Comparative Study of Silicone-Hydrogel Contact Lenses Surfaces Before and After Wear Using Atomic Force Microscopy

M. Lira,[1] L. Santos,[2] J. Azeredo,[2] E. Yebra-Pimentel,[3] M. Elisabete C. D. Real Oliveira[1]

[1] Department of Physics (Optometry), School of Sciences, University of Minho, Portugal

[2] Center of Biological Engineering, University of Minho, Portugal

[3] School of Optics and Optometry, University of Santiago de Compostela, Spain

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Abstract: ***Purpose:*** **The aim of this study was to analyze surface changes of three silicone-hydrogel contact lenses after daily wear. The lenses used in this study were balafilcon A, lotrafilcon B (both surface-treated), and galyfilcon A (non surface-treated).** ***Methods:*** **To understand how and where proteins, lipids, and other contaminants change contact lenses, surface roughness was assessed through Atomic Force Microscopy Tapping Mode^{TM}. Roughness parameters were Mean Surface roughness (R_a), Mean-square-roughness (R_q), and Maximum roughness (R_{max}). The surface topography of unworn and worn lenses was also mapped in great detail.** ***Results:*** **Contact lenses roughness parameters exhibited different values before and after wear and the surface appearance also changed. After wear, balafilcon A and galyfilcon A showed a significant increase on surface roughness parameters, being this increase more accentuated to galyfilcon A. In lotrafilcon B materials no significant changes were observed with wear.** ***Conclusions:*** **The present study suggests that surface treatment of silicone-hydrogel contact lenses can play a role in the prevention of a significant increase in roughness, and contribute to the better clinical tolerance of these lenses.** © 2007 Wiley Periodicals, Inc. J Biomed Mater Res Part B: Appl Biomater 85B: 361–367, 2008

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INTRODUCTION

Hydrogel contact lenses have been used for vision correction for over 30 years. However, because the oxygen permeability of conventional hydrogel contact lenses that depend mainly of their water content could not satisfy the needs of the cornea, particularly under the closed eye, motivate the introduction of silicone-hydrogel contact lenses.

The introduction of silicone-hydrogel contact lenses resulted in a new generation of soft contact lenses exhibiting almost the same comfort but significant higher oxygen permeability than that of conventional hydrogels.

Considering that the surface of a contact lens is in direct contact with the cornea and conjunctiva, it seems obvious that the surface properties are an important factor to be addressed due to the clinical implications that might have. Silicone-hydrogel contact lenses materials exhibit different surface properties on account of the incorporation of different chemical composition and surface treatment.[1,2] Surface treatments are performed in order to obtain wettable surfaces.[3] Balafilcon A (Purevision^{TM}, Bausch & Lomb, Rochester, NY) and lotrafilcon B (O_2Optix^{TM}, CIBA Vision, Duluth, GA) are treated using gas plasma techniques. Balafilcon A by plasma oxidation which transforms the silicone components into glassy islands on the surface, and lotrafilcon B through hydrocarbon plasma that reacts with air creating continuous hydrophilic surfaces.[1,4] Galyfilcon A (Acuvue® Advance^{TM}, Vistakon, Jacksonville, FL) has no surface treatment but incorporates an internal wetting agent that apparently leaches to the lens surface.

Atomic Force Microscopy (AFM) was shown to be a powerful tool for studying the surface properties of hydrophilic contact lens material in aqueous environments.[5,6] AFM is an excellent technique that enables the analysis of surface topography and roughness by means of a nondestructively methodology. The AFM consists of a microscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically made of silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into the proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. The great advantage of AFM compared with conventional microscopic techniques or scanning

Correspondence to: Dr. Maria Lira (e-mail: mlira@fisica.uminho.pt)

TABLE I. Silicone-Hydrogel Contact Lenses Used in This Study

Brand	Acuvue® Advance™	Purevision™	O_2Optix™
Manufacturer	Johnson & Johnson	Baush & Lomb	CIBA Vision
USAN	Galyfilcon A	Balafilcon A	Lotrafilcon B
Charge	Nonionic	Ionic	Nonionic
Water content (%)	47	36	33
Surface treatment	No surface treatment	Gas plasma oxidation	Plasma coating
RI	1.4055[a]	1.426[a]	1.42[a]
Principal monomers	mPDMS+DMA+EGDMA +HEMA+siloxane macromer +PVP+visibility tint+UV blocker	NVP+TPVC+NCVE +PBVC	DMA+TRIS+siloxane macromer+visibility tint

