

Dental Tribology at the Microscale

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Abstract

Dental wear caused by tooth care is a complex phenomenon that depends on the quality of the tooth material, the type of tooth brush and the brushing slurry. Tooth wear is commonly determined in abrasion experiments using a standardized tooth brush in contact with a radioactively labeled dentin sample (RDA method). The increase of radioactivity in the slurry is a direct and highly-sensitive indicator for wear. It is, however, detrimental, that RDA provides an integral view of the tribological processes leaving microscopic issues undetected. Therefore, in this contribution the macroscopic system of brush versus tooth was reduced to a microtribological setup analyzing the contact between a single bristle (monofilament) and a tooth sample. This setup allowed to correlate friction and wear events to topography and structure of the tooth and will enable the evaluation of cleaning processes microscopically in the future. In addition, results of this work were related to the literature results of RDA experiments.

Keywords

dental analysis, friction, wear, microtribology

1 Introduction

Daily dental care is commonly pursued by brushing teeth using a tooth brush, some water and tooth paste. The tooth paste is mainly composed of cleansing particles, foamer, moisturizer, binding agent, sweetener, flavoring agent, preservative and fluoride containing ingredients. The cleansing particles serve as mild abrasives to remove stain and plaque from the tooth surface. As a consequence, besides attrition (wear due to direct tooth-to-tooth contact) brushing teeth using tooth paste is the most important wear process and the wear rate determines the lifetime of the tooth, especially the dentine [1-2]. Several studies simulated attrition [3-8] and brushing teeth using toothbrushes [9-13]. In these studies different materials were used, e.g., human enamel [3-6] or teeth [9,10], different restoration materials [4,6-8,11], bovine incisors [12] or PMMA [13]. The majority of tribological tests were carried out in natural or artificial saliva [3-8] using a mechanical setup with reciprocating motion [3-8,11,13]. Friction turned out to be lower in fluid media compared to dry environments [13]. Generally, the friction coefficient does not depend on fluid viscosity [5] and in this particular experiment [8] not on sliding velocity. The addition of small amounts of

particles to the fluid rapidly increases friction [4,8,13], while any further addition hardly impacts friction [13]. The action of particles was examined by the addition of food particles [3], Al_2O_3 particles [4,5], calcium pyrophosphate [10,11], calcium carbonate [11], calcite [13] or commercial products for dental care [9,10]. Particle shape was shown to have a great impact on abrasivity [14]. Lewis et al. [15] analyzed the trapping of particles in the contact between filament and tooth as function of load, deflection of the filaments, particle size and particle hardness. Only some particles are trapped stochastically in the contact. After a few thousand brushing cycles different patterns of scratches caused by the filaments appeared [3,13].

In regard to wear on tooth enamel, literature reports on different influencing factors. Wear increases with increasing load [3,10,11,13], but decreases when the load reaches the point when the filaments spread [11]. Wear often correlates with friction energy [6] and increases with the number of brushing cycles [10]. Wear in fluid media is smaller than wear caused by dry friction. However, the addition of abrasives to the fluid causes strong wear [3-5,7,9]. Furthermore, abrasion is dependent on the type of abrasive and the composition of the toothpaste [10]. The American Dental Association proposes to use RDA (relative dentin abrasion) values to verify the abrasiveness of toothpastes [16-18]. By means of thermal neutrons, tracer atoms are generated in the enamel. Due to radiation, ^{31}P is transferred to ^{32}P . The dentin sample is then subjected to brushing in a standardized brushing apparatus. In the course of brushing, wear particles appear in the slurry. With the help of a highly-sensitive detector, the radioactive traces in the slurry are monitored. This technique is very similar to radionuclide technique, as used in mechanical engineering [19]. Abrasive wear and RDA value increase with decreasing filament diameter of the toothbrush [12].

Unlike previous studies, which used entire toothbrushes for the tests, this approach reduced the system to the microtribological contact between a monofilament of a toothbrush and a sample of human enamel. The intention was to identify friction and wear mechanisms of enamel in various media by reduction of external influences. The experimental set-up allows us to correlate friction and wear effects with microscopic topography changes of the tooth surface.

