

# Surface of root cementum following air-polishing with bioactive hydroxyapatite (Ca and P mapping). A pilot study

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Air-polishing is a well-known and common technique to remove plaque, tartar and different kind of stains from teeth, using abrasive materials. Commonly used cleaning powders contain Al (aluminium) which is still controversial in terms of its inertness and harmfulness for human body. Nowadays, new air-polishing materials, including biomaterials, are being introduced. In particular, biomaterials the structure of which imitates that of natural tissue are very promising materials of reparative and reconstructive features. The purpose of the study was to assess in vitro the influence of natural hydroxyapatite on cementum surface and to assess superficial qualitative distribution of such elements as calcium and phosphorus before and after air-polishing. Four teeth extracted for periodontal reasons were air-polished. Bioactive hydroxyapatite (prepared in the Cracow Institute of Technology) was a cleaning powder with particle size of up to 10  $\mu\text{m}$ . Bioactive natural hydroxyapatite is a very effective cleaning powder, which removes efficiently tartar from cementum surface and does not cause any damage. The qualitative analysis of cementum images after air-polishing with natural hydroxyapatite showed that the cementum surface was fully saturated with such elements as calcium and phosphorus, which was not observed on control cementum images.

*Key words: air-polishing, biomaterials, natural hydroxyapatite*

## 1. Introduction

The utilization of air-abrasion for cavity preparation was first described by BLACK [1]. The author describes the technique as a minimally invasive process of dental air-abrasion with alumina which involves the acceleration of abrasive particles in a stream of compressed gas directed to the tooth through a nozzle [1]. It may be compared with air-abrasion (airbrasive technology) which was first introduced as an alternative means of cutting dental tissue.

Contrary to air-abrasion, air-polishing technique was introduced into dentistry to remove plaque, tartar and stains from dental tissue. BOYDE [2] describes

“air-polishing” as the abrasive material largely water soluble, since it contains a high proportion of sodium bicarbonate and that material is widely used for this purposes till now. Sodium bicarbonate is a soft material and its eroding particles are not hard or sharp.

The conventional sodium bicarbonate powder ( $\text{NaHCO}_3$ ) is regarded as high-level abrasive material that meets presently valid standards set for air-polishing technique. The application of the abrasive jet consisting of  $\text{NaHCO}_3$  powder, water and pressurized air to root surfaces may, however, result in severe substance removal within short application time. The use of the air-polishing technique in the case of denuded root surfaces is contraindicated, as  $\text{NaHCO}_3$  powders remove clinically relevant amounts of root

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cementum or dentin within short instrumentation times. Defect depths lower than 630  $\mu\text{m}$  after 30 s in vitro instrumentation may affect root dentin [3].

A powder consisting of crystals of amino acid glycine (Clinpro Prophypowder, 3M ESPE) instead of the conventional sodium bicarbonate powder reduces the abrasiveness on root surfaces by approximately 80%.

PETERSILKA [4] describes an in vitro evaluation of novel low abrasive air-polishing powders and reports that one of evaluated powders (powder D) can be used for plaque removal from denuded root surfaces or dentine without causing relevant damage to the root, even if the instrument tip is held close to the surface to be treated.

Air-polishing applied to enamel surfaces caused no detectable change in the microscopic surface structure, even if the treatment was continued for the periods of 15–30 s. According to BOYDE's hypothesis concerning the mechanism of air-polishing abrasion of enamel, the enamel without discontinuities is not affected by the technique [2].

Hydroxyapatite (HA) is biocompatible, osteoconductive, non-toxic, noninflammatory, no immunogenic and bioactive agent, i.e., it has the ability to form a direct chemical bond with living tissues [5]–[6]. Hydroxyapatite has been used in orthopaedics, stomatology, dental and facial surgery, ophthalmology, laryngology and traumatology [7]–[8]. Hydroxyapatite bioceramics in medical practice could be applied in the following forms: powders, granules, compact or porous materials, composites or films on different kind of surface [9]–[10]. Hydroxyapatite powders and granules are used in dentistry in dental pulp therapy (as dental cements), in the case of dentine hypersensitivity and defect of enamel. HA could also be applied as root canal sealer and cleaning agent [11].

