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Wettability modification of human tooth surface by water and UV and electron-beam radiation

Gaby E. Tiznado-Orozco [a,b], José Reyes-Gasga [a,c,*], Florina Elefterie [a], Christophe Beyens [a], Ulrich Maschke [a], Etienne F. Brès [a]

[a] UMET, Bâtiment C6, Université de Lille 1, Sciences et Technologies, 59650 Villeneuve d'Ascq, France
[b] Unidad Académica de Odontología, Universidad Autónoma de Nayarit, Edificio E7, Ciudad de la Cultura "Amado Nervo", C.P. 63190 Tepic, Nayarit, Mexico
[c] Instituto de Física, UNAM, Circuito de la Investigación s/n, Ciudad Universitaria, 04510 Coyoacan, México, D.F., Mexico

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ABSTRACT

The wettability of the human tooth enamel and dentin was analyzed by measuring the contact angles of a drop of distilled water deposited on the surface. The samples were cut along the transverse and longitudinal directions, and their surfaces were subjected to metallographic mirror-finish polishing. Some samples were also acid etched until their microstructure became exposed. Wettability measurements of the samples were done in dry and wet conditions and after ultraviolet (UV) and electron beam (EB) irradiations. The results indicate that water by itself was able to increase the hydrophobicity of these materials. The UV irradiation momentarily reduced the contact angle values, but they recovered after a short time. EB irradiation raised the contact angle and maintained it for a long time. Both enamel and dentin surfaces showed a wide range of contact angles, from approximately 10° (hydrophilic) to 90° (hydrophobic), although the contact angle showed more variability on enamel than on dentin surfaces. Whether the sample's surface had been polished or etched did not influence the contact angle value in wet conditions.

1. Introduction

Surface wettability indicates the ability of a liquid to wet the surface of a solid [1]. In biomaterials, surface wettability is an important property because it may indicate cells immobilization, drug delivery and gene transfer, among other applications [2,3]. For human tooth and skin, wettability is of interest in the study of pharmaceutical and cosmetic products [4]. In tooth, hydrophobicity affects initial water absorption and the adhesion of oral bacteria [5]. The success of any dental restorative treatment depends on the adhesion to the dental tissue; but the improvement of adhesion implies the need for acid etching of enamel surfaces [3]. On the other hand, the analysis of adhesive systems in dentistry could identify a way to eliminate, for example, the metal brackets [6].

The wettability of a material is usually determined by measuring the contact angle formed between the surface of the material and the line tangent to the curved surface of a liquid drop at the point of contact [7]. The contact angle depends on the surface energy of the material and the surface tension of the liquid. In addition, the surface energy is correlated with different physical parameters, such as elastic modulus, melting point, and heat and energy of vaporization [1,4,8]. The contact angle is also related to the surface roughness of the substrate, which can be modified during the sample preparation process [5]. A brief theoretical analysis of the relationship between the contact angle and the surface energy is given by Aronov et al. [9].

Contact angle measurement is probably the most popular method to determine the hydrophobicity and/or hydrophilicity of the surfaces of materials. A greater contact angle indicates hydrophobicity (poor wetting). Several authors [6,10] consider that a contact angle of less than 90° is an indication of a hydrophilic material, whereas a contact angle larger than 90° indicates a hydrophobic material. However, several others [11] consider that an angle of 65° is more appropriate for this differentiation.

Wettability modification of hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(OH)_2$) by electron irradiation has been reported by Aronov et al. [9,12]. These researchers showed that the use of low-energy electron irradiation modifies the wettability of the HAP surface in a wide range of contact angles, extending from 10° (hydrophilic) to 100° (hydrophobic). They indicated that the incident electrons generate electron/hole pairs, resulting in significant variation of the surface potential of the hydroxyapatite (the penetration depth is approximately 2 nm for 100 eV incident electrons) and giving rise to the wettability modification observed.

Based on this information and our experience in HAP and human tooth material, we undertook the task of studying the contact angles

* Corresponding author at: Instituto de Física, UNAM, Circuito de la Investigación s/n, Ciudad Universitaria, 04510 Coyoacan, México, D.F., Mexico.
E-mail addresses: gab0409@gmail.com (G.E. Tiznado-Orozco), jreyes@fisica.unam.mx (J. Reyes-Gasga), elefterie_florina@yahoo.com (F. Elefterie), christophe.beyens@ed.univ-lille1.fr (C. Beyens), Ulrich.Maschke@univ-lille1.fr (U. Maschke), etienne.bres@univ-lille1.fr (E.F. Brès).

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on the surface of the human tooth enamel and dentin before and after electron beam (EB) radiation. The samples were cut in such a way that their surfaces were transverse (T) and longitudinal (L) to the tooth surface. The surfaces of the samples were mirror-finish polished, and some of them were also etched with phosphoric acid to reveal their microstructure. The samples were placed under dry and wet conditions, and the EB radiation dose was set at three different values. Because ultraviolet (UV) radiation can modify the wettability of the surface of certain ceramics, and due to the importance of this radiation in dental work, we decided to include UV irradiation as a condition in this study.

Human tooth enamel and dentin are composed mostly of natural HAP crystals [13]. Enamel is comprised of 96% HAP and 4% organic material, while dentin is 74% HAP and 26% organic material. Water content varies from 1 to 6% by weight in enamel and from 15 to 30% in dentin [14]. In enamel, HAP crystals have a tablet shape with dimensions of approximately $30 \times 50 \times 100$ nm. These tablets are arranged in a textured manner and provide form to the enamel prisms. The organic material in enamel wraps the HAP crystals. In dentin, conversely, HAP crystals have a platelet-like shape, and they are smaller than those in enamel. These crystals are immersed in an organic matrix and their density is higher around the dentinal tubules than in the space between them.

