

Influence of self-made saliva substitutes on tribological characteristics of human enamel

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This paper describes the results of tests on the influence of human saliva and its substitutes on tribological characteristics of friction pairs. Each pair consists of enamel and one of the following materials: ceramics, the Meridian B2 dental composite, the GK dental amalgam, and Ti-6Al-4V titanium alloy. The saliva substitutes used were prepared using pyrophosphates, xanthan gum, and mucins dissolved in a saline buffer. The results of the tribological tests show that the values of the parameters under investigation (coefficient of friction and linear wear) were different from each other. Some similarity was observed between the evaluated level of wear characteristics after the friction process in the environment of human saliva and that in the environment of one of the mucins tested. Microscopic observations of the surfaces of the enamel samples after friction revealed varied forms of tribological wear.

Key words: coefficient of friction, enamel, human saliva, linear wear, saliva substitute

1. Introduction

Human saliva is an important body fluid consisting mainly of water and a small amount of organic and inorganic ingredients, thanks to which it fulfills many important functions in the human body. These are, among others: wetting of the mucous membranes and “lubrication” of natural tooth-tooth friction pairs [1] or friction pairs with dental filling materials and prosthodontic elements. It also plays a significant role in processes such as mastication, swallowing, and digestion of food, as well as correct pronunciation [2]–[7].

Disorders of secretion of human saliva may lead to health problems in patients related to dryness of mouth (e.g., hyposalivation or xerostomia). One of the methods of treatment of these disorders is to apply, e.g., lubricants (vaseline or glycerin) or preparations

substituting saliva in the form of fluids or gels [5], [8]. It should, however, be noted that commercial saliva substitutes should improve the comfort of life of sick persons and also positively impact the utilitarian properties of dental fillings and of ceramic and metallic biomaterials in the oral cavity. Studies concerning the creation of new substitutes have been ongoing for many years. Analysis of biochemical, physical, chemical, mechanical, and tribological properties of human saliva and its commercial substitutes that are currently offered on the pharmaceutical market, makes artificial substitutes more and more similar to natural saliva in terms of their properties. Unfortunately, the saliva substitutes that are the fruits of these works are still far from the original. Patients complain of the short duration of the effect, bad taste and consistency, and low effectiveness in cases where it is used to facilitate talking, swallowing, or chewing. That is why numerous studies are undertaken in order to search for

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preparations to substitute saliva that would be characterized by beneficial general properties in comparison to those currently being used. These studies also include tribological tests, during which the influence of saliva substitutes on, among other things, wear of stomatological materials and elements of dental prosthetics is evaluated [5]–[7]. Studies are also conducted in which wear of tooth enamel, including human enamel, is evaluated. For this purpose, special enamel samples with the shape of a truncated cone are prepared. Such a shape makes it possible to evaluate the volume of wear of the enamel tested [9].

The purpose of this work was to evaluate selected tribological properties (friction coefficient and linear wear) of enamel and stomatological materials under in vitro conditions in the environment of self-made compositions of saliva substitutes.

2. Materials and test methodology

In the research experiments described, human saliva was used besides three saliva substitutes whose compositions had been developed in the Department of Materials and Biomedical Engineering of the Białystok University of Technology. The saliva substitutes had been selected on the basis of their wide use in the pharmaceutical industry (toothpastes, mouthwashes, etc.) The above ingredients are considered to be compounds that have similar physical, chemical, rheological, and tribological properties to human saliva.

Information on the ingredients of self-made compositions of saliva substitutes used for tests in this work is listed in Table 1. Human saliva was also used for tests. In order to provide repeatable test conditions for natural saliva, the previously developed methods of collecting saliva samples were applied [10]:

- saliva was always collected at the same time of day, and tribological tests were conducted as soon as possible following its collection;
- the patient could not eat any meals or drink and fluids for about 1 h before saliva collection;
- the patient had to brush their teeth after their last meal, but not just before saliva collection;
- before saliva collection, the patient had to precisely rinse their mouth with water, and only after 5 minutes could they spit it out into sterile containers (5 ml of saliva were collected for tribological tests);
- collection lasted for a strictly determined time interval, 10 minutes.