In the present paper, we examined the abrasiveness of natural hydroxyapatite (N-HA) and its impact on enamel and cementum in air-polishing technique. Both powder and hard dental tissues after N-HA application were examined by scanning electron microscopy and X-ray microanalysis.

## 2. Materials and methods

### 2.1. Collection and processing of teeth

6 extracted premolars were collected for a pilot study (3 were extracted for orthodontic reasons and used to assess enamel before and after sandblasting – 2 test teeth and 1 control, and 3 were extracted for

periodontal reasons to evaluate air-polished cementum).

Prior to airpolishing, the extracted teeth were rinsed with water but all soft tissue remnants and tartar were left for the procedure. One third of tooth root's aspect was mounted in separate plaster blocks to have only one tooth exposed to air-polishing with one sandblast release.

### 2.2. Experimental natural biomaterial used as an air-polishing powder

Preparation method of natural hydroxyapatite was developed at the Institute of Inorganic Chemistry and Technology, Cracow University of Technology [12]. A deproteinised and defatted bone pulp, called "bone sludge", was applied as a raw material. Hydroxyapatite was obtained in two-stage calcining process at temperatures of 600 °C and 850 °C, respectively, for each stage. Material was calcined for 3 hours at maximum temperature. Calcining process took place in chamber kiln with electric heating in air atmosphere. In all the tests, sieve fraction did not exceed 0.063 mm. The size of natural hydroxyapatite particles applied in our research was estimated and it ranged from 1 to 10  $\mu\text{m}$ .

### 2.3. Sandblasting of enamel and cementum surfaces and the correlation between backscatter electron microscopic images and the energy dispersive X-ray diffraction

Prophy-Mate (NSK) removable tip with 60-degree nozzle angle was used after adjustment to standard dental unit facilities. It was connected to the joint of high-speed manual machine of complex treatment unit, the air source (0.3–0.4 MPa) and water source (0.2–0.4 MPa) of complex treatment unit with control air pressure and the water pressure of Prophy-Mate.

The distance and angulations of the instrument nozzle (0.8-mm diameter) were adjusted to enamel or cementum surface, with the jet being directed to coronal aspect of the teeth to imitate working conditions of airpolishing technique in the case of potential danger of soft tissue damage. The air-polishing technique was applied manually from the working distance of approximately 5 mm. There were 4 releases of air with water and powder for each tooth, 2 of them were directed to enamel and 2 of them to cementum.

Then sandblasted surfaces of enamel and cementum were covered with Au-Pd layer to evaluate and analyze their chemical composition.

## 2.4. SEM images evaluation and X-ray chemical microanalysis

The microstructure of samples was examined using S-4700 Hitachi scanning electron microscope. Chemical analysis was carried out using an energy dispersive X-ray spectroscope (EDX) at 20.0 kV and 15.0 mA. Particle size was estimated on the basis of SEM pictures.

## 2.5. Chemical analyses of natural hydroxyapatite used as air-polishing powder

The phosphorus content in samples was determined by the spectrophotometric method with a Marcel Media UV-VIS spectrophotometer after former their mineralization in a mixture of concentrated hydrochloric and nitric acids. The determination is based on the formation of a yellow-coloured phosphorus–vanadium–molybdenum complex and a photometric measurement of absorbance at a wavelength of 430 nm.

Calcium concentration was determined by the titration method. First, the sample is dissolved in nitric acid, then phosphates are precipitated as bismuth(III) phosphate(V) ( $\text{BiPO}_4$ ), and finally the calcium content can be determined by complexometric titration with disodium versenate (EDTA) in the presence of a mixed indicator (calcein and thymolphthalein).