2 Experimental Procedure

2.1 Microtribometry

The head of a tooth brush holds usually more than 1,500 single bristles, the so-called monofilaments. To determine the adequate normal force for the Pin-On-Disk (POD) microtribometer experiment using a Tetra BASALT-PT[®], the averaged force acting on a toothbrush was divided by the number of monofilaments. It turned out that a normal force range between 1 and 5 mN generates a contact pressure similar to that for a single filament in an entire brush [20]. Thus, the complex macroscopic system was successfully replaced by a microtribological setup consisting of a single filament and a flat piece of human enamel with an interferometer-determined average roughness of 20 nm measured over an area of 1 mm × 1 mm. For this work a monofilament of a cylindrical toothbrush (elmex inter X, GABA International AG, Therwil) with a diameter of approximately 0.2 mm ± 0.02 mm and a length of 5 mm was used. The

filament was shortened to improve the signal to noise ratio by reducing lateral fluctuations. The tooth sample was placed inside a container filled with water or tooth paste slurry. The container was situated at the center of a disk connected to a drive to rotate the disk and specimen at constant velocity (0.75 cm/s). Loads and brushing speeds used in the tests were based on reported measurements. Typical loads ranged from 2.4 mN to 8.8 mN and brushing speeds from 30 mm/s to 150 mm/s [21,22]. The pin, i.e. the monofilament, was attached to a double leaf spring, which was placed outside the water above the container. The separation between filament and tooth sample was controlled by a vertical drive. Upon down-motion of the spring the contact was established. Continued approach sets the normal force by deflecting the leaf spring in normal direction. The spring was fabricated from steel with a spring constant in tangential direction of 195 N/m and in normal direction of 162 N/m. The experimental setup is shown in Fig. 1.

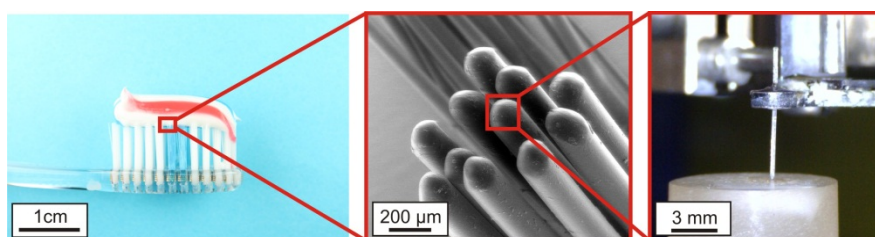


Fig. 1: Tooth brush, set of filaments and tribological pairing. The right photo shows the monofilament attached to the spring in contact with the tooth sample.

The microtribometer was used to gather data about the long-term behavior of the system. For the dry contact, distilled water coverage and toothpaste slurries with different RDA values (toothpaste provided by GABA International AG) 8,000 revolutions were recorded. The friction values were averaged over 200 revolutions to give one data point in the diagram.

In order to measure time-resolved friction coefficients along a single linear path across the enamel, a nanoindenter (Nano Indenter G200, Agilent Technologies) was used at low sliding velocity (25 $\mu\text{m/s}$). This kind of approach allowed us to analyze the interaction at the tip of the filament. The device is sensitive enough to detect subtle effects. The water film thickness was 2 mm in both types of experiment. Thus the amount of fluid was sufficient to fully lubricate the system. However, the system was operated in boundary lubrication this means that the tip of the filament was always in contact with the tooth and/or trapped particles. Since this is a stochastic process, it cannot be ruled out that situations exist where no particles are entrained between tip and tooth.