Friction tests were conducted using a pin-on-disc tester in an artificial saliva environment. A general view of the research station and the friction pair is shown elsewhere [11]. The tribological tests were conducted with the use of the T-11 type pin-on-disc tester. The friction pair was composed of a pin (enamel) the tip of which was a truncated cone with a slant height angle of 20° and the initial truncated area of approximately 1 mm^2 (as the sample). Such sample geometry makes it possible to obtain great unit pressures at low normal load. The countersample was a disc (medical material), the diameter of which was 25.4 mm.

Table 1. Characteristics of ingredients of self-made substitutes

| Substance | Manufacturer product No. | Characteristics |
|--------------------------------------|--------------------------|--|
| mucin from porcine stomach, type II | Sigma-Aldrich, M2378 | – type II mucin from porcine stomach; – unrefined powder with a cloudy consistency after dissolution; – contains sialic acid $\leq 1.2\%$. |
| mucin from porcine stomach, type III | Sigma-Aldrich, M1778 | – type III mucin from porcine stomach; – powder that is partially refined, with a cloudy consistency after dissolution; – contains sialic acid (0.5–1.5%). |
| xanthan gum | Sigma-Aldrich, G1253 | – mixture of anionic polysaccharides; – white powder giving a gel consistency after dissolution. |
| hydrated tetrasodium pyrophosphate | Sigma-Aldrich, 30411 | – chemical formula: $\text{Na}_4\text{P}_2\text{O}_7 \times 10\text{H}_2\text{O}$; – molecular mass: 446.06 [g/mol]. |
| disodium dihydrogen pyrophosphate | Sigma-Aldrich, 71499 | – chemical formula: $\text{H}_2\text{Na}_2\text{P}_2\text{O}_7$; – molecular mass: 221.94 [g/mol]. |
| tetrapotassium pyrophosphate | Sigma-Aldrich, 322431 | – chemical formula: $\text{K}_4\text{P}_2\text{O}_7$; – molecular mass: 330.34 [g/mol]. |
| buffered saline solution | – | – PBS (pH = 7.0); – contains NaCl, KH_2PO_4 , Na_2HPO_4 . |

Table 2. Materials and lubricants used in the tribological tests

| Friction node | Materials, lubricants |
|-----------------------|--|
| Pin (sample) | – cylinder-shaped samples of enamel |
| Lubricating substance | – human saliva; – Solution A: hydrated tetra-sodium pyrophosphate (Sigma-Aldrich) + Di-sodium dihydrogen phosphate (Sigma-Aldrich) + tetra-potassium pyrophosphate (Sigma-Aldrich) + xanthan gum (Sigma-Aldrich) in PBS (phosphate buffered saline) of pH = 7.0; – Solution B: solution of type II mucins (Sigma-Aldrich) in PBS of pH = 7.0 – Solution C: solution of type III mucins (Sigma-Aldrich) in PBS of pH = 7.0 |
| Disc (countersample) | – corundum ceramics (Institute of Ceramics and Building Materials in Warsaw); – <i>Meridian B2</i> composite filling (Ardent); – <i>GK</i> amalgam filling (AT&M Biomaterials Co., Ltd.); – Ti-6Al-4V titanium alloy; Ra = 0.4 μm (ATI Germany) |

Friction tests were conducted in the environment of natural saliva and three model saliva compositions (Table 2). The first composition contained three types of pyrophosphates as well as xanthan gum, while the second and the third compositions contained natural animal mucin of type II and type III, respectively. All the solutions had been prepared on the basis of the PBS (phosphate buffered saline) solution. Due to the structure of the friction mechanism, an identical volume of the lubricating substance was used for each friction pair, in the amount of 5 ml. Table 2 presents information on the materials and lubricants used in the tribological tests.