Microelements such as Cu, Co, Ni, Cr and Cd were mineralized in concentrated nitric acid and then their contents determined by the AAS method with Analyst 300 Perkin Elmer Spectrometer. Contents of

heavy metals such as Pb, Hg and As were measured using the ICP (Inductively Coupled Plasma) – ELAN 6100 Perkin Elmer.

## 2.6. Infrared and X-ray analyses of N-HA

Phase composition was analysed using X-ray method with Philips X'Pert diffractometer with graphite monochromator using  $\text{Cu K}\alpha$  1.54, Ni filter (40 kV, 30 mA).

FT-IR spectroscopic investigations were conducted with spectrophotometer Scimitar Series FTS 2000 Digilab in the range of middle infrared 400–4000  $\text{cm}^{-1}$ . 0.0007 g sample and 0.2000 g of KBr were pressed together. In these measurements, the number of scans was 16 and the resolution, 4  $\text{cm}^{-1}$ .

## 3. Results

### 3.1. X-ray microanalysis of N-HA

A natural hydroxyapatite is composed of grains of regular shape, close to sphere, with the diameter of approximately 1–10  $\mu\text{m}$ . It could be noticed that particular grains were sintered.

Figure 1 presents the distribution of chemical elements on the hydroxyapatite surface. These images demonstrate very good homogeneity of the powder surface under analysis. The distribution of chemical elements proved to be almost uniform.

Typical EDX spectrum representing various elements present in N-HA is shown in figure 2. The analysis indicates that inorganic phases of bone are composed of calcium and phosphorus as the major constituents and some minor components such as C, O, Na, Mg and Si.

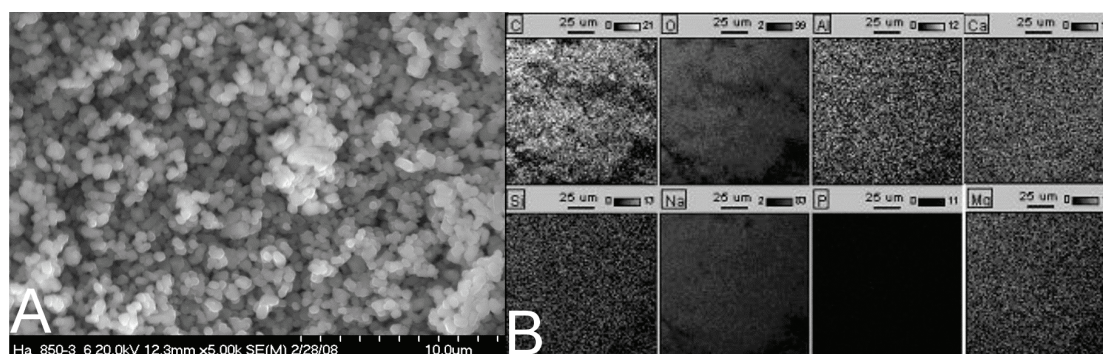


Fig. 1. SEM image of natural hydroxyapatite surface (a), chemical elements' distribution on hydroxyapatite powder surface (b)