2.2 Samples

All experiments were carried out with human teeth, obtained from human third molars. First the teeth were mechanically cleaned to remove adherent tissue. Then the samples were disinfected with hydrogen peroxide at 5 wt. % and cleaned in an ultrasonic bath. The teeth were stored at 4°C in double-distilled water with additions of ethanol and thymol. With a minitome (Struers) the roots of the teeth were cut off. One tooth crown was separated into 8 pieces which were separately embedded in acrylic resin. These samples were ground with wet abrasive paper with decreasing grit size (P500, P1,200, P2,500, P4,000) and samples were kept in a cool place in distilled water until the beginning of the measurements. The topography of the

samples was evaluated before and after the tests with a confocal laser scanning microscope (CLSM, Leica TCS SL from Leica Microsystems CMS GmbH, Germany) and a scanning electron microscope (SEM, Quanta 3D FEG from FEI, Netherlands). The depth of scratches caused by the monofilament provided information about wear. The experiments were carried out in different fluids, i.e., distilled water and three toothpaste slurries and compared to dry sliding. The different toothpastes (toothpaste 1: RDA 30, toothpaste 2: RDA 75 and toothpaste 3: RDA 165) were mixed with distilled water to obtain a slurry with a ratio of 1:2 w/w. Five samples per treatment were tested.

2.3 Characterization of abrasives

In order to extract the abrasive from the toothpaste formulation, ethanol was used to remove binding agents which are ingredients of toothpastes 1-3. About 5 g of the toothpaste were applied to a test tube with 50 ml of ethanol and mixed. The separation was achieved by sedimentation and centrifugation of the suspension. Before taking out the solid material from the test tube, this procedure was repeated three times. Afterwards, the remaining solid components were air dried. The abrasives were investigated by SEM (same instrument as mentioned above).

3 Results

3.1 Friction and wear measurements

With the help of the POD tribometer friction was measured as function of the number of disk revolutions. Figure 2a) shows the coefficient of friction between cylindrical monofilament and enamel sample in different environments; dry, water-lubrication, and the 3 tooth paste slurries. For dry and water-lubricated sliding conditions, similar friction coefficients were obtained ($\mu_{\text{dry}} = 0.08 \pm 0.02$, $\mu_{\text{water}} = 0.6 \pm 0.02$), whereas all slurries showed significantly higher values ($\mu_{1+2} = 0.15 \pm 0.05$, $\mu_3 = 0.17 \pm 0.05$). At the end of the experiments the slurry with the highest RDA value was accompanied by highest friction. At about 3,000 revolutions the curves of slurry 1, 2 and 3 intersect, showing similar friction values.

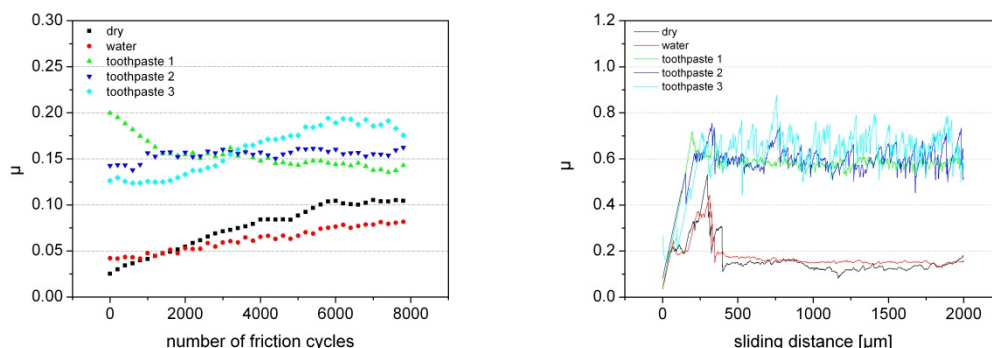


Fig. 2: Friction as function of the number of revolutions for different environments.
a) POD microtribometer, b) nanoindenter.

