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AFM in mode Peak Force applied to the study of un-worn contact lenses

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ABSTRACT

Contact lenses (CLs) are of common use and the biocompatibility, topography and mechanical properties of the used materials are of major importance. The objective of this contribution is to apply the AFM in mode Peak Force to obtain surface topography and mechanical characteristics of un-worn CLs of different materials. One material of hydrogel, two of siloxane-hydrogel and one of rigid gas-permeable were used in the study. The results obtained with different materials have been compared, at a nanoscopic level, and the conclusions are diverse. There is no significant influence of the two environments used to measure the characteristics of the CLs, either water or saline solution. The pHEMA hydrogel CL (Polymacon of Soflens) shows the highest values of roughness, adhesion and elastic modulus. The siloxane-hydrogel CL named Asmofilcon A of PremiO presents the lowest values of mean roughness (R_a), root-mean-square roughness (RMS or R_q), adhesion (Adh) and elastic modulus (Y_m), meanwhile the siloxane-hydrogel CL named Lotrafilcon B of Air Optix presents the lowest value of skewness (R_{sk}) and the rigid gas-permeable CL, named RXD, presents the lowest values of kurtosis (R_{ku}) and maximum roughness (R_{max}).

1. Introduction

Contact lenses (CLs) are of common use for visual-defect corrections. CLs, in contrast to spectacles, are in contact with the eye and consequently the biocompatible properties of the used materials and their topographic and mechanical characteristics are of major importance in order to full fit the ocular tissues requirements and do not provoke injuries during the wear. Manufacturers of CLs look for materials with low values of the elastic modulus, as well as a good level of wettability. Topographic and more specifically mechanical properties at a nanometric level have been considered only in a limited number of papers. The atomic force microscopy (AFM) is a technique that becomes useful to determine these properties, as was pointed out in an earlier work by Rabke et al. [1]. Consequently, several authors [2–15] performed studies on CLs using the AFM technique, but none of them used the Peak Force Quantitative Nanomechanics mode to determine the nanomechanical properties, such as the adhesion or the Young modulus, also named elastic modulus.

Grobe et al. [2] studied the surface chemistry and topography of Etafilcon-A hydrogel CLs as a function of polymer processing, by using AFM, and concluded that the processing technique has influence in both the surface chemistry (surface composition, wettability) and the topography. Maldonado-Codina and Efron [8] examined the impact of manufacturing and material composition on the surface topographic characteristics of five types of hydrogel CLs. Observations were made using both scanning electron microscopy (SEM) and AFM, indicating that cast-molded lenses show lower root-mean-square (RMS) roughness values, in agreement with the results of Rabke et al. [1] and Grobe et al. [2]. Kim et al. [4,5] investigated the surface of hydrogel pHEMA-based CLs by using AFM and measuring the friction and adhesive forces. They found that in saline solution, these magnitudes were significantly reduced, compared to those measured for the surface-dehydrated state. Opdahl et al. [6] and Koffas et al. [7] investigated the surface mechanical properties of pHEMA-based hydrogel CLs as a function of ambient relative humidity and bulk water content by using AFM force curves. They found a balance in between the dehydration and hydration rates at around 50–60% relative humidity.

From the development of the new siloxane-hydrogel (Si-H) materials, studies were also made on this type of CLs.

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Table 1
Topographic values for different hydrogel (H) and siloxane-hydrogel (Si-H) CLs, reported by different authors. R_a: mean roughness, R_q: root-mean-square roughness.

Name	Material and manufacturer	R_a (nm)	R_q (nm)	Reference
Lotrafilcon A	Si-H Ciba Vision	3.6	4.67	[10]
		7.3	9.8	[12]
Balafilcon A	Si-H Bausch & Lomb	9.55	12.26	[10]
		4.8	6.6	[12]
		7.04	9.5	[13]
Galyfilcon A	Si-H Johnson & Johnson	5.39	6.75	[10]
		0.7	0.8	[12]
		2.32	3.04	[13]
Etafilcon A	H Johnson & Johnson	3.2	3.9	[12]
Nefilcon A	H Ciba Vision	4.8	2.9	[12]
		11.25	15.41	[14]
Vasurfilcon	H Ciba Vision	2.0	2.6	[12]
Filcon 1A	H Wilens	0.9-2.0	1.2-2.8	[12]
	H Wichterle & Vacik	16.8	26.4	
Filcon 4A	H Ciba Vision	18.8	24.1	[12]
Lotrafilcon B	Si-H Ciba Vision	5.1	7.3	[12]
		4.5	5.7	[13]
Omafilcon	H Cooper Vision	1.9	2.78	[14]
Hioxifilcon	H MarkEnnovy	4.31	5.5	[14]
Ocufilcon B	H Cooper Vision	11.01	14.38	[14]
Senofilcon A	Si-H Johnson & Johnson	3.33	4.06	[14]
Comfilcon A	Si-H Cooper Vision	1.56	2.34	[14]