To prepare enamel samples, healthy teeth, removed on the basis of an orthodontist's opinion, were used. The criterion for selection of enamel samples was their similar microhardness value ($HV_{0.1}$). Samples were stored in Hanks solution at a temperature of approx. 5 °C. Cylindrical enamel samples were cut out from the chewing surfaces of teeth (the cylinder face included the surface of the tooth cusp) and glued to an aluminum socket of 6 mm in diameter using bone cement. The samples thus prepared were subjected to further machining that consisted in facing, polishing of the flank for the purpose of obtaining a truncated cone with a slant height angle equal to 20° and a diameter of 1 mm, and final super-finishing of the cone top with 1000 and 2000 grain sandpaper.

Based on the initial research performed and literature review, the following parameters were adopted for the tribological tests: friction velocity, $v = 0.1$ m/s; friction radius $r = 8$ mm; countersample (disc) rotational velocity $n = 120$ rpm; diameter of the truncated area of the sample $\phi = 1$ mm; contact pressure applied on the sample $p = 20$ MPa; time of friction in a single test $t = 3$ h (10800 s); lubricant volume $V = 5$ cm³.

The results of the tribological tests were statistically processed using the STATISTICA software package, StatSoft company. All the observed characteristics are the mean values obtained from three tests performed under the same measurement conditions at ambient temperature. The linear wear of the friction node was continuously recorded with the use of a displacement transducer (T-11 tester). The surface topography of the samples and their chemical composition were examined using a Hitachi S-3000N scanning microscope (equipped with an EDS attachment for X-ray microanalysis).

3. Test results and discussion

During the tribological tests, the coefficient of friction and the linear wear of cooperating pairs in the solutions of human saliva and its three substitutes were examined. Figures 1–8 show the results of tribological tests, and Figures 9–11 show the enamel surface topography after friction tests in various environments.

Figures 1 and 2 illustrate the tribological test results obtained for the enamel–GK dental amalgam friction pair in the environments of the artificial saliva solutions under research. For all the lubricants applied, varied friction coefficient values were recorded during the friction tests (Fig. 1), although these values were similar in the analysed preparations. The lowest value of the coefficient of friction was obtained for friction pair in solution of human saliva. From the linear wear diagram (Fig. 2), it can be observed that the best lubricating substitute was tailor-made solution A on the basis of pyrophosphates, while the highest wear of the applied enamel–GK dental amalgam friction

pair was recorded for PBS with addition of mucin II solution.

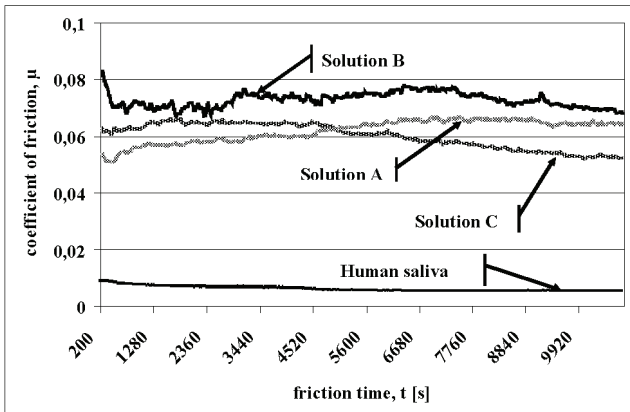


Fig. 1. The coefficient of friction as a function of friction time for the enamel-GK dental amalgam friction pair in the environment of human saliva and its three substitutes, respectively

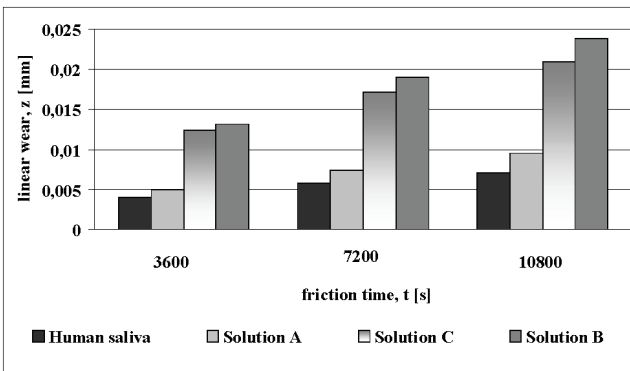


Fig. 2. Linear wear as a function of friction time for the enamel-GK dental amalgam friction pair in the environment of human saliva and its three substitutes, respectively