Using the nanoindenter a single path across the dental surface was monitored with high resolution, see Fig. 2b). The nanoindenter allowed a sliding distance of 2 mm,

over which a significant number of stick-slip events was received for all slurries. In agreement with the POD tests dry and water-lubricated sliding showed the lowest friction coefficients, however at higher level ($0.05 \rightarrow 0.15$). The friction coefficients of the experiments with slurry exhibited even higher values ($0.6 \dots 0.7$). It should be noted that the sliding velocity in the nanoindenter experiment is about 3 orders of magnitude lower than for the POD setup, i.e., $25 \mu\text{m/s}$ versus 0.75 cm/s , causing higher coefficients of friction due to pronounced boundary lubrication. Friction increased with increasing RDA value. In addition to higher friction coefficients the data scatter of dynamic friction increased as well. The higher the RDA value was, the larger was the scatter.

Figure 3 shows the circular wear track on the enamel surface for each slurry experiment. The depth of the track increases with increasing RDA value. Details of the wear track were analyzed with a scanning electron microscope, see insets of Fig. 3, lower right corner. To determine the wear depth the laser scanning microscope was applied. The tooth paste slurry 1 with the lowest RDA value introduced only subtle changes to the topography (left image). In sharp contrast, slurry 3 resulted in clear features of abrasion (right image). For dry sliding, as well as sliding with water, lubrication wear was not detectable. When the wear depth is plotted as function of the RDA values, a linear dependence was observed, see Fig. 3.

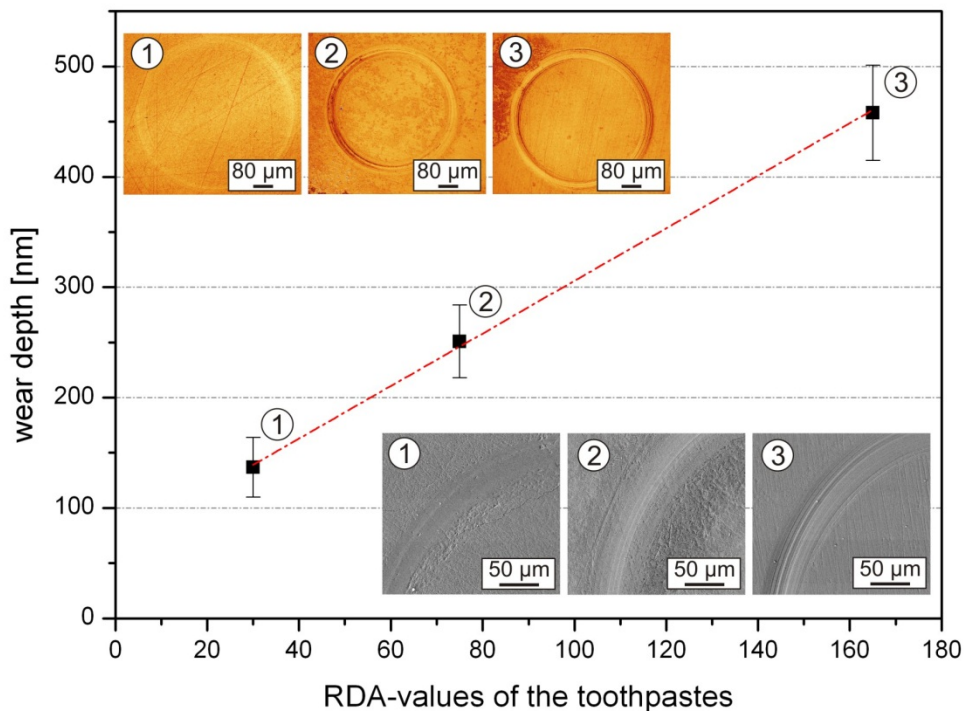


Fig. 3: Wear depth as function of RDA values. Insets: Light microscopy images of circular wear tracks (top row) and magnifications by scanning electron microscopy (bottom row).

3.2 Analysis of slurry

In order to obtain further information about the wear mechanism caused by the toothpaste slurry, scanning electron microscopy images were taken at different magnifications, see Fig. 4. All tested toothpastes solely (toothpaste slurry 2) or rather mainly (toothpaste 1, 3) consisted of irregular shaped silica particles. The size of these silica particles varied between 1 and 30 μm . Besides the mentioned silica particles, toothpaste 1 contained spherical polyethylene agglomerates. In contrast to toothpaste 1, toothpaste 3 additionally contained alumina abrasives, differing considerably in geometry from the silica and polyethylene particles. Alumina abrasives are sharp-edged, flaky particles with maximal widths of 25 μm .