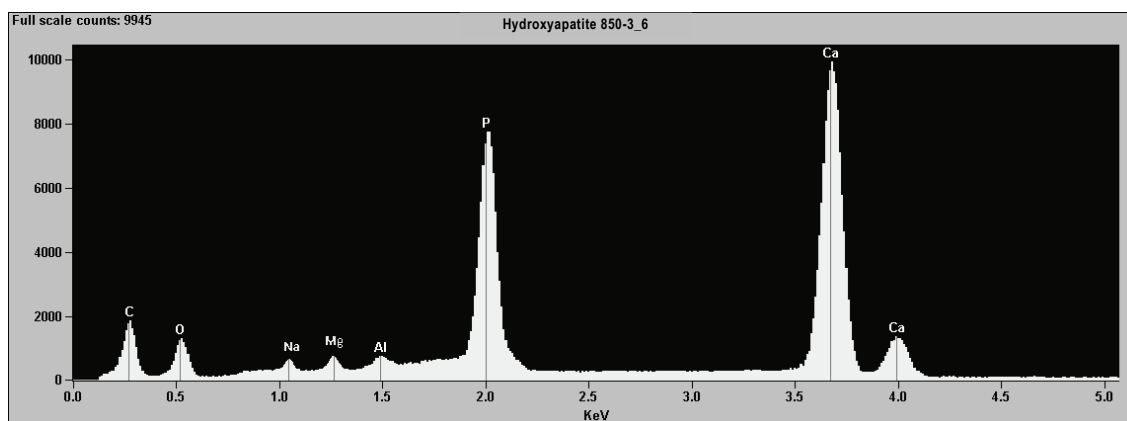


Fig. 2. EDX spectrum of N-HA powder

### 3.2. Chemical analyses of natural hydroxyapatite

Table 1 presents chemical composition of a natural hydroxyapatite. The content of calcium and phosphorus in this mineral was lower than that in synthetic, stoichiometric hydroxyapatite; however, the molar ratio of Ca/P was close to stoichiometric value (1.67). The lowest content of two main components in bone-hydroxyapatite could result from the occurrence of such microelements as magnesium, sodium and also carbonate group, which are natural constituents of bone tissue.

Table 1. Chemical composition of hydroxyapatite

Element	Unit	Natural hydroxyapatite	ISO 13779:2008
Calcium	%	38.77	–
Phosphorus	%	17.83	–
Molar ratio of Ca/P		1.68	1.65–1.85
Phosphorus soluble in hydrochloric acid	%	3.19	–
Phosphorus soluble in citric acid	%	2.64	–
Cobalt	ppm	7.88	–
Copper	ppm	6.09	–
Nickel	ppm	10.72	–
Chromium	ppm	9.50	–
Cadmium	ppm	1.00	1.50
Lead	ppm	0.59	10.0
Mercury	ppm	0.0043	1.50
Arsenic	ppm	0.19	1.00

The content of all chemical elements, including heavy metals, was found to be within the standard limits [13].

### 3.3. X-ray and FT-IR study

Figure 3 presents X-ray diffraction pattern of natural hydroxyapatite. X-ray analysis shows that hydroxyapatite is the only crystalline phase observed in this material.

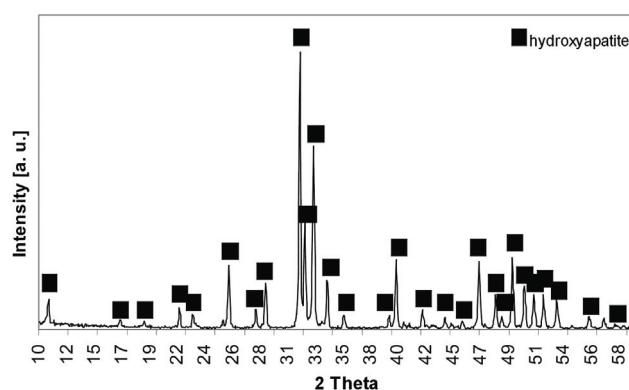


Fig. 3. X-ray pattern of N-HA powder

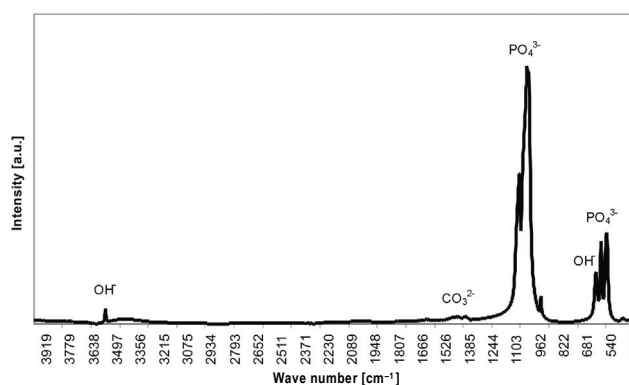


Fig. 4. FT-IR spectrum of N-HA powder

The FT-IR spectrum of natural hydroxyapatite shown in figure 4 exhibited only the characteristic

