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Supplemental Appendix

Energy Dispersive X-Ray Spectroscopy (EDS) Mapping

All experimental EDS data that are not included in the main text are included in this section. Accompanying HAADF-STEM images indicate the areas that were scanned during EDS acquisition. The obtained two-dimensional EDS maps of relevant elements are presented in this section. Elemental mappings provided a qualitative distribution of the elements: a higher intensity in the map represents more X-ray counts (and indirectly a higher concentration) of that element. The atomic percentage of elements such as calcium (Ca), phosphorous (P), carbon (C), oxygen (O), fluoride (F), nitrogen (N), sodium (Na), magnesium (Mg) and chloride (Cl) were calculated from the spectra in the 2-D elemental mappings and should be regarded as semi-quantitative: X-rays generated by lighter elements could be (partly) absorbed by the TEM sample which might yield deviations from the actual concentrations.

The enamel structures contained Ca, P and O in the crystal facets and C on the rod sheaths depending on treatment. Dentine material contained Ca, P and O in the dense peritubular and open-fibered inter-tubular regions. In contrast to enamel, N was found in all dentine material which suggests the presence of more protein in dentine. The N-amount found in the inter-tubular regions was often higher than in the peritubular regions. For both enamel and dentine, traces of minerals like Na, Mg, Cl and F were found. F appeared to be the only element present that could be influenced by Ga ion milling and/or electron beam during EDS acquisition. If such artefacts occurred, the fluoride map was not included in the results.

Atomic percentages were calculated from the spectra in the 2-D elemental mappings and should be considered as semi-quantitative: X-rays generated by lighter elements could be (partly) absorbed by the TEM sample which might yield deviations from the actual concentrations. Moreover, the TEM sample thickness may influence the quantification result.

As expected, Ca, P and O were found in atomic ratios close to the expected hydroxyl apatite ($Ca_{10}P_6O_{24}$) for all tooth structures. However, it should be noted that EDS neither sees the difference between O from an OH-group or O-from calcium phosphate nor can it detect hydrogen. Small fluctuations seen in O concentration are believed to be rather an EDS (quantification) artifact than to be intrinsic to the teeth and/or a result of the treatments. This is because O is the lightest element of Ca, P and O. Therefore the O generated by X-rays is absorbed more by the TEM sample itself. There is no evidence found that any of the other elements (Cl or F) replaces O. For example, the fluctuations observed for F do not show any relation to the fluctuations seen for O (Cl-amount is negligibly small to be linked to fluctuations caused by treatment). Na^- and Mg^- were present only in minor amounts to show any significant fluctuation.

The individual tooth characteristic observations are mentioned separately with the mappings and the EDS findings are summarized in the table below.

Appendix Table 1. Table showing the characteristic feature differentiating untreated and treated samples.

Characteristic features	Untreated		30% H_2O_2 @ pH7		30% H_2O_2		NaOH	
	dentine	enamel	dentine	enamel	dentine	enamel	dentine	enamel
Mobility of F/relation between F and Ga	no	yes	variable	no	no	yes	no	variable
C present at rod sheaths	---	yes	---	no	---	no	---	no
N-amount [atom %]	5	0	3	0	5	0	3	0
F-amount [atom %]	1	1.5	7	2	0.5	4	2-3	~1
Ca:P:O ratio [atom %]	10:06:25	10:06:25	10:06:24	10:07:29	10:07:27	10:06:21	10:07:21	10:06:21

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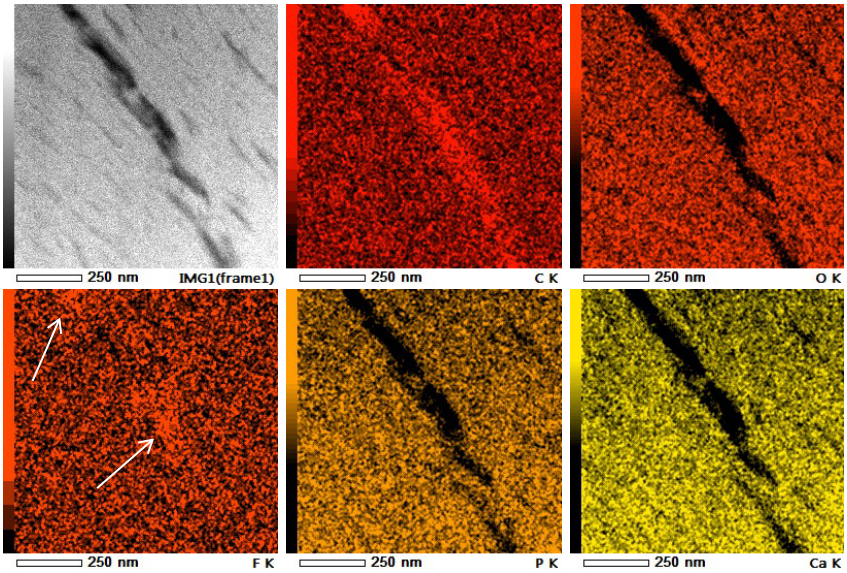
EDS Mappings of Enamel Samples

Appendix figure 1. Raw counts EDS mappings of untreated enamel (intensities represent counts).