All mentioned types of particles clearly differ with respect to form and size distribution as well as hardness. By means of the great diversity of abrasives used in toothpastes, the hardness of these particles is not clearly indicated in the literature. A differentiation concerning the hardness may be determined only on the basis on the Moh's scale. Literature values obtained for silica are between 2.5 and 5, whereas for alumina around 9 [23]. Therefore, the hardness of alumina abrasives is much higher than the hardness of silica particles.

The particles were dispersed in an aqueous slurry that is adapted to the bio-chemical environment of the oral cavity. Therefore, the free surface energies of the slurries ($\gamma = 25 \dots 35 \text{ mN/m}$) are different from that of water. The energies were measured with a Krüss tensiometer using a Wilhelmy plate according to $\gamma = F/(2l \cos \theta)$, where F is the force to pull the plate, l is the wetted length of the plate and θ is the contact angle between the liquid phase and the plate.

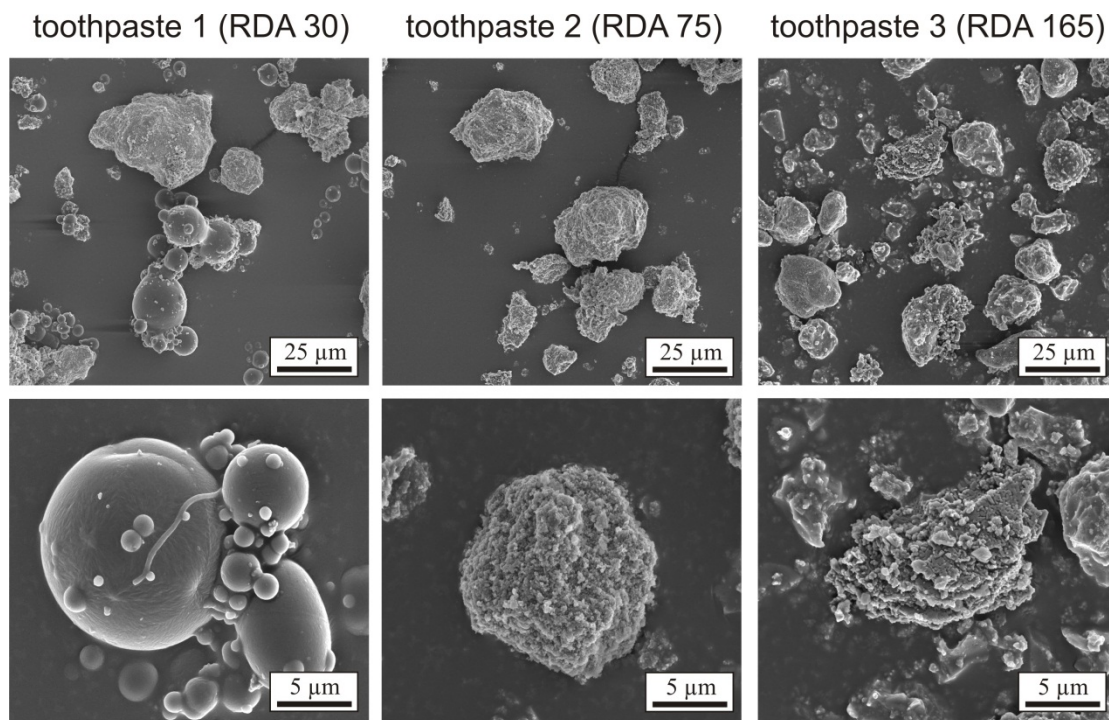


Fig. 4: SEM images of particles in the toothpaste slurries.