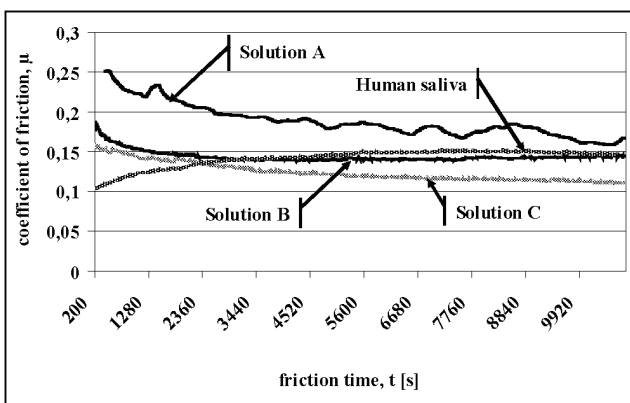


Fig. 3. The coefficient of friction as a function of friction time for the enamel-ceramics friction pair in the environment of human saliva and its three substitutes, respectively

For the enamel-corundum ceramics friction pair, enamel in a pyrophosphate solution with xanthan gum exhibited the highest values of linear wear (Fig. 4) and

of the friction coefficient (Fig. 3) in comparison with the other artificial saliva substitutes. The best tribological properties were observed for the substitute containing type III mucin in a PBS solution (the lowest values of the coefficient of friction and linear wear of the enamel).

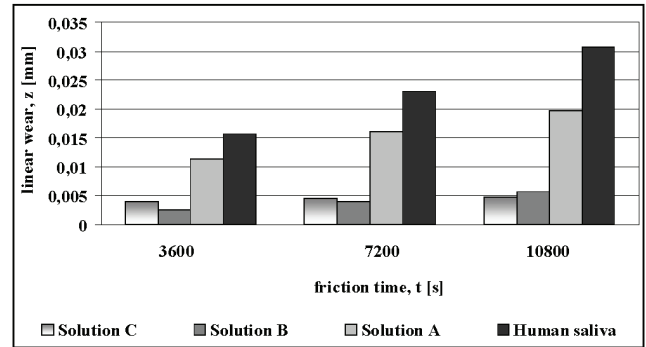


Fig. 4. Linear wear as a function of friction time for the enamel-ceramics friction pair in the environment of human saliva and its three substitutes, respectively

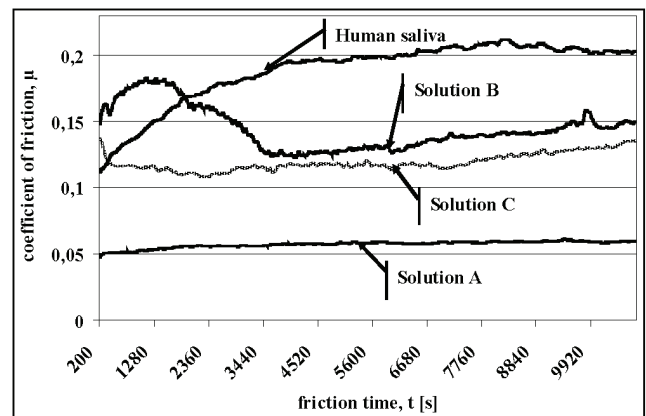


Fig. 5. The coefficient of friction as a function of friction time for the enamel-Meridian B2 dental composite friction pair in the environment of human saliva and its three substitutes, respectively