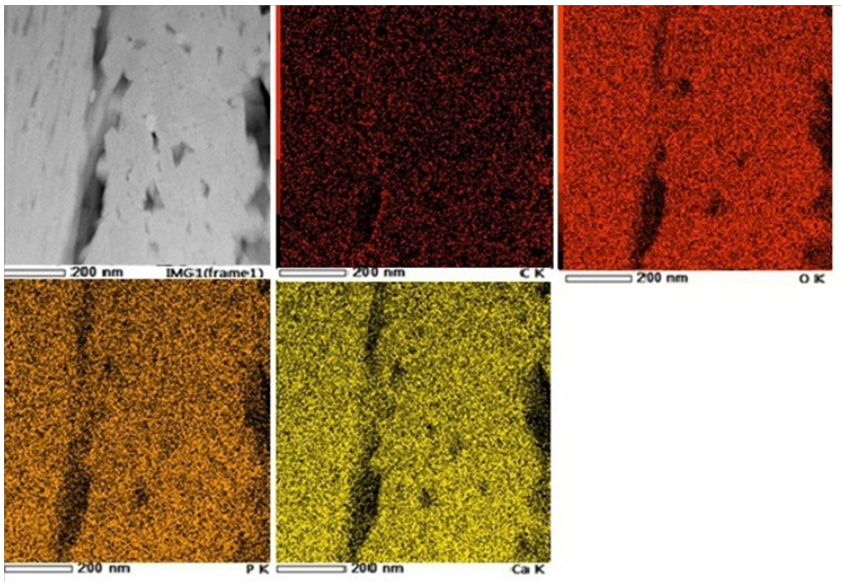


256*256 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 269 frames.

Observations:

A small amount (<0.5 atom%) of Mg , about 1 atom% Na were found.
F appeared to be concentrated at two locations at the interface between two adjacent prisms (see arrows).

Appendix Figure 2. Raw counts EDS mappings of enamel treated with 30% HP, pH 3 (intensities represent counts).

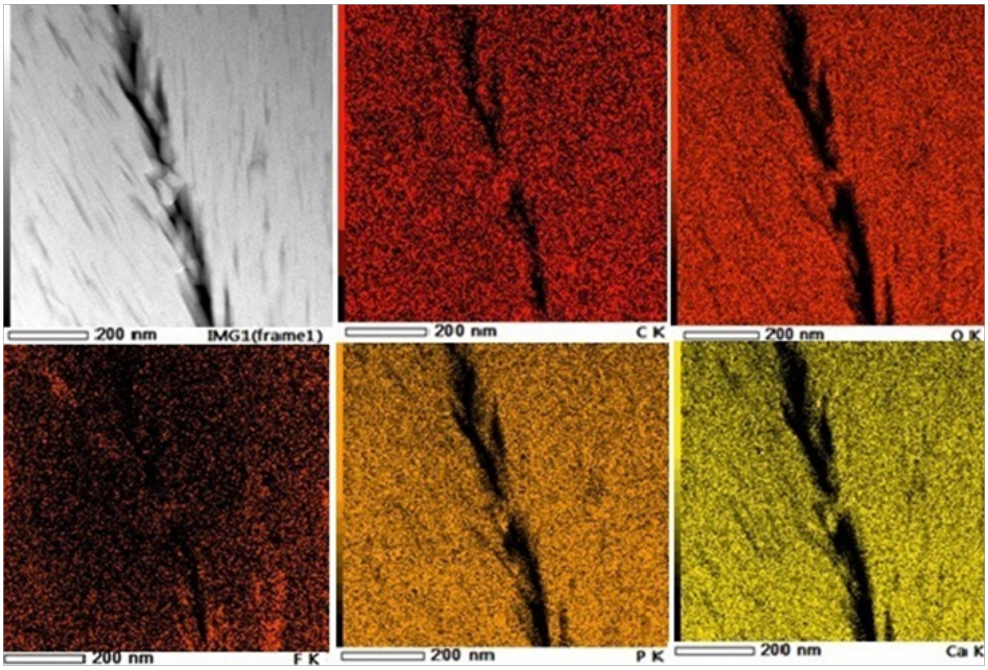


512*512 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 31 frames.