González-Méijome et al. [10] studied several Si-H materials, shown in Table 1, and concluded that Balafilcon A presents a more irregular surface, probably due to the plasma oxidation treatment used to improve wettability, having Galyfilcon A and Lotrafilcon A the smother surface. Lira et al. [13] studied three different Si-H materials (see Table 1) and also observed that Galyfilcon has the smother surface, meanwhile Balafilcon A has the more irregular one. Guryca et al. [12] studied several Si-H and hydrogel materials and obtained that the surface roughness depends on the technique used to fabricate the CLs. They observed that the lathe-cutting technique provides more roughness, meanwhile both the spin-coating (as hydrogel Filcon 1A from Wilens) and cast-molding (as Si-H Galyfilcon A) techniques provide less roughness. Giraldez et al. [14] also studied several hydrogel and Si-H materials and observed that the flattest surface corresponds to both hydrogel Omafilcon A and Si-H Comfilcon A, among those studied. From Table 1, it can be seen that the roughness shown by the CLs depends more on the manufacturer, the method of manufacture, or the surface treatment, than on the material, since some hydrogel CLs are less rough than other Si-H CLs, and on the contrary. Also, different authors report different values for the same material. Finally, Zhou et al. [15] proposed to measure the frictional properties of CLs since they could have a great impact on their clinical performance.

The main goal of this paper is to obtain the adhesion and the Young's modulus of different CL materials in different aqueous media, water and saline solution, using the Peak Force mode, and to compare the values. The Peak Force mode is a recent development in AFM that permits to obtain nanomechanical properties, as Young's modulus and adhesion, at the same time that a topographic image of the surface sample is registered. The Peak Force mode covers a wide range of Young's modulus values, between 1 MPa and 50 GPa, and adhesion values, between 10 pN and 10 μN. The Peak Force mode operates in intermittent contact mode controlling the maximum applied force and performing force curves in each contact point. More details on the AFM technique can be found in Refs. [16,17] and on the Peak Force mode in Refs. [18,19]. For one of the selected materials (Si-H), there is no references in literature concerning the adhesion and elastic modulus at a nanometric level. The treatment of the obtained data also provides topographic images and statistical values of mean roughness (R_a), root-mean-square roughness (RMS or R_q), kurtosis (R_{ku}) and skewness (R_{sk}), and for one of the CLs, named PremiO, there was not a reported topographic analysis in literature. Finally, the work provides these values for different materials of CLs using the same technique and procedure, which affords a higher level of confidence in comparing them.

2. Materials and methods

Soft CLs of two different siloxane-hydrogel (Si-H) materials, commercialized as PremiO (P), from Menicon, and Air Optix (AO), from CIBA Vision, one soft CL of pHEMA hydrogel (H) material (Soflens, from Bausch & Lomb) and one rigid gas-permeable CL of silicone-based material, RGP (RXD, from Boston), were used. The technical names and characteristics of the materials are shown in Table 2.

2.1. Sample preparation

The samples were prepared taking the CLs from the blister, washing gently with water, cutting a small piece of the central part (1×1 mm^2), fixing with glue on a clean Teflon support mounted on a magnetic plate, and immersing it either in saline solution or water. Prepared samples were left 30 min before imaging so as to ensure hydration equilibrium. Finally, the sample was mounted in a cell for liquid systems to perform the AFM experiment. In the case of RGP lenses, which are provided in a wetting solution, it is important to remove the wetting layer in order to obtain good AFM images and mechanical values.

2.2. Techniques and equipment

AFM in Peak Force Quantitative Nanomechanics mode was done, using a Multimode 8 and Nanoscope V electronics (Bruker). The study was carried out in liquid media using silicon nitride triangular cantilevers with pyramid tips of silicon oxide of low spring constant (0.35 nN/nm nominal). The resonant frequency in liquid was of 2 kHz and the peak force amplitude of 300 nm. A maximum vertical force between 0.5 and 3 nN was applied depending on the Young's modulus of the sample (a sample deformation over 3 nm is required). The selected piezo-scanner was for scanning small areas. The scanned area of the images here presented is 5×5 μm^2 and the analyzed parameters refer to this area (it is important to specify this information since the obtained values of the parameters depend

Table 2
Characteristics of the different CLs used in the study provided by manufacturers. Dk: oxygen permeability.

	PremiO	Air Optix	Soflens	RXD
Name	Asmofilcon A	Lotrafilcon B	Polymacon	Itabisfluorofocon A
FDA Group	I	I	I	–
% water	40	33	38	–
Surface treatment	Nanogloss™	Plasma polymerization	–	–
Contact angle	27	78	17	39
Dk·10^{11} (cm^2/s)·(ml O_2/ml mmHg)	129	110	8.4	24
Young's modulus (MPa)	0.9	1.2	0.44	≈5