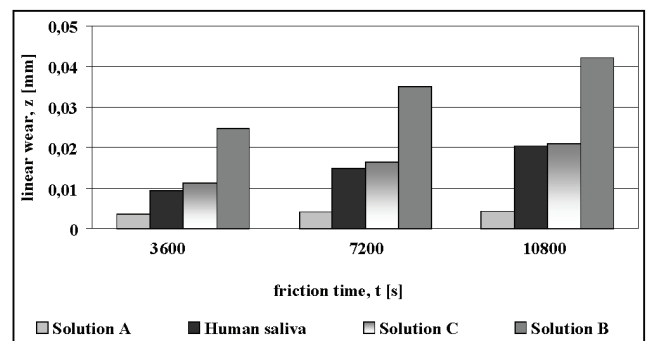


Fig. 6. Linear wear as a function of friction time for the enamel-Meridian B2 dental composite friction pair in the environment of human saliva and its three substitutes, respectively

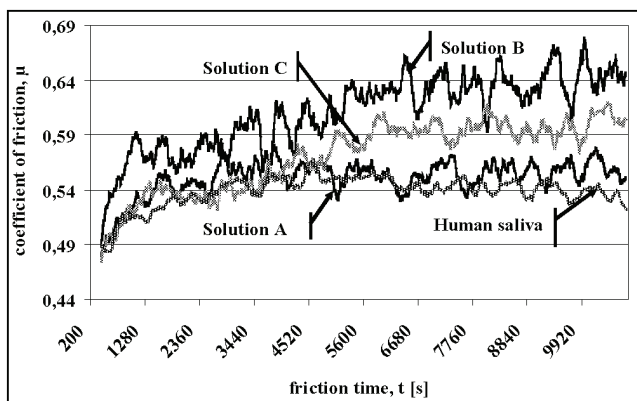


Fig. 7. The coefficient of friction as a function of friction time for the enamel–Ti-6Al-4V titanium alloy friction pair in the environment of human saliva and its three substitutes, respectively

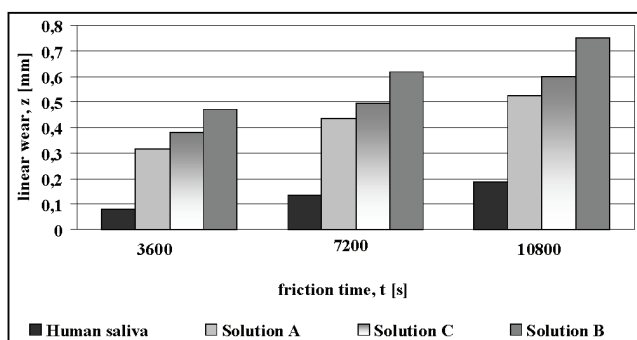


Fig. 8. Linear wear as a function of friction time for the enamel–Ti-6Al-4V titanium alloy in the environment of human saliva and its three substitutes, respectively

In the case of the enamel–Ti-6Al-4V titanium alloy friction pair, very high values of the coefficient of friction and enamel wear were observed (Figs. 7, 8). Throughout the entire friction process, sudden jumps of parameter μ were observed for all saliva substitutes.

For this friction pair, the pyrophosphate substitute with xanthan gum was characterized by the best lubricating properties, and type II mucin solution exhibited the worst lubricating properties.

After the friction processes were finished, the surface topography of enamel samples was evaluated with the use of a scanning microscope. Figures 9 through 11 present some selected microphotographs of the sample surfaces after the three-hour friction process in the environment of natural saliva and its artificial substitutes, respectively.

The microscopic analysis of the sample after the friction process in the environment of the solution of type III mucins in the PBS solution (photographs not shown here) revealed destruction of the surface, with elements of abrasive wear. The topography of the enamel surface (250× magnification) shows a series of parallel scratches. At a magnification of 1000×, some cavities and cracks, emphasizing the brittle nature of enamel, can be observed.

Characteristic cracks and scratches that may be the result of abrasive wear can be seen on the registered image of the topography of the enamel surface in the environment of type II mucins (Fig. 9). Gaps were filled by products of the abrasive material from the amalgam (light lines in Fig. 9).