Observations:

Small amounts (<0.5 atom%) of Mg and Cl and about 1 atom% Na were detected.

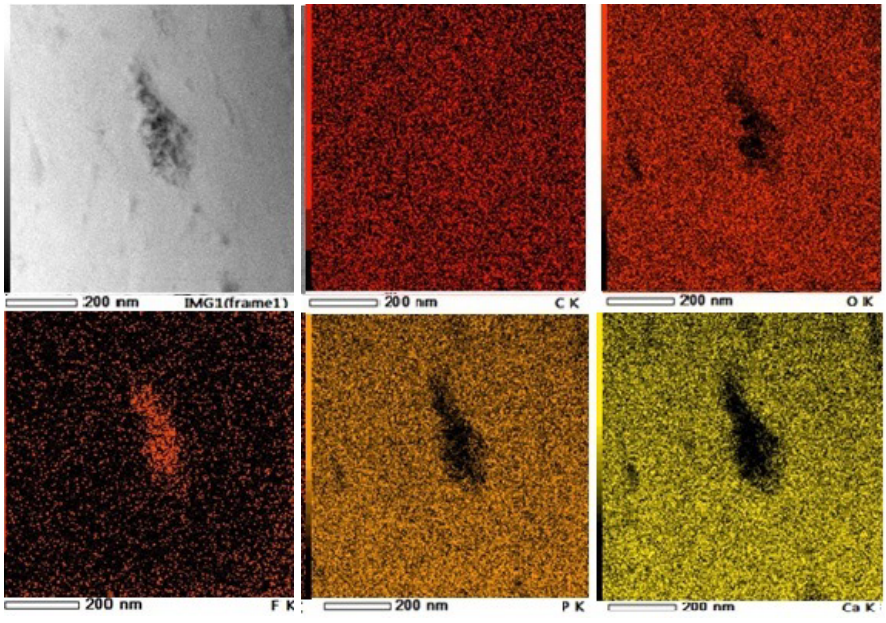
Appendix Figure 3. Raw counts EDS mappings of enamel treated with 30% HP, pH 7 (intensities represent counts).



512*512 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 61 frames.

Observations:
An average F amount of 2.5 atom% was found.

Appendix Figure 4. Raw Counts EDS mappings of enamel treated with NaOH (intensities represent counts).

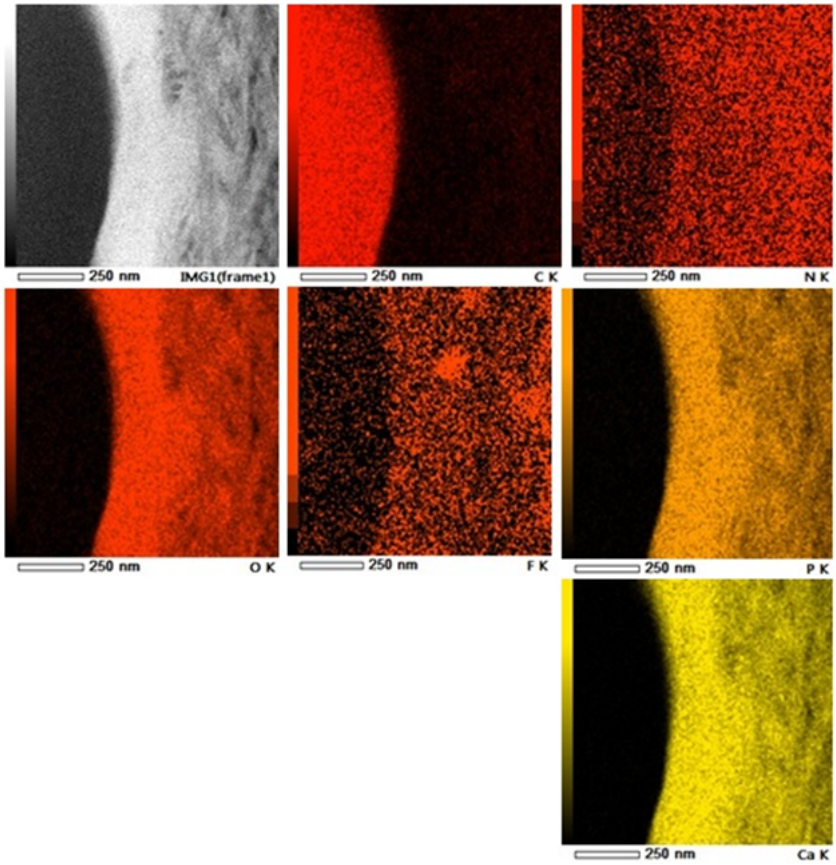


512*512 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 64 frames.