USAN, United States adopted names; DMA N, *N*-dimethylacrylamide; PDMS, polydimethylsiloxane; EGDMA, ethylene glycol dimethacrylate; HEMA, poly-2-hydroxiethyl-methacrylate; MA, methacrylic acid; NVP, *N*-vinyl pyrrolidone; TPVC, tris-(trimethylsiloxysilyl) propylvinyl carbamate; NCVE, *N*-carboxyvinyl ester; PBVC, poly[dimethysiloxy] di (silylbutanol) bis [vinyl carbamate].

[a] Obtained from Food and Drug Administration.

electron microscopy (SEM) are the high level resolution in three dimensions and its ability to obtain topographic information from the surface in aqueous, nonaqueous or dry conditions eliminating the need of sample preparation like, dehydration, freezing, coating, and so forth.

Conventional soft contact lens surfaces were imaged previously with AFM.[7] This technique brought important insights into the problem of adsorption of lachrymal components on the soft contact lenses.[8] More or less tear proteins can be deposited onto contact lenses depending of chemical composition. It is well known that ionic lenses with negative charges adsorb larger quantities of positively charge lysozime than the nonionic lenses.[9,10]

Tear deposits on contact lenses may cause discomfort to the patient.[11] Rabke et al.,[12] showed that AFM can be used to compare the surfaces of different conventional soft contact lenses. Also, Grobe et al. studied the surface roughness of soft contact lenses as a function of polymer processing.[13] Theses studies demonstrated that AFM can be useful in clinical studies to identify with more confidence the possible causes of contact lenses related discomfort or complications.

The aim of this study is to analyze by AFM the surface changes after daily wear, and the influence of the surface properties of two surface-treated silicone-hydrogel contact lenses, ionic and nonionic, respectively, and a third nonionic silicone-hydrogel lens not surface treated. It is expected that AFM analysis of the surface topography at a nanometric resolution may bring insights about the influence of lens surface treatment in the accumulation of deposits during wear and thus it is expected that this type of information will contribute to fasten the improvement of polymers and coatings agents. As far as we know, there are not earlier reported studies that analyzed lotrafilcon B by AFM.

MATERIAL AND METHODS

Contact Lenses

Three commercially available silicone-hydrogel contact lenses were examined. Lens specifications are described in Table I. One sample of unworn and worn lens from each of the three lenses was analyzed. It was performed at least three measurements per contact lens material. This analysis was performed on the anterior surface of each lens.

Clinical Trial

All lenses were used in a daily wear schedule for 30 days to establish comparisons. Overnight the lenses were kept in a commercially appropriate lens care solution (Renu Multiplus™; Bausch & Lomb, Rochester, NY). The patients were educated about regular cleansing and hygiene. After the worn period, lenses were removed from the patient's eyes and placed in sealed sterilised glass bottles with 5 mL of a preservative-free sterile saline solution (0.9 % NaCl, Sigma-Aldrich, Germany) and preserved at 4°C before being analyzed (48 h after removed from the eye). This procedure allows the contact lenses to maintain its properties and to prevent bacterial growth on the lens surface.

AFM Analysis

Surface roughness analysis was performed trough AFM using Tapping® Mode (PicoScan Controller 2500, Molecular Imaging, USA) using standard silicon tips. Tapping Mode® AFM is a technique in which the imaging probe is vertically oscillated near the resonant frequency of the cantilever. Electro-mechanical feedback maintains the oscillation at constant amplitude during scanning. A stiff cantilever is oscillated closer to the sample than in noncontact mode. Part of the oscillation extends into the repulsive regime, so the tip intermittently touches or ''taps'' the surface. The main advantage of tapping mode is the elimination of the lateral shear forces present in contact mode, which can damage the structure being imaged.