The topography of the enamel surface (250× magnification) after tribological tests (photographs not shown here) in the environment of human saliva shows a series of parallel scratches. At 1000× magnification, some cracks can also be observed.

In the case of the enamel–ceramics pair and the enamel–Meridian B2 dental composite pair tested in the environment of the pyrophosphate and xanthan gum solution, a very smooth enamel surface with numerous cracks was observed (photographs not shown here).

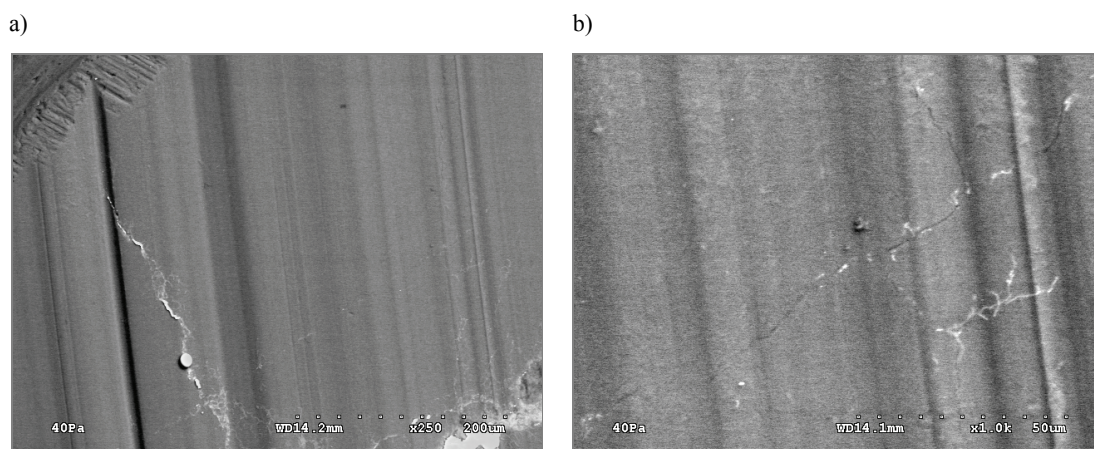


Fig. 9. Microphotograph of enamel after tribological tests in type II mucin solution for the enamel–GK dental amalgam friction pair; magnification: (a) 250×, (b) 1000×

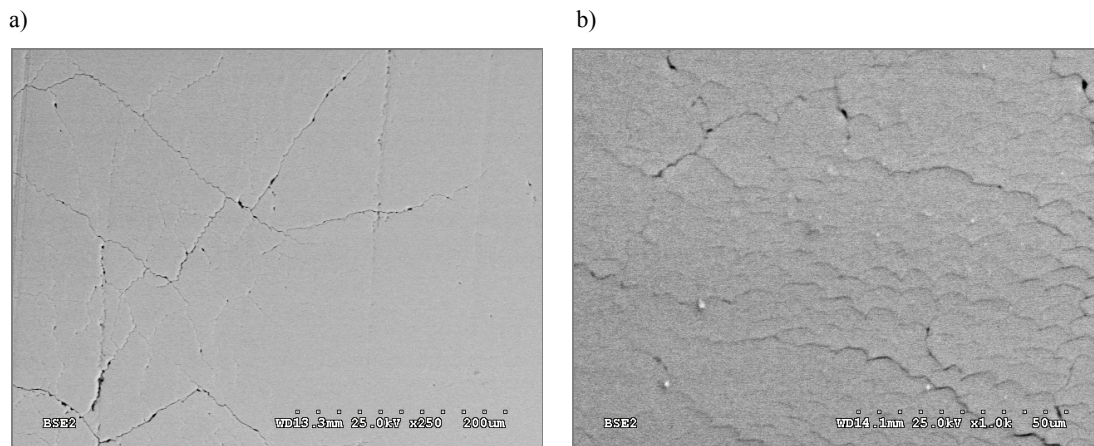


Fig. 10. Microphotograph of enamel after tribological tests in the pyrophosphate with xanthan gum solution for the enamel–ceramics friction pair; magnification: (a) 250 \times , (b) 1000 \times