Observations:
Small amounts (<0.5 atom%) of Mg and Cl and about 1 atom% Na were observed.
About 1 atom% of F was found in the region analyzed. The F appeared to be concentrated at the location of the porous material.

EDS Mappings of Dentine Samples

Appendix Figure 5. Raw counts EDS mappings of dentine treated with 30% HP, pH 3 (intensities represent counts).

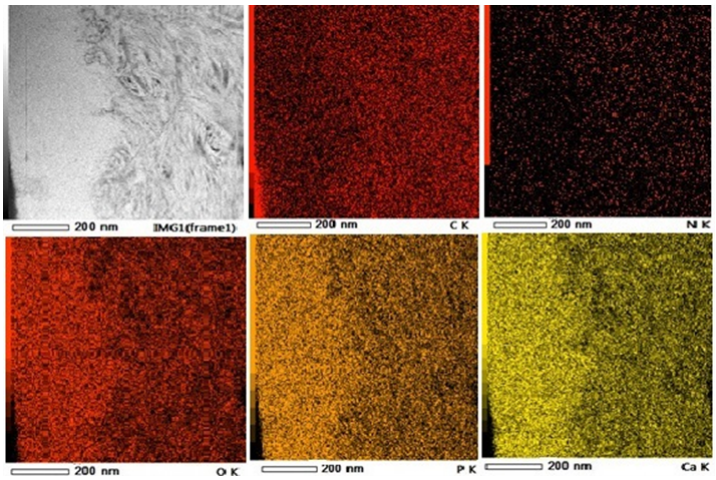


256*256 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 219 frames.

Observations:

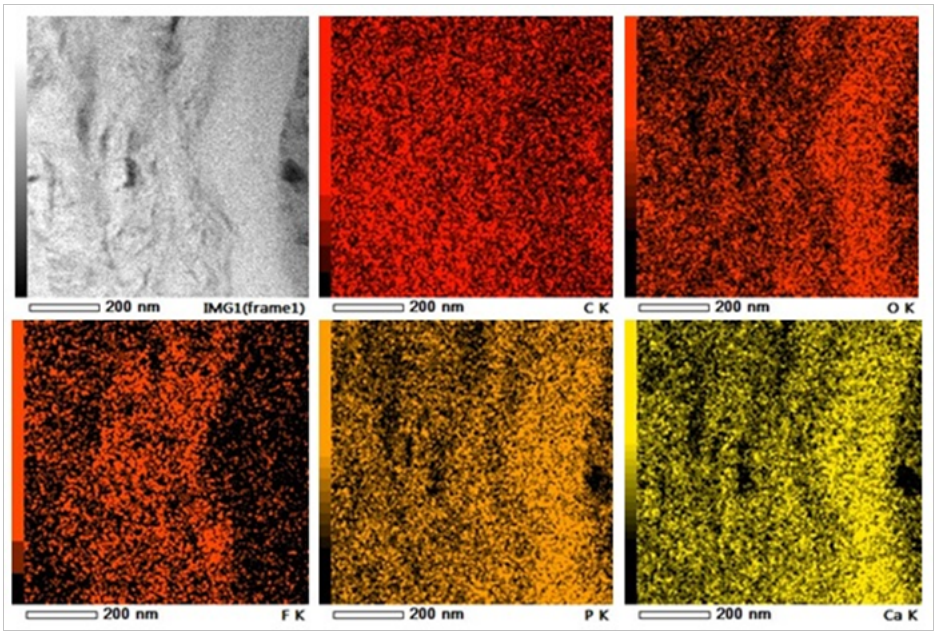
More C was found in the inter-tubular region than in the peri-tubular region.
Small amounts (~0.5 atom%) of Mg and Na were found.
About 5 atom% N was found in the entire region and comparatively less N was present (~1 atom%) in the denser material around the tubule.
About 0.5 atom% of F is found in the region analyzed (the highest intensity in the map corresponds with 2.5 atom%).

Appendix Figure 6. Raw counts EDS mappings of dentine treated with 30% HP, pH 7 (intensities represent counts).



512*512 pixels full EDS spectrum acquisition.
0.1 msec, pixel acquisition time.
Summation over 85 frames.

Appendix figure 7. Raw counts EDS mappings of dentine treated with NaOH (intensities represent counts).



256*256 pixels full EDS spectrum acquisition (3x3L filter).
0.1 msec/pixel acquisition time.
Summation over 127 frames.

Observations:

Small amounts (~1 atom%) of Mg and Na and about 3 atom% N were detected.