2. Experimental procedure

Human tooth enamel and dentin samples were taken from permanent human premolar teeth from persons 18 to 25 years old. All of the extractions were done for orthodontic or periodontal reasons.

2.1. Preparation of samples

The health of the teeth was carefully and clinically examined before and after being split into halves using a low-speed diamond micrometer saw (IsoMet, Buehler). Enamel and dentin were mechanically separated from each other using a hand-guided dental drill and a light microscope (Carl Zeiss model Axiovert 25).

Enamel and dentin were cut in such a way that the surfaces of the samples were flat and parallel (named “transverse”, or “T”, samples hereafter) or perpendicular (named “longitudinal”, or “L”, samples hereafter) to the tooth surface. Next, the surface of the samples was mirror finish-polished with silicon carbide paper and alumina slurries in a grinder–polisher (MiniMet, Buehler). Fig. 1 shows the light microscopy images of one of the L samples (Fig. 1A) and one of the T samples (Fig. 1B).

Some of the samples were left as obtained after the mirror-finish polishing (named “polished”, or “P”, samples hereafter) and others were etched with phosphoric acid until their microstructure was revealed (named “etched”, or “E”, samples hereafter). Fig. 2 shows the SEM image of one of the mirror finish-polished surfaces but, because of the smear layer produced during polishing, no contrast of the human tooth structure is observed. However, when these surfaces are etched with phosphoric acid, the enamel prisms and the dentinal tubules are observed (Fig. 3). All the samples were ultra-sonicated at 30 °C for 10 min in an ultrasonic bath (S30 H Elmasonic). The samples were kept in a desiccator following this procedure.

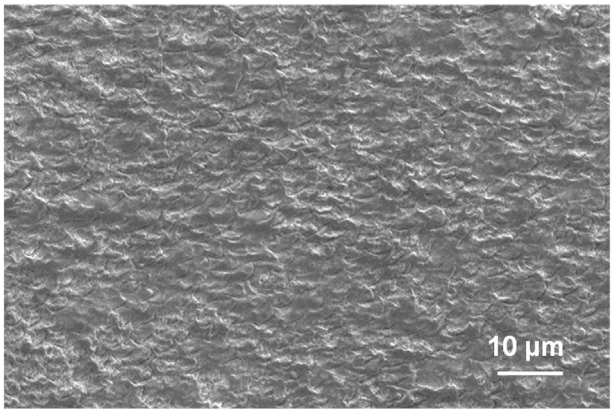

Fig. 2. SEM image of the surface of one of the samples just after polishing. The smear layer produced during polishing covers the microstructure of human tooth.

Therefore, we worked with 4 enamel samples (2 in transversal direction: one polished and one etched; and 2 in longitudinal direction: one polished and one etched), and 4 dentin samples (2 in transversal direction: one polished and one etched; and 2 in longitudinal direction: one polished and one etched). The dentin samples, as well as the enamel ones, were labeled TE, TP, LE and LP according to the cutting direction and to the surface treatment.

2.2. The contact angle measurement

For measuring the contact angle, a distilled water sessile drop measuring 7 mm in diameter was deposited on the surface of the sample by a syringe and the contact angle was measured using the Contact Angle Meter Digidrop (GBX Instrumentation Scientifique, France). For this equipment, the standard deviation for the contact angle measurements was $\pm 5°$. Optical inspection of the drop was performed by a camera device and digital imaging techniques. The measurement of the contact angle was taken 60 s after the deposition of the drop, the estimated time for equilibrium to be reached. For data collection, measurements were made in two drops at the same time and the average was used for the graphs. After each measurement, the water drop was wiped with

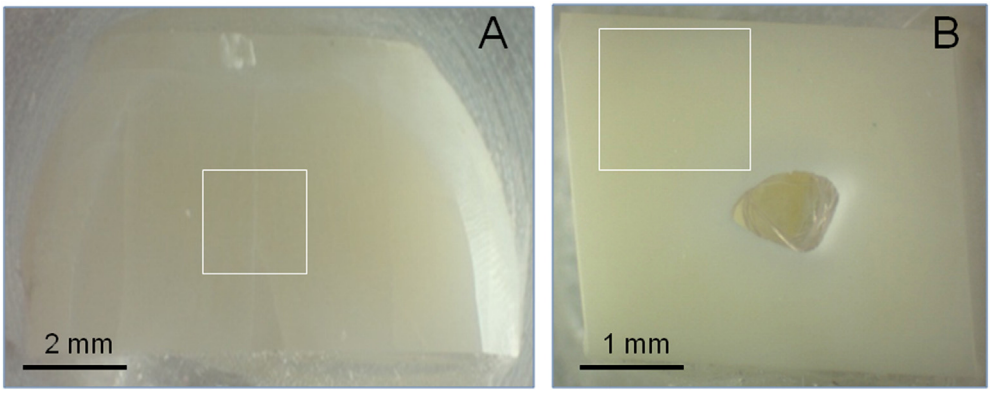

Fig. 1. Light microscopy images of two of the human tooth samples. A) Enamel in the longitudinal direction. B) Dentin in the transverse direction. The area where the drops of water were placed is indicated by white rectangles.