The roughness of a surface can be measured in statistical descriptors that give average behavior of the surface height. The roughness measurements regarding R_a, R_q, and R_{max}, were determined using the Scanning Probe Image Processor, SPIP™, version 4.2.2.0 software. R_a indicates the average distance of the roughness profile to the center plane of the profile and gives a good general description of

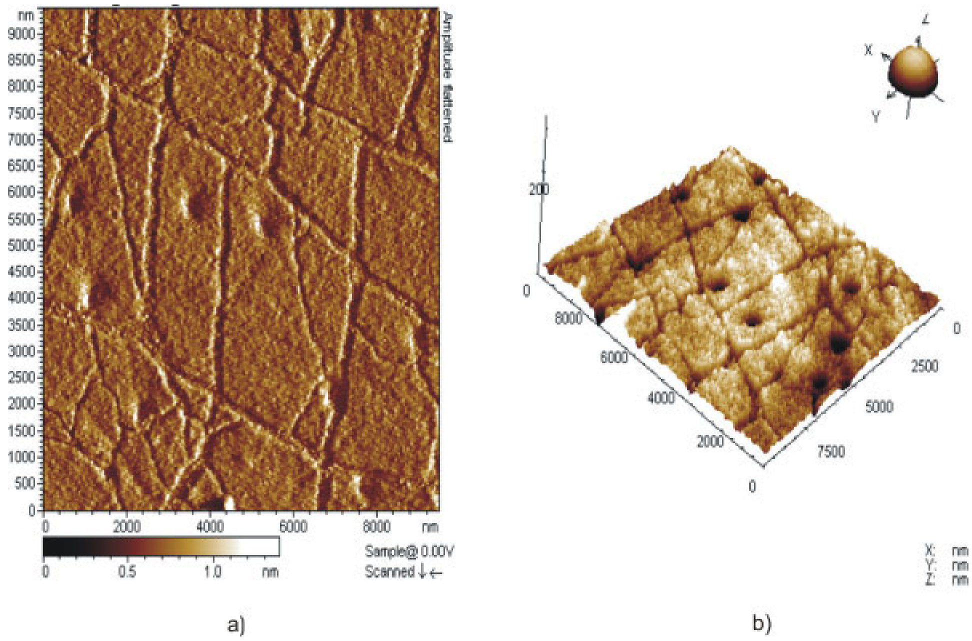


Figure 1. AFM analyses of unworn balafilcon A (100 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the height variations in the surface. R_q represents the standard deviation from the mean surface plane and R_{max} is the difference in height between the highest and lowest points on the observed area. Roughness measurements were performed according to two reference areas. For galyfilcon A and lotrafilcon B it was 5 μm × 5 μm B and for balafilcon A 10 μm × 10 μm in order to see certain structure which is not visible with lower magnifications. High quality images in three dimensions (3D) of the lens surface were recorded at randomly different contact lens surface locations to verify the reproducibility of the observed characteristics, from which the mean roughness (R_a) was calculated.

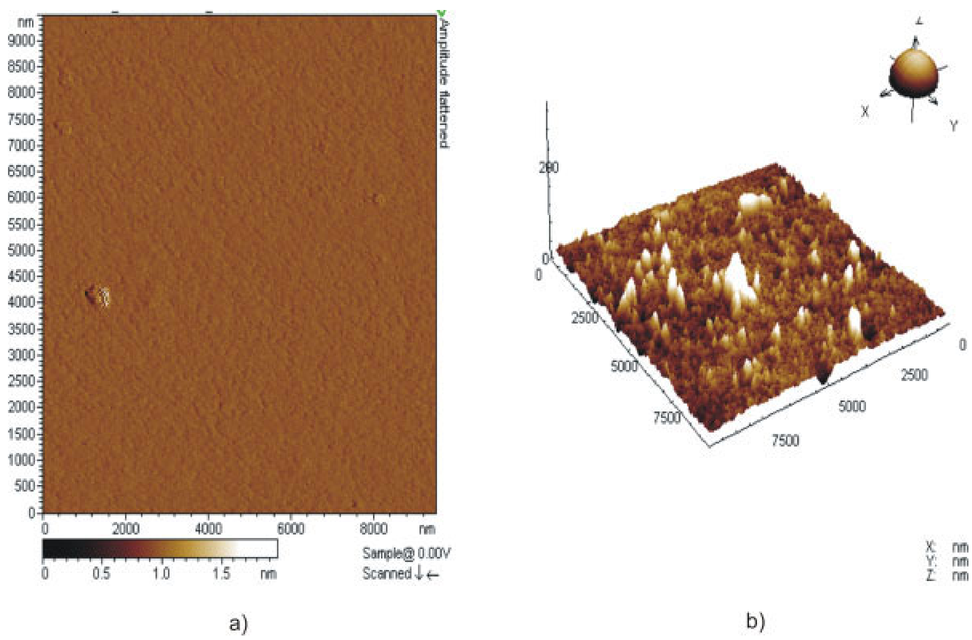


Figure 2. AFM analyses of worn balafilcon A (100 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