absorption peaks of hydroxyapatite. Absorption bands were ascribed to particular bonds according to [14]–[15]. Vibrations of O–P–O bonds were assigned to the absorption bands within the wave number ranges of 570–636  $\text{cm}^{-1}$ . The most intensive bands in the range of 1047–1095  $\text{cm}^{-1}$  corresponded to asymmetric stretching vibrations of P–O, whereas maximum of absorption at wave numbers of 964  $\text{cm}^{-1}$  came from symmetric vibration of P–O. The band with high wave number range resulted from O–H bond vibration. The low-intensity band at 1427  $\text{cm}^{-1}$  observed could correspond to the stretching vibrations of  $\text{CO}_3^{2-}$ . In the spectrum, there were no bands corresponding to the vibrations of C–H and C–C bonds presented in organic compounds. It was confirmed that the whole organic material was removed after the second stage of calcining process.

### 3.4. X-ray microanalysis of sandblasted enamel and cementum surfaces

The EDX mapping was attempted to show that untreated enamel surface is almost the same as the enamel surfaces abraded with N-HA. The concentration of calcium and the concentration of phosphorous seem to be very similar on both mapping images (table 2). A quantitative chemical microanalysis of enamel surface of 2 teeth (samples 2 and 3) after air-polishing, compared to non-abraded enamel surface, reveals almost no changes or a slight change in its chemical composition of Ca and P (table 3).

Table 2. Phosphorus and calcium content in enamel without air-polishing

Enamel	Sample 1
P	28.12%
Ca	71.88%
P	28.48%
Ca	71.52%
P	28.76%
Ca	71.24%

EDX mapping and backscattered electron microscopy demonstrated cementum surface modification and substantial benefits from surface debris. The SEM image (figure 7, upper left square) shows tartar on cementum surface that was removed using air-polishing technique with N-HA (figure 8, upper left square). The cementum surface after cleaning with N-HA

seems to have no damages, no cracks but some grooves may be observed.

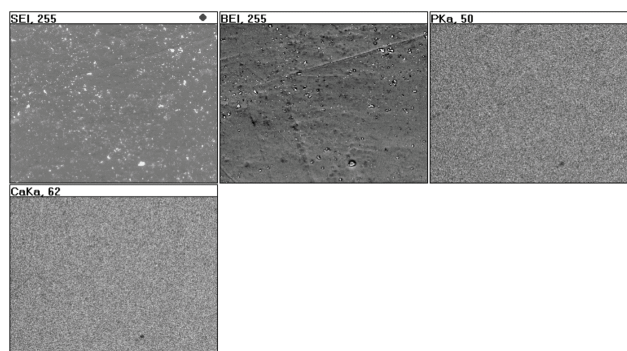


Fig. 5. Chemical elements' distribution on enamel without air-polishing, 500 $\times$

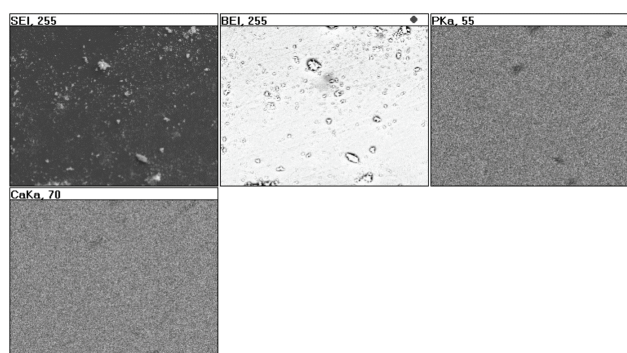


Fig. 6. Chemical elements' distribution on enamel, N-HA, 500 $\times$

Table 3. Phosphorus and calcium content in enamel after air-polishing with N-HA

Enamel	Sample 2	Sample 3
P	28.00%	29.00%
Ca	72.00%	71.00%
P	28.00%	29.51%
Ca	72.00%	70.49%
P	28.39%	28.46%
Ca	71.61%	71.54%

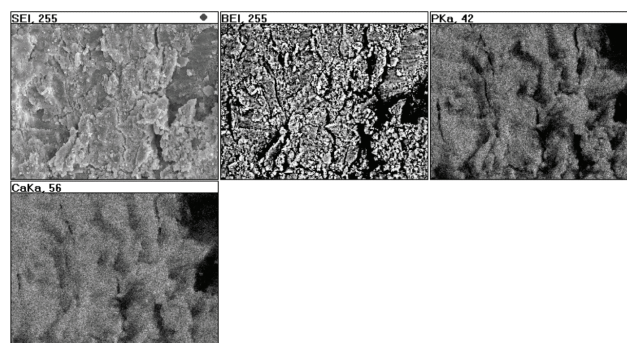


Fig. 7. Chemical elements' distribution on cementum without air-polishing, 500 $\times$

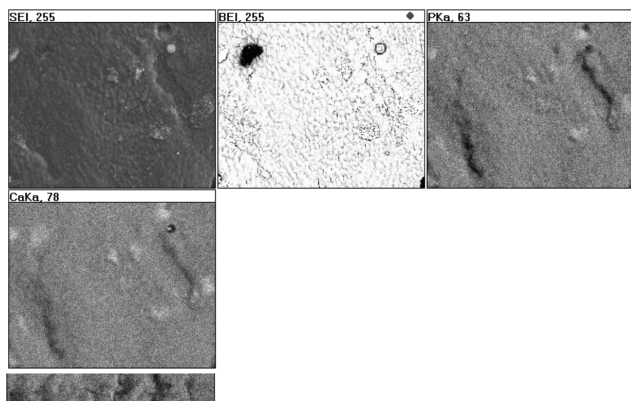


Fig. 8. Chemical elements' distribution on cementum, N-HA, 500 $\times$

The concentration of Ca and P is different when air-polishing was applied (tables 4 and 5). In contrast to mapping images, where air-polishing was not applied to the cementum, the surface of cementum subjected to N-NA abrading demonstrated regular concentration of Ca and P.

Table 4. Phosphorus and calcium content in cement without air-polishing

Cement	Sample 4
P	22.55%
Ca	77.45%
P	21.70%
Ca	78.30%
P	24.07%
Ca	75.93%

Table 5. Phosphorus and calcium content in cement after air-polishing with N-HA

Cement	Sample 5	Sample 6
P	28.17%	26.11%
Ca	71.83%	73.89%
P	25.71%	26.71%
Ca	74.29%	73.29%
P	24.31%	27.14%
Ca	75.69%	72.86%

The results of quantitative chemical microanalysis of localized cementum surface significantly differ from the results obtained for enamel surface analysis, both when the surfaces are and are not abraded.

The localized quantitative mapping of non-abraded cementum surface (figure 7) is different when measured in various places, in contrast to measurements carried out for enamel. The content of P was higher in enamel compared with cementum, and the concentration of Ca was much higher on non-abraded cementum surfaces, compared with enamel.

The concentration of P on abraded cementum surface is significantly higher compared with untreated one. The results are completely different when Ca is analysed, as in some cementum areas, its concentration significantly decreases after the application of N-HA with air-abrasion.

Analysing the localized quantitative mapping measurements of any surface it should be remembered that the results in percentage are divided between the chemical elements analyzed and as a whole they represent 100% and in this way they should be interpreted.

## 4. Discussion

In the present in vitro study, our hypothesis that air-polishing when applied to enamel surfaces causes change being no detectable in SEM images and enamel is thus unaffected by technique is confirmed in the case of the natural hydroxyapatite used as cleaning powder (figures 5–8). Obviously we have to take under consideration such features as the abrasion time, emitted powder flow rate at the nozzle tip and propellant pressure [16]–[17].

Previously published studies have suggested that even at moderate powder and water settings, the application of APDs (air-polishing devices) using conventional  $\text{NaHCO}_3$  powder may cause substantial substance loss [18]. Furthermore, root defects following air-polishing with  $\text{NaHCO}_3$  powder for various periods of time have been reported, e.g., the defects of  $484 \pm 196 \mu\text{m}$  after 5 s [19],  $163.1 \pm 71.1 \mu\text{m}$  after 20 s [4], and  $636.6 \pm 103.6 \mu\text{m}$  after 30 s [20]. Currently, in air-polishing system we use glycine, which less infiltrates tissue and is softer than the conventional  $\text{NaHCO}_3$ , hence it erodes the root surface significantly less, i.e.,  $33.9 \pm 19.6 \mu\text{m}$  after 20 s of polishing [21].

There is a tendency to replace the currently used air-polishing abrasives with biocompatible, osteoconductive, bioactive and non-toxic materials (potentially toxic alumina is sometimes added to air-polishing powders [22]), which are supposed not only to remove tartar and stains from tooth surface without any damage, but also to remineralise dental tissue, improving its chemical composition. We showed that N-HA can be utilised, to some degree, as air-polishing powder.

EDXA mapping and backscattered electron microscopy demonstrated the retention of N-HA on cementum surface. Abraded cementum surface was

modified and showed substantial benefits from surface debris. Although the N-HA particles clean the cementum surface, some fraction of the airbrasive is retained on the surface after impingement, which is confirmed by changes in chemical composition followed air-polishing (figure 8).

Any cementum surface damages were observed after applying air-polishing technique.

On the contrary, electron microscopy did not visualize any changes on enamel surface followed air-polishing (figure 6) and chemical analysis of enamel confirmed the results of SEM images, giving just a minor difference compared with a control data (figure 5).

Such results may be achieved because N-HA is a soft polishing material, and enamel is a much harder dental tissue than cementum.

It was found that 1–10  $\mu\text{m}$  particles of the N-HA used in this research within abrasion blend were responsible for the formation of agglomerates which clogged nozzles. There was a relatively high level of dusting in a room followed the air-polishing procedure.

This may lead to conclusion that both size and shape of particles involved in the studies should be changed.

## 5. Conclusion

1. The abraded enamel surface seems to be unaffected on a microscopic scale and almost unchanged in its chemical composition of Ca and P. This may lead to a conclusion that the material used for air-polishing is either too soft or the pressure applied too low, or both factors occur simultaneously, to change hard enamel tissue. However, a clinical in vitro assessment reveals that plaque and discolorations were removed from enamel. On the contrary, abraded cementum surface was modified which was confirmed by chemical composition followed air-polishing. This allowed us to infer that the pressure applied and physical properties of the powder were efficient and appropriately selected, which enabled cementum cleaning and remineralization. On the other hand, they have to be changed in order to improve enamel tissue. However, the time of powder effect on teeth and its resistance to being washed out of tooth surface still need to be assessed in vitro.

2. A natural hydroxyapatite used as a cleaning powder does not cause the clogging of 0.8 mm nozzle of Prophy Mate head, but considerable amount of dust is emitted in the place of its application. For this rea-

son, diameter and form of the nozzle have to be properly selected.

3. The surface of enamel and cementum is covered with a layer, which we call a “film” because it cannot be removed using water spray from a dental unit. Assuming that enrichment of tooth surface with natural hydroxyapatite is a beneficial phenomenon, the presence of the “film” can confirm this fact. High saturation of superficial enamel and cementum layers with such elements as Ca and P following air-polishing supports natural remineralization of tooth hard tissue.

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