

# 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 considered<sup>[1-3]</sup>, and the mechanical properties of these lenses could affect importantly several aspects of clinical response of the ocular surface.<sup>[4]</sup> Scanning electron microscopy (SEM) has been previously applied to contact lens (CL) polymers,<sup>[5]</sup> as well as atomic force microscopy (AFM).<sup>[6]</sup> 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<sup>TM</sup> (lotrafilcon A; CIBA Vision, Duluth, GA) and Purevision<sup>TM</sup> (balafilcon A; Bausch & Lomb, Rochester, NY), and newer Si-Hi SCL Acuvue Advance<sup>TM</sup> (galyfilcon A; Vistakon, Jacksonville, FL) were used in this study. Technical details are summarized in table 1.

Table 1. Technical details of the CL

	Acuvue Advance	Focus Night & Day	Purevision
Manufacturer	Vistakon J&J	Ciba Vision	Bausch & Lomb
Material (USAN)	galyfilcon A	lotrafilcon A	balafilcon A
FDA Group	I	I	III
Manufacturing Procedure	Cast molding	Cast molding	Cast molding
Surface Treatment	-	Plasma coating	Plasma oxidation
Hydration	47%	24%	36%
Dk (barrier)	60	140	99

### 2.2) ATOMIC FORCE MICROSCOPY (AFM)

AFM examination was carried out in the hydrated state using the Tapping<sup>TM</sup> Mode facility of a Nanoscope III, (Digital Instruments, Santa Barbara, CA). Cantilevers with a nominal force constants of  $k=0.094$  and  $k=0.022$  N/m and oxide-sharpened Si<sub>3</sub>N<sub>4</sub> 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  $\mu\text{m}$  given a surface area of approximately 400, 100, 25 and 1  $\mu\text{m}^2$ .

### 2.3) Cryo-SCANNING ELECTRON MICROSCOPY

Samples were frozen in slush N<sub>2</sub> 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 -90°C and samples were examined at an accelerating voltage of 15KeV.

## 2.4) SCANNING ELECTRON MICROSCOPY (SEM)

The lenses were dehydrated in graded ethanol 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 CO<sub>2</sub> in an Autosamdri 814 (Tousimis). This was done by repetitive exchanges with liquid CO<sub>2</sub>, and then warming the chamber to the critical point of CO<sub>2</sub> (31.04 °C, at 73 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 microphotographs of each CL material are presented in figure 1. Quantitative roughness parameters are listed in figure 2. Acuvue Advance<sup>TM</sup> (galyfilcon A) shows a particular surface structure that consists of a homogeneously distributed pattern of globular formations. Focus N&D<sup>TM</sup> (Lotrafilcon A) material exhibits a pattern of linear marks on the material Galyfilcon A was significantly smoother than lotrafilcon A. Silicate islands structure, which significantly increase surface roughness and macro pores (diameter could reach 0.5 microns) are seen with excellent resolution on Purevision<sup>TM</sup> (Balafilcon A) contact lens.

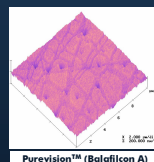
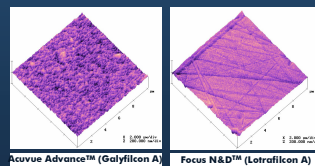


Figure 1. Surface appearance of Si-Hi SCL under AFM for a scanning surface areas of 100  $\mu\text{m}^2$ .

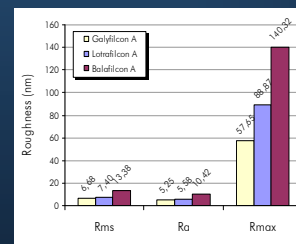


Figure 2. Quantitative roughness parameters of the three materials for a 100  $\mu\text{m}^2$  scanning area.

### 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. Balafilcon 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.

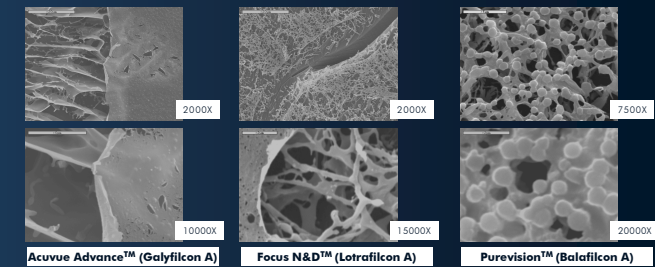


Figure 3. Surface appearance of CL under CryoSEM at different magnification.

### 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 Galyfilcon A, resolution of SEM seems not to offer additional information regarding of surface structure.

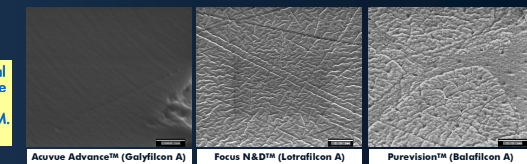


Figure 4. External aspect of the siloxane-hydrogel materials under SEM.

## 4) CONCLUSION

- The three materials present different characteristics under the three microscopy devices.
- AFM allows qualitative and quantitative 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.
- Although not used with CL, CryoSEM depicts important features about surface and ultra-structure 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.
- 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.

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References available upon request to the corresponding author: jgmeijome@fisica.uminho.pt