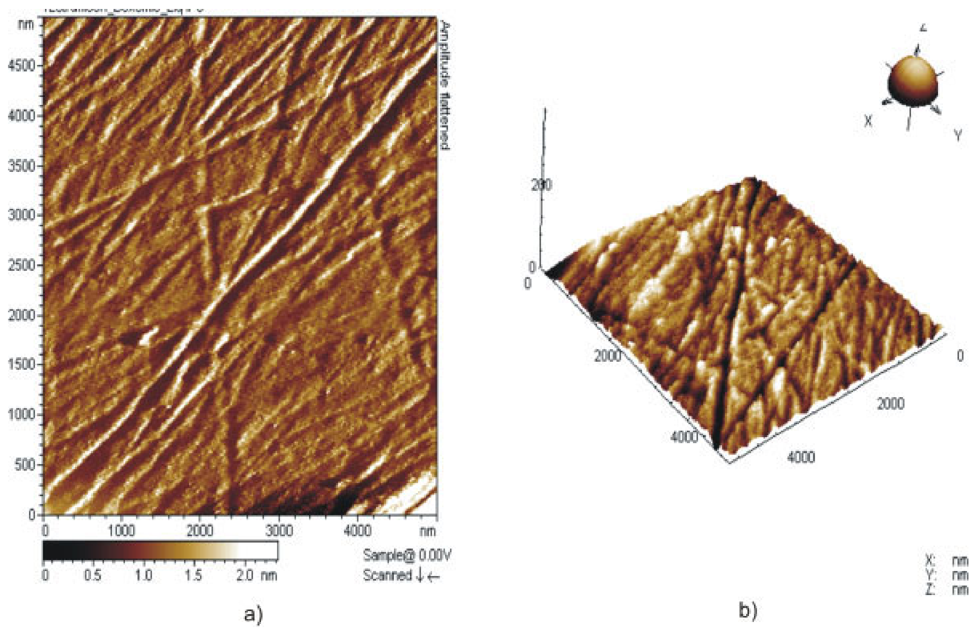


Figure 3. AFM analyses of unworn lotrafilcon B (25 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Samples were analyzed in a liquid cell (Agilent Technologies, USA) with preservative-free saline solution (0.9% NaCl, Sigma-Aldrich, Germany) to maintain contact lenses hydration during the observation and at room temperature. To perform AFM analyses, hydrated half lenses were fixed on a microscope slide with double-side sticky tape after cutting using a fresh scalpel blade and tweezers without inducing material bending. All preparation and handling were carried out using clean tools while the operator was wearing powder-free gloves. The measurements were repeated three times per contact lens material.

Statistical Analyses

The differences between roughness parameters for unworn and worn contact lenses were compared using Statistical Package for Social Sciences, SPSS Version 14, using the

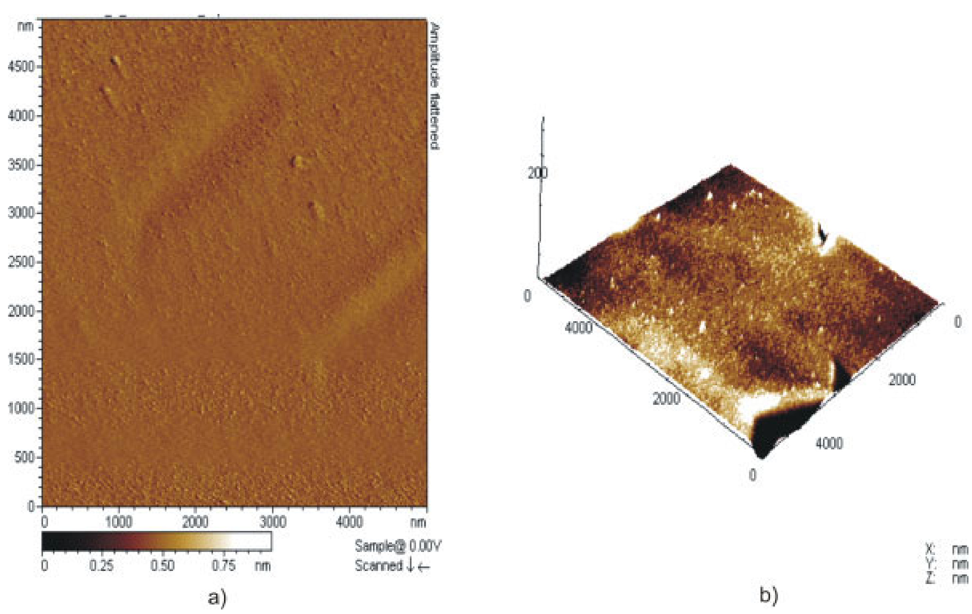
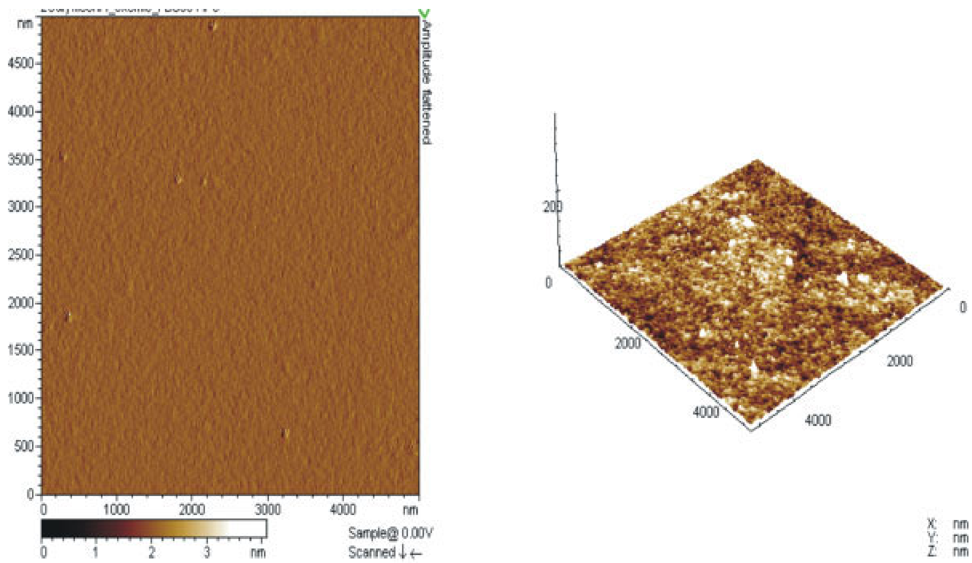


Figure 4. AFM analyses of worn lotrafilcon B (25 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



a) b)

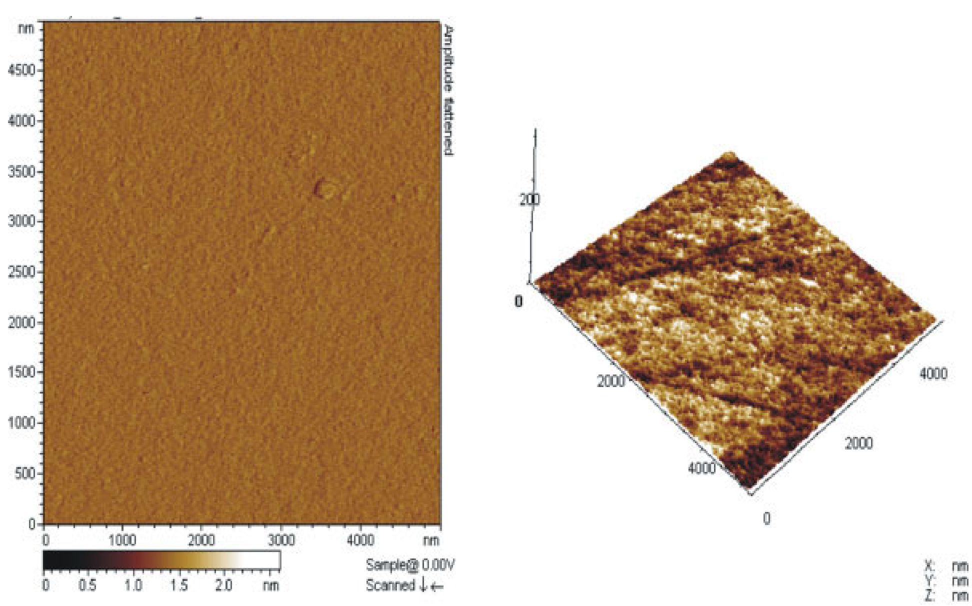
Figure 5. AFM analyses of unworn galyfilcon A (25 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

paired sample *T*-test for parametric data. For all hypotheses testing, $p \leq 0.05$ was considered statistically significant.

RESULTS

Roughness parameter R_a e R_q seems to be the most helpful and consistent to characterize surface topography of contact lenses.[14] R_{max} can be affected by local imperfections or sample contamination leading to higher values than expected and so material characterization based on this parameter could be unreliable. Figures 1(b)–6(b) show the 3D measurements of unworn and worn contact lenses. Table II summarizes the mean roughness parameters: the mean surface roughness (R_a), mean-square-roughness (R_q), and maximum roughness (R_{max}) obtained from those measurements.

Unworn galyfilcon A presented the smoothest, and flattened surface with a multitude of small peak (R_a = 2.32 ±



a) b)

Figure 6. AFM analyses of worn galyfilcon A (25 μm^2) (a) Topography (b) Three-dimensional image. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE II. Mean and Std Deviation Roughness Parameters of Contact Lenses Determined by AFM

	R_a (nm)	R_q (nm)	R_{max} (nm)
Galyfilcon A[a]	2.32 ± 0.1	3.04 ± 0.1	30.1 ± 5.5
Galyfilcon A[b]	30.9 ± 11.3*	40.0 ± 17.3	189.3 ± 93.9
Lotrafilcon B[a]	4.5 ± 2.3	5.7 ± 2.8	40.8 ± 12.0
Lotrafilcon B[b]	4.96 ± 4.1	7.3 ± 5.5	52.7 ± 23.5
Balafilcon A[a]	7.04 ± 0.7	9.5 ± 0.7	81.5 ± 7.3
Balafilcon A[b]	17.6 ± 14.8	23.7 ± 15.2	138.7 ± 40.5

* Statistically different compared to unworn contact lenses ($p \leq 0.05$).
Number of measurements per contact lens material: 3.
[a] Unworn.
[b] Worn.

0.1 nm, R_{max} = 30.1 ± 5.5 nm) followed by lotrafilcon B (R_a = 4.5 ± 2.3 nm, R_{max} = 40.8 ± 12 nm). Balafilcon A exhibited the rougher surface (R_a = 7.04 ± 0.7 nm, R_{max} = 81.5 ± 7.3 nm).

The surface topography of unworn lenses can be observed in Figures 1(a), 3(a), and 5(a). Balafilcon A was observed over 100 μm^2 and at higher magnification in order to see the pores that are not visible at lower magnification. The lens plasma surface treatment can be seen with an excellent resolution. Lotrafilcon B and galyfilcon A were scanned over an area of 25 μm^2. From the observation of these figures we can see that the surface-treated contact lenses, balafilcon A and lotrafilcon B exhibits typical structures, respectively pores and grooves, which is probably related with the presence of surface treatment. Galyfilcon A showed a smooth surface corroborating the obtained values for roughness.

Regarding surface roughness parameters changes with wear (Table II) it was observed a greater increase of roughness on galyfilcon A from 2.32 ± 0.1 nm to 30.9 ± 11.3 nm and on Balafilcon A from 7.04 ± 0.7 nm to 17.6 ± 14.8 nm. Lotrafilcon B did not register any noticeable change with wear.

From observation of the Figures 2(a), 4(a), and 6(a) we can see that the surface appearance observed for the surface-treated lenses dramatically change. The large pores observed for balafilcon A and the grooves present in lotrafilcon B stop being visible probably on account of accumulation of tear deposits.

DISCUSSION

Tapping Mode® is currently the most successful mode for high-resolution imaging of soft or delicate samples. In the present study, AFM Tapping Mode® was used to evaluate any changes induced by wear regarding roughness and surface appearance to help understand corneal damage mechanisms and interfacial deposit formation.

It was found that unworn lenses exhibit different roughness parameters and surface topography probably on account of different surface treatment or the lack of it.[14] In balafilcon A [Figure 1(a)] and lotrafilcon B [Figure 3(a)] which are surface-treated lenses it was observed the presence of pores and a pattern of numerous grooves with different orientations respectively. These structures are different due to the distinct surface treatments, as described in the Introduction. Galyfilcon A exhibits a more homogeneously structure [Figure 5(a)] with a granulated appearance, as it was also observed with Cryogenic Scanning Electron microscopy (cryoSEM).[15] Balafilcon A was observed over 100 μm^2 and at higher magnification in order to see the pores that are not visible at lower magnification. The lens plasma surface treatment can be seen with an excellent resolution. Lotrafilcon B and galyfilcon A were scanned over an area of 25 μm^2.

It is clear from this study that the surface of worn silicone-hydrogel contact lenses are damaged and significantly altered during wear. The surface damage of these contact lenses may have occurred due to formation of interfacial deposits and wear debris, and also by abrasion of the lens surface. Regarding these surface changes with wear, the main findings are that the roughness in galyfilcon A and balafilcon A largely increase when we compare unworn with worn contact lenses suggesting that these lenses are more prone to the formation of deposits than lotrafilcon B. After wear, balafilcon A does not display either islands or ridges as it can be observed in Figure 2(a). The pores seen before wear are not observed after wear, which suggest that this ''holes'' are probably filled with components possibly derived from the adsorption of the tear film. The same observation can be made in the case of lotrafilcon B [Figure 4(a)], in which the existing groves in unworn lenses disappeared after worn. It seems that during wear, deposits are formed on the lens surface covering their original irregularities, giving a more uniform appearance, nevertheless contributing to an increase in their surface roughness parameters. Previous studies have reported that silicone-hydrogels are more susceptible to lipids than protein adsorption,[16] and that the lipids on the lens surface may diminish protein adsorption.[17] Changes on the surface of worn contact lenses could affect the clinical performance of the lenses, due to lens spoliation with tear residues and bacteria colonization that leads to biofilm formation and also changing the biocompatibility of the contact lens materials.

Galyfilcon A, the lens without surface treatment showed a significant increase in the roughness parameter with the higher score of values. Additional related experiments were also conducted to help explain the mechanisms that can lead to contact lens surface damage and formation of deposits.[18] It was proved that although galyfilcon A is not more prone to total protein adsorption than the other silicone-hydrogel contact lenses, this lens exhibited a greater diversity of adsorbed proteins when compared with all the other lenses,[18] which may be related to this change in roughness. Also, the proteins profiles that have been reported for galyfilcon A have the highest molecular weight than in other contact lenses.[18] Despite of this increase in roughness, it was not proved that after wear, Galyfilcon A was more prone to bacterial adhesion than the other lenses studied,[18] although Vermeltfoort et al.,[19] demonstrated that bacterial transfer is determined by the roughness and hydrophobicity of the contact lens surface which receives the bacteria.

Although bulk properties of the contact lenses material, ionicity and water content may influence the wettability, resistance to deposits, and bacterial attachement,[20] the interaction with the tear film can also determine contact lens performance and its biocompatibility. Changes in surface roughness have also been suggested to affect the comfort of wearing contact lenses[21] so, the usual discomfort noted by patients at the end of each recommended period of wearing time could be related to these increments in surface roughness.

The present study suggests that surface treatment of silicone-hydrogel contact lenses can play a role in the prevention of a significant increase in roughness, and contribute to the better clinical tolerance of these lenses.

The mechanical interaction between the ocular surface and silicone-hydrogel contact lenses has been one of the main concerns for the clinical tolerance of these lenses, since contact lens spoilage has been implicated in a wide range of complications, including reduced wettability of the lens surface, symptoms of dryness, discomfort, and visual disturbance.[22] It is hoped that the observations described here might stimulate the development of improve surface treatments for new silicone-hydrogel lenses, and/or develop newer polymers for hydrogels with similar high oxygen permeability of the currently available silicone-hydrogel contact lenses.

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