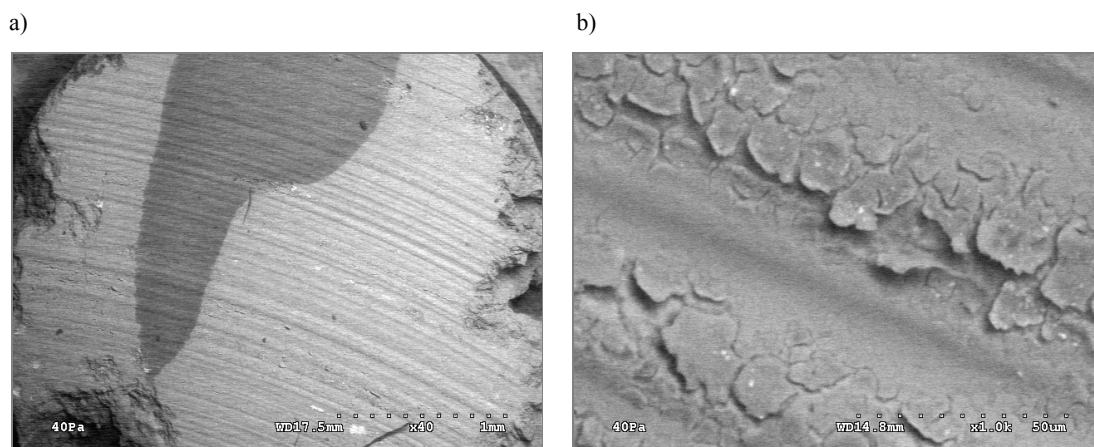


Fig. 11. Microphotograph of enamel after tribological tests in the type III mucin solution for the enamel–Ti-6Al-4V titanium alloy friction pair; magnification: a) 40 \times , (b) 1000 \times

Microscopic observations of enamel after tribological tests in the environment of human saliva and of type III and type II mucins did not reveal significant traces of destruction on the enamel surface (photographs not shown here). A smoothed structure was observed with cracks in certain areas resulting from the brittle nature of human tooth enamel.

Areas of deeper loss of material, characteristic of adhesive wear, were visible on the surface (at the magnification of 1000 \times). Numerous scratches and small surface cracks were observed on the surface of the enamel after the friction process in the environment of the pyrophosphate and xanthan gum solution (photographs not shown here). Microscopic observation indicates a brittle nature of human tooth enamel and elements of abrasive wear.

In the images taken at 1000 \times magnification after the friction tests on the enamel–Ti-6Al-4V alloy in the environment of mucin type III were finished (Fig. 11), numerous wear products formed as a result of de-

struction of the counterpart surface can be seen on the sample surface, and fragments of thin films of secondary structures were revealed. Secondary structures separating from the surface, being the result of fatigue processes and small particles of wear products were also observed, which may indicate secondary wear processes of the elements in friction with each other. Dark enamel discoloration can be seen in Fig. 11a, indicating “close” contact with the dentine – the layer under the enamel surface.

4. Discussion

Salivary-protein biofilm is formed on all surfaces which have contact with oral cavity, for example, dental filling materials, prosthodontic elements. The composition of natural biofilm is different and contains, besides microorganisms, also inorganic and organic

substances, mainly a wide variety of proteins and glycoproteins [2], [3], [6]. One of the main roles of this kind of film is lubrication of natural tooth–tooth or tooth–dental material friction pairs' body [1]. This phenomena is evaluated by different methods. One of them is friction tests, where friction pair consists of enamel–dental material [12]–[22]. Dental materials could be used in the form of porcelain [12], [23], gold alloy, nickel-chromium alloy [14], [15], titanium alloy [20], [21], or steel [22]. Different friction parameters (loads and rotational cycles) are used during tribological tests [16], [21], [24]. In work [16], the friction and wear behaviour of human teeth under different wear conditions (loads – 10, 20, 40 N and 2000 cycles) were examined with the aim to understand the tooth wear process. The authors of this work point out that friction and wear behaviour of human teeth depend strongly upon wear condition. Generally, in literature the load between 3 and 30 N is used. In our work, we used 15.7 N.

