Structural Changes of Polysulfone Membrane Use for Hemodialysis in the Consecutive Regime. Nanometric Analysis by AFM

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ABSTRACT

Nowadays, the hemodialytic treatment of patients with either acute or chronic renal failure has been improved by promoting biocompatibility in the use of new materials and improve membrane surface characteristics. Low and high flux polysulfone membranes (PM) used in dialysis and ultra filtration have been studied in order to understand the geometry and surface chemistry of the pores at inner (nanometric) and outer (micrometric) membrane parts. The surface changes of polysulfone cartridge membrane (PM) during different number of consecutive reuse trials: after 1st, 10th and 23th times of use. The morphology of the hollow fibers surfaces was studied by means of the atomic force microscopy (AFM) imaging and the surface roughness analysis. The roughness of both inner and outer part of PM surface increases with numbers of reuse trails. Thus, small and medium size pores were wiped out when the number of uses changed from zero to 23 on the outer surface. The pore density decreases. The inner part of membrane shows some nanometric size deformation in forms of new openings and raptures. The AFM analysis show differences in the PM morphology at the nanometric level, not previously revealed, which could be important in the evaluation of the PM.
Keywords: hemdialysis, AFM, nanoporosity, membrane, imaging, surface, roughness, polysulfone

1. INTRODUCTION

Number of persons affected by some kind of kidney failure reach around 1% of Mexican population, from which around 20 000 are in the critical phase and need transplantation or kidney replacement. Among the existed therapies to substitute the kidney function, the hemodialysis could be a favorable option. Patients need to take treatment just two to three times per week, to eliminate toxic metabolite residues from the body and control retention of the body liquids [1]. Therefore it is not a surprise that hemodialysis is one of the favorable option. Still many problems should be resolve to achieve more optimal solutions: (a) decrease a treatment time from nowadays 3.5 h, (b) increase efficiency of the purification, especially for urea, creatinine, phosphates and decrease the immunologic reaction [1,2]. All need to be taken into account during the design and manufacture of the hemodialysis cartridges. There are several type of membranes that may be used for hemodialysis, made from polysulfone (the most commune) , polyamide, polyethersulfone, etc. Membrane surface morphology is an important factor in the process the cartridge quality and biocompatibility evaluation. Often it is based on the pore size and pore distribution measurements, on the membrane surface. So far different microscopes have been used for membrane analysis; however the Atomic Force Microscopy, poses a significant advantage than other techniques, offering: 3D images at the nanometric level of resolution, quantitative interpretation and work in the water solutions [4]. Here we used AFM analysis to understand the membrane morphology changes during the consecutive reused trail of dialyzer cartridges, up to 23 times. The effect of the consecutive and multiple uses was monitored by analysis of the pore size and the membrane surface roughness [5-8]. The analysis was performed at the inner (nanometric size porosity) and external part of the hollow polysulfone fibers.

Fourth International Conference on Smart Materials and Nanotechnology in Engineering,
edited by Jayantha A. Epaarachchi, Alan Kin-tak Lau, Jinsong Leng, Proc. of SPIE Vol. 8793,
87931Q · © 2013 SPIE · CCC code: 0277-786X/13/$18 · doi: 10.1117/12.2027844

The obtained results clearly show the process of the membrane contamination and indicate on the possible solutions for the future membrane design.

2. EXPERIMENTAL SECTION

The new and used hemodialysis cartridges were obtained from the hospital: Instituto Nacional de Crdiologia, The Kidney Transplant Unit., Mexico D.F. The process of the sample preparation is presented at Fig.1. During the first steps, cartridges were disconnected from the hemodialyzer and immediately rinsed and preserved in the formaldehyde solution. As such, were transported in the laboratory for analysis, rinsed by ultrapure MiliQ water and physically open to extract the hollow fibers of polysulfone. In order to prevent a possible contamination of the fibers as well as viral infection of operators, all was performed in an especially designed clean aseptic space. As following, fibers were drayed 72 h at the room temperature and then cut to expose the inner part to further analysis, too. External and inner parts of each hollow fiber were analyzed by AFM (Nanoscope III, Digital Instruments, Santa Barbara, USA). All images were collected in the tapping mode, with Si-TESP tips (L: 125 µm, F: 304-383 Hz. For the quantitative interpretation the software accompanied to AFM, was used.

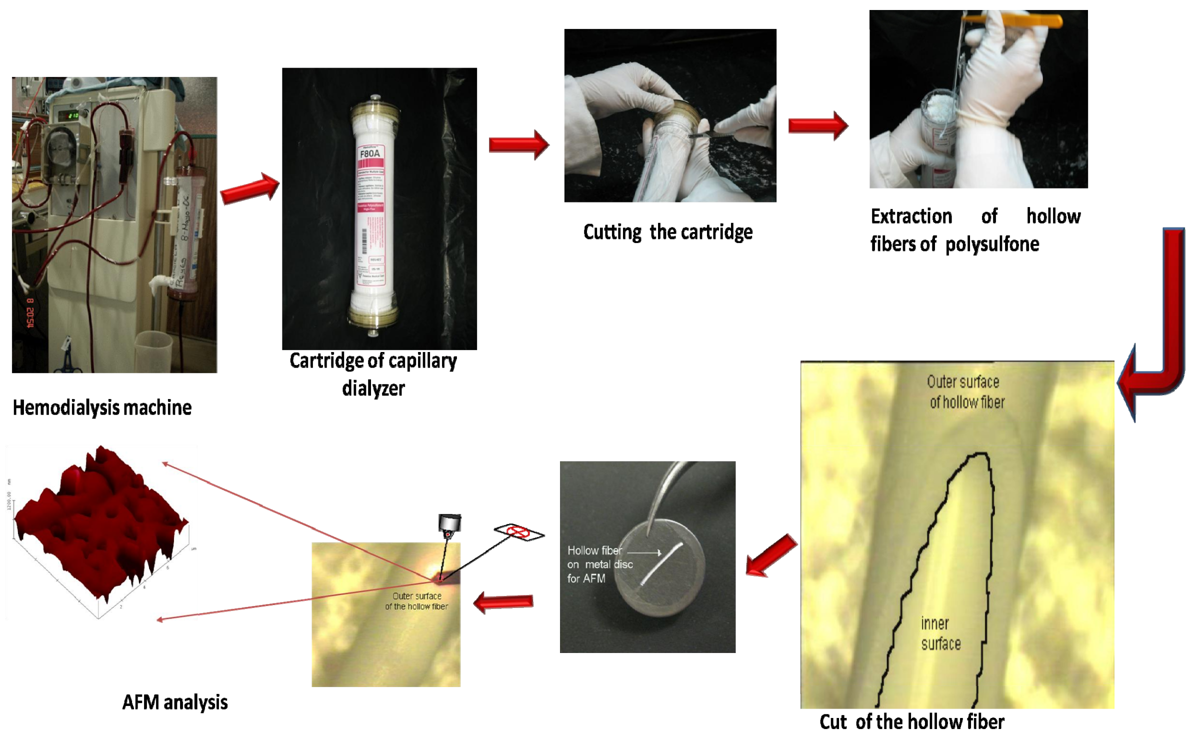


Fig.1. Schematic representation of the sample preparation for AFM analysis.

Process described above was repeated for all samples with different usage: 0, 1, 10 and 23 times.

3. RESULTS AND DISCUSSION

Before to present results of the AFM analysis, we should point out that each fiber of the polysulfone membrane has external (outer) and inner part. Across the fiber body is a conical type of pore with large (micron size) openings at the external part and small (nanosize) pores at the inner part. Fig.2. shows membrane structure obtained for new cartridge previous to any use. The 3D AFM image (a) clearly show existence of the large, micrometer size pores with diameter between 0.5 and 1.5 µm, at the membrane surface, equally distributed all over the sample. After consecutive use the surface morphology of the outer part significantly changes. Number of pores decreases (b), pore diameter increases, surface is swallowing (c) and finally after 23 times of use, the surface poses just few pores, much larger diameter than at beginning (3 – 5 µm) and with different texture.

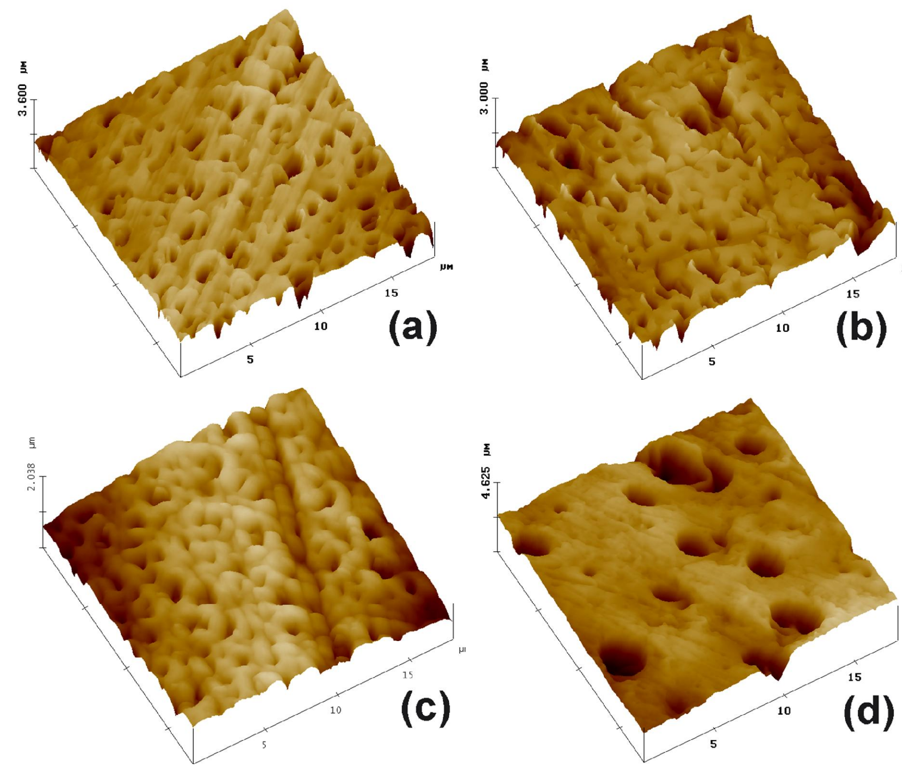


Fig.2. AFM images of the external (outer) membrane surface, image size 17.9 µm x 17.9 µm.: (a) zero uses (control); (b) single trail; (c) after 10 time of use; (d) after 23 times of use.