4 Discussion

Due to reduction in scale of conventional macroscopic tribological systems, comprising of toothbrush and enamel to the microscopic contact of monofilament and human enamel, elemental mechanisms of dental friction and wear were probed in detail.

The friction coefficients of dry sliding and sliding in distilled water were significantly lower than the coefficients measured for all toothpaste slurries. Capillary forces which are directly proportional to the free energy of the slurry can not be made responsible for this increase, since the free surface energy of the slurry was lower than the energy of water, see above. Moreover, according to the simple approach $F_c = 4\pi\gamma R \approx 90 \mu\text{N}$ (R is the radius of the monofilament tip), capillary force was more than thirty times lower than the applied normal force of 3 mN. Thus, capillary force played a minor role, which is also reflected in the small differences between dry sliding and water-lubricated sliding.

It should be noted that fluid flow effects may obstruct the motion of the filament through the slurry. However, since the force acting on the filament was directly proportional to the density of the fluid, the slurry must have double the density of distilled water in order to increase the friction coefficient by a factor of 2.

Another possible mechanism for the increase of friction is the trapping of particles between the tip of the monofilament and the tooth. This effect was already discussed by Lewis et al. [14] and is similar to sanding icy ground. The interaction of the filament tip and the particles in the interface causes interlocking, thereby accumulating energy that is released in stochastic stick-slips. Their intensity was directly proportional to the RDA value of the tooth paste.

The time-resolved measurements using the nanoindenter showed significant stick-slips for the tooth paste slurries. In general, the amplitude of the stick-slips increased with increasing RDA value. The largest scatter was obtained for the tooth paste with the highest RDA value. Figure 3 provides the answer for this behavior. After initiation of the test, the surface of the tooth undergoes topographical changes. Especially tooth paste 2 and 3 caused wear accompanied by a roughening of the surface. The rougher a surface is, the more pronounced are the stick-slip events [24]. This effect becomes underlined when, in addition to the tooth surface, the filament tip shows increased roughness as well. Another cause for increased stick-slip behavior could be a mechanically induced change of the shape of the particles. However, this effect was not investigated here.

The wear analysis showed that abrasion effects cannot be obtained by dry sliding or brushing with water alone. Abrasion only emerged when slurry was used and were always accompanied by the removal of dental material. Thus, step by step, the thickness of enamel was reduced. In general, abrasion depth obtained with the monofilament experiments were directly proportional to the abrasion data received from RDA.

The width of the wear track of the slurries corresponded well with the Hertzian contact radius calculated for a spherical tip of the filament for the given normal force and a Youngs modulus of about 2 GPa for Nylon. With increasing RDA value the width of the wear track increased. This increase was caused by abrasive particles sticking at the tip of the filament. The harder these particles are, the clearer are the wear marks outside the Hertzian contact.

With respect to the obtained wear rate, the received data seem to be very realistic. From the abrasion depth shown in Fig. 3, one receives wear rates of 128 nm/h for RDA=30, 228 nm/h for RDA=75 and 416 nm/h for RDA=165 (8,000 revolutions at 0.75 cm/s). Assuming a brushing time of 1 minute per day, 365 days a year times 50 years leads to total time of about 300 hours. The obtained wear rates multiplied by the total brushing time result in an extrapolated total abrasion depth between 38 μm and about 125 μm . These values appear definitely realistic. Due to other influencing factors, not investigated here, for example acid-related erosion and brushing force, the local *in vivo* wear can be higher.

5 Conclusions

This study at hand showed that research about the complex process of tooth abrasion can successfully be reduced to the analysis of a monofilament in contact with a tooth sample. The findings recommend microtribometry as a simple and robust tool for tooth paste development and characterization. Moreover, microtribology showed that the effect of trapped particles introduces frictional power causing wear. Frictional power is released as a sequence of slip events. Further studies have to show how microtribometry can help to characterize the potential to remove stain and/or plaque. Due to simplicity of the approach, it is feasible to operate many microtribometers or miniaturized tribometer setups simultaneously to increase throughput and improve statistics in dental research.

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