Various solutions are used in tribological tests. In work [25], deionized water, mucin-based artificial saliva and carboxymethylcellulose-based saliva were examined. In other works [12], [24], lubricants in the form of hydrochloric acid solutions (pH = 1.2, 3.0), human saliva were used. Also, functional contacts in an artificial oral environment with and without an abrasion material (Al_2O_3) were tested [14].

In this work, the tribological characteristics of a kinematic pair (pin on disc) made from enamel–dental material (composite filling material, titanium alloy, dental amalgam, corundum ceramics) was evaluated in the environment of human saliva and in the environment of its substitutes. A novelty of this work, comparing to other investigations [11], lies in the tribological tests of different kinematics pairs in solutions of a pyrophosphate mixture with addition of xanthan gum and two mucins as a proposition for ingredients of artificial saliva solution. The results obtained in this work confirm literature reports [25] that saliva solutions prepared using a mucin III base, exhibit similar tribological characteristics to human saliva for all the saliva solutions tested.

One of the most important factors in tribological investigations, besides friction coefficient, is wear. This term in dentistry [16] is used to describe the phenomena of attrition (describes tooth surface loss by tooth-to-tooth or tooth-to-dental material contact, and is often regarded as two-body wear) and abrasion (three-body wear caused by movement of food over both antagonizing teeth surfaces during mastication). The wear of tooth sample after tribological processes is evaluated by, e.g., volume method [16], [24], ste-

reomicroscope method [12]. In our work, the wear resistance was analysed by measuring the linear wear of testing friction pair. The wear rate of testing materials as a function of time increased as the friction time increased. Like in literature [25], its values were most similar for friction in a contact environment based on natural saliva and type III mucin. The amount of wear of the kinematic pair was the lowest when opposed by the porcelain in PBS solution with addition of mucin III. The findings of our work show that the wear of nonceramic prosthetic materials (titanium alloy) in contact with enamel was the greatest.

The worn enamel surfaces were observed by scanning electron microscope to determine the wear characteristics. Micrographic assessment showed cracks and extensive surface destruction of enamel due to abrasion, like in works [15], [20]. Furrows and granular debris were observed on the worn surfaces of enamel while sliding, especially against the titanium alloy, indicating an abrasive wear mechanism, as in research presented in [15]. The particles obtained from the worn enamel are mainly the products of microscratching induced in the process of wear.

5. Conclusions

The tribological tests and analysis of available literature data made it possible to develop our own saliva substitutes with emphasis on their tribological properties. Compositions with similar properties to human saliva in terms of tribological characteristics were obtained.

Tests of tribological properties of the developed compositions of saliva substitutes revealed differences between the tested pairs. On the basis of the results of the present research, the following general conclusions can be made:

1. The saliva substitute containing pyrophosphates and xanthan gum exhibited the best lubricating properties for the majority of friction pairs.
2. The highest values of wear and of the coefficient of friction were observed for the enamel–Ti-6Al-4V titanium alloy friction pair, and the lowest values were obtained for the case of the enamel–Meridian B2 dental composite friction pair.
3. The results of the microscopic studies revealed varied forms of tribological wear for the cooperating materials. The formation of secondary top layers containing wear products and lubricant components seems to be significant. Considering the nature of the operation of the friction pair, it

might be concluded that these layers are formed as a result of interaction between the components of the different lubricants and the surfaces of the co-operating materials.

The preparations developed can find applications in treatment of many diseases (e.g., xerostomia) and in dental prosthetics. In the latter group of applications, they may serve to decrease the wear of metallic elements and thus increase their life.

The test results obtained in the present research confirm and justify the need to search for new substitutes of human saliva for the purposes of dental surgery.

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