Gum Care	+66	+20
Colgate Total	+72	+43
Arm & Hammer Tartar Control	+84	+66
Crest Tartar Control	+79	+69
Crest Multicare	+72	+70
Blend-a-Med Synergy	+84	+78

Surface Chemical Composition Post Treatment - Dipping Only

Dentifrice	% N	% P
Water Brush	+10	+03
Crest Regular	+05	+07
Mentadent	+11	+03
Crest Gum Care	+10	+04
Colgate Total	+08	+06
Arm & Hammer Tartar Control	+11	+03
Crest Tartar Control	+03	+08
Crest Multicare	+04	+10
Blend-a-Med Synergy	+03	+11

DISCUSSION

Dentifrice treatments produce profound changes on enamel conditioning films. Direct toothbrushing with dentifrice removes significant quantities of conditioning films - and only marginal differences appear between toothpastes. The treatment of enamel without brushing (reflecting areas hard to reach during hygiene) reveals significant differences between various commercial dentifrices, with some dentifrices showing exceptional capability in dispersing conditioning film from surfaces. Chemical analysis confirmed XPS film measurements, with good cleaners showing decreased N content (arising primarily from pellicle proteins), and with phosphate surface active dentifrices (Crest Tartar Control and Crest Multicare and to the largest degree Blend-a-Med Synergy) showing elevated surface P (deposited tartar control ingredient)

CONCLUSION

- Dentifrice brushing and rinsing produce strong, at least temporary changes in pellicle conditioning films
- Dentifrices differ significantly in chemical dispersive actions on conditioning films
- Dentifrices containing soluble pyrophosphate and polymeric pyrophosphate (polyphosphate) produce enhanced surface cleaning of conditioning films