MICROSCOPIC OBSERVATION OF SILOXANE-HYDROGELS WITH THREE DIFFERENT TECHNIQUES

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1) INTRODUCTION
The mechanical impact of siloxane-hydrogel soft contact lenses (Si-Hi SCL) on corneal surface has been considered⁵,⁶, and the mechanical properties of these lenses could affect importantly several aspects of clinical response of the ocular surface.⁷ Scanning electron microscopy (SEM) has been previously applied to contact lens (CL) polymers,⁸ as well as atomic force microscopy (AFM)⁹,¹⁰ The purpose of this study was to observe different siloxane-hydrogel contact lens materials under three microscopic techniques in order to identify which one gives us more conclusive information of the surface polymer structure.

2) METHODS
2.1) CONTACT LENSES
First generation of Si-Hi SCL Focus Night & Day™ (Iolaris A: CIBA Vision, Duluth, GA) and Purevision™ (Balatrac A; Bausch & Lomb, Rochester, NY), and newer Si-Hi SCL Acuvue Advance™ (golyficon A: Vistakon, Jacksonville, FL) were used in this study. Technical details are summarized in Table 1.

2.2) ATOMIC FORCE MICROSCOPY (AFM)
AFM examination was carried out in the hydrated state using the Tapping™ Mode facility of a Nanoscope III (Digital Instruments, Santa Barbara, CA). Cantilevers with a nominal force constants of k=0.07 N/m and k=0.022 N/m, and oxide-sharpened SiN4 tips (Olympus Ltd., Tokyo, Japan) were used for imaging. AFM statistics, including mean roughness (Ra) and maximum height (Rmax) were explored for the three lens materials. The three samples were scanned over lengths of 20, 10, 5 and 1 µm, given a surface area of approximately 400, 100, 25 and 1 µm².

2.3) Cryo-SCANNING ELECTRON MICROSCOPY (CryoSEM)
Samples were frozen in slush N₂ and attached to the specimen holder of a CT-100C Cryo-transfer system (Oxford Instruments, Oxford, UK) interfaced with a JEOL JSM-5410 scanning electron microscope (SEM). Surface water was sublimed by controlled warming to -99°C and samples were examined at an accelerating voltage of 15kV.

2.4) SCANNING ELECTRON MICROSCOPY (SEM)
The lenses were dehydrated in graded ethanols in 0.9% NaCl (15, 30, 50, 70, 96, and 100%) being kept in the respective ethanol concentration twice for 15 min each, using a fresh solution each time. Then, the lenses were dried to critical-point in CO₂ in an Autosamdi 814 (Tousimis). This was done by repetitive exchanges with liquid CO₂, and then warming the chamber to the critical point of CO₂ (31.04°C, at 72 atm). After 10 min the chamber pressure was slowly reduced to atmospheric pressure to obtain the dry lens.

3) RESULTS
3.1) ATOMIC FORCE MICROSCOPY (AFM)
Representative AFM micrographs of each CL material are presented in figure 1. Quantitative roughness parameters are listed in figure 2. Acuvue Advance™ (golyficon A) shows a particular surface structure that consists of a homogeneously distributed pattern of globular formations. Focus NAD™ (Golyficon A) material exhibits a pattern of linear marks on the material Golyficon A was significantly smoother than Iolaris A. Silicone islands structure, which significantly increase surface roughness and macro pores (diameter could reach 0.6 microns) are seen with excellent resolution on Purevision™ (Balatrac A) contact lens.

3.2) Cryo-SCANNING ELECTRON MICROSCOPY (CryoSEM)
Microphotographs presented in figure 3 highlight the ultrastructure linking the outer layer and the polymer bulk at lower and higher magnification. These images show how different is the assembly pattern in the three polymers. Balatrac A CL shows the tighter network whose pores vary in size and density. Of particular relevance is the round appearance adopted by the terminal ramifications of the structure.

3.3) SCANNING ELECTRON MICROSCOPY (SEM)
Differences between the outer aspect of the three Si-Hi materials are also seen under SEM (figure 4). Nevertheless, it seems that for smoother surfaces as lenses made of Golyficon A, resolution of SEM seems to not offer additional information regarding of surface structure.

4) CONCLUSION
1) The three materials present different characteristics under the three microscopy devices.
2) AFM allows quantitative and qualitative evaluation of CL in the hydrated state, specially to study surface topography, although other important properties as friction, Young modulus and adhesion can also be studied.
3) Although not used with CL, CryoSEM depicts important features about surface and ultrastructure characteristics, with meanings still to be clarified, and could be of special relevance in CL submitted to surface plasma treatment in order to elucidate the interaction of plasma with underlying material.
4) SEM does not seem a very useful tool for microscopic study of soft CL, as it is the most time consuming technique, seriously affecting integrity and structure of the material without offering additional information compared to other techniques.

References available upon request to the corresponding author: jmgmejia@feesca.uminho.pt

[Table 1: Technical details of the CL]

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Material (USAN)</th>
<th>FDA Group</th>
<th>Manufacturing Process</th>
<th>Surface Treatment</th>
<th>Hydration (%)</th>
<th>1D (kex)</th>
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<td>Plasma oxidation</td>
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<td>III</td>
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[Figure 1: Surface appearance of Si-Hi SCL, under AFM for a scanning surface area of 100 µm²]

[Figure 2: Quantitative roughness parameters of the three materials for a 100 µm² scanning area]

[Figure 3: Surface appearance of CL under CryoSEM at different magnification]

[Figure 4: External aspect of the siloxane-hydrogel materials under SEM]