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Original Research Article

Atomic density of elements on the surface of orthodontic bands

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ABSTRACT

The study was performed on new and used (from *in vitro* and *in vivo* experiments) orthodontic bands by using SEM-EDX technique. The bands were retrieved from earlier experiments: *in vitro* tests, in which the bands were incubated in a continuous flow system with various media: artificial saliva, lysozyme, orange juice and Coca Cola®. The bands were also retrieved from previously conducted *in vivo* tests on animals (pigs) and humans (patients). The micrographs of bands were presented as well as their chemical composition, reported in terms of atomic density. The bands that were used showed a significant contribution of oxygen as compared to brand new ones, and the contribution of Fe and Ni decreased, whereas the Cr contribution remained unchanged. The elements were inter-correlated. An antagonistic, statistically significant dependence was found between Fe and O, as well as between Fe and Cr. This could signify that that protective passivation layer of Cr_2O_3 was formed, which did not fully protect Ni and Fe from dissolution.

1. Introduction

Metallic devices (brackets, wires and bands) which constitute the orthodontic fixed appliances are held in the oral cavity for 2 years on average treatment. Corrosion is one of the major concerns related to biocompatibility of metallic biomaterials, because impacts the release of ions [1,2]. Chemical composition has very much to do with corrosion. Orthodontic stainless steel alloys usually contain Fe, Cr, Ni, Co, Si which can be transferred to the human organism during corrosion [3–5]. Some of the elements mentioned above are known as cytototoxic, mutagenic and sensitizing agents [6]. High degree of biocompatibility is being expected from orthodontic materials because of prolonged contact with the surrounding tissues.

The existing ISO standards are not obligatory for manufactures and the companies can create their own standards. Resulting, it is reported that some manufacturers do not pay sufficient attention to the final processing stages (finishing). The consequence could be the lower biocompatibility [7].

The evaluation of the characteristics of orthodontic materials is an important step in understanding the mechanisms of metal ions release under *in vitro* and *in vivo* conditions. Various properties play an important role in the search for ideal materials (*e.g.* biostability). Surface properties (roughness, surface topography, elemental composition) affect the biocompatibility and the performance, as well as

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corrosion and esthetics. Moreover, surface roughness influences plaque accumulation [8]. Orthodontic alloys are exposed to various substances in oral cavity: saliva which becomes acidic in contact with food and activity of microflora in biofilm (biodeterioration) [6].

Metallic orthodontic devices with a high surface area exposed to the oral environment can cause a problem because of the possibility of adverse biological effects due to the release of heavy metal ions (corrosion and wear) [9].

The biocompatibility of the material is related to the passive film present on the surface. Chromium forms a thin and adherent oxide Cr_2O_3-based passive layer that provides corrosion resistance by blocking the diffusion of oxygen into the basic bulk alloy [3,6]. A minimum of 12 percent of chromium is bound to transmitting the necessary corrosion resistance [10]. This factor is essential for the preservation of biological materials. Changes in the passive layer make material susceptible to corrosion [11].

The aim of the present study was evaluation of atomic density of elements on the surface of orthodontic bands by the SEM-EDX technique exposed to various conditions of *in vitro* and *in vivo* (patients, animals) experiments.

2. Materials and methods

2.1. *Tested material*

The evaluated materials were new and used orthodontic bands (size 37+; 3M Unitek, Monrovia, CA, USA). The latter were obtained from the orthodontic appliances of three series of experiments (*in vitro* tests with various solutions, animal tests and *in vivo* tests on humans). Wires, brackets and bands were all made of stainless steel. The chemical composition (%) of bands, provided by the manufacturer was: 65 Fe, 17 Cr, 12 Ni, 2.5 Mo, 2 Mn, 1 Si, 0.045 P, 0.03 C, 0.03 S.

2.2. In vitro *tests*

Four series of *in vitro* tests were performed (with (I) artificial saliva, (II) artificial saliva with lysozyme, (III) orange juice and artificial saliva, (IV) Coca Cola® and artificial saliva). The orthodontic appliance which consisted of two wires, four bands, 20 brackets and 20 elastic ligatures, were placed in the thermostatic glass reactor assuring the conditions of continuous flow of artificial saliva, and incubated at 37 °C for 28 days. In the first and second series, artificial saliva and artificial saliva with lysozyme, respectively, was flowing through the system with the flow rate reflecting the flow of saliva in the human oral cavity (0.5 mL/min). The details of the experiment and *in vitro* system were described in previous studies [12]. In the third and fourth series, orange juice and Coca Cola® (330 mL), respectively, were flowing through the system for 5.5 h (1.0 mL/min) every day, while artificial saliva – for the rest of day (0.5 mL/min) [13].

2.3. In vivo *test on animals*

The animal experiment was conducted on pigs, chosen as a model organism. The plates that aimed to simulate the orthodontic appliance were made of bands and were placed on the buccal side of pig's cheek for 6 months. The details of the experiment were described earlier [14].

2.4. In vivo *test on humans*

Used bands were collected from orthodontic patients after 12 months of treatment. The details of the experiment were described earlier [15].

2.5. *Analytical methods*

The external surface of the new and used orthodontic bands was evaluated by the SEM-EDX technique. Before SEM-EDX analysis, all samples were degreased with ethanol. Samples were mounted on an appropriate stub and were subjected to Roentgen microanalysis using the Phenom ProX desktop scanning electron microscope with BSD detector, operating at EHT = 15 kV. For each orthodontic band five analyses were performed.

2.6. *Statistical methods*

The results were elaborated statistically by *Statistica ver. 10.0*. Descriptive statistics (means, standard deviations) were reported. The normality of distribution of the experimental results was assessed by the Shapiro–Wilk test. Statistical differences between new and used bands were assessed by the Tukey test and Kruskal–Wallis test. Results were considered significantly different when $p < 0.05$.

3. Results and discussion

Evaluated bands were retrieved from the experiments conducted earlier: *in vitro* (artificial saliva, artificial saliva with orange juice or Coca Cola®) and *in vivo* (patients, animals – pigs). Previously, metal ions release in these studies was discussed. The total mass of released metal ions during 4 weeks of the *in vitro* test in the continuous flow system (in the environment of artificial saliva) was: nickel 18.7 μg, chromium 5.47 μg and copper 31.3 μg [12]. Similar experiments were conducted with the use of soft drinks (orange juice and Coca Cola®, respectively). The total mass of ions released was, μg: Ni (15.33; 37.75), Cr (3.604; 1.052), Fe (48.42; ≥156.1), Cu (57.87, 32.91), Mn (9.164; 41.16), Mo (9.999; 30.12), Cd (0.5967; 2.173). It was found that orange juice did not intensify the release of metal ions from orthodontic appliances, whereas Coca Cola® caused increased release of Ni ions [13]. *In vivo* experiments (conducted on animals) revealed that Ni and Cr were released and accumulated in various tissues. The sites of accumulation were: aorta (4.8 times higher of Ni), in the cheek (Ni 3.5 times higher), and in the hair sampled after 3 months (Cr 3.4 times higher), as related to the control group. The doses of toxic metal ions released from the appliance did not reach toxic levels [14]. The trials on patients were conducted. Hair was sampled in time (1 year period) as a non-invasive biomarker of exposure. The following masses of ions were transferred to hair tissue: 7.42 ± 14.19 μg of Ni, 8.94 ± 13.1 μg of Cr, and 131 ± 279 μg of Fe. The content of Cr was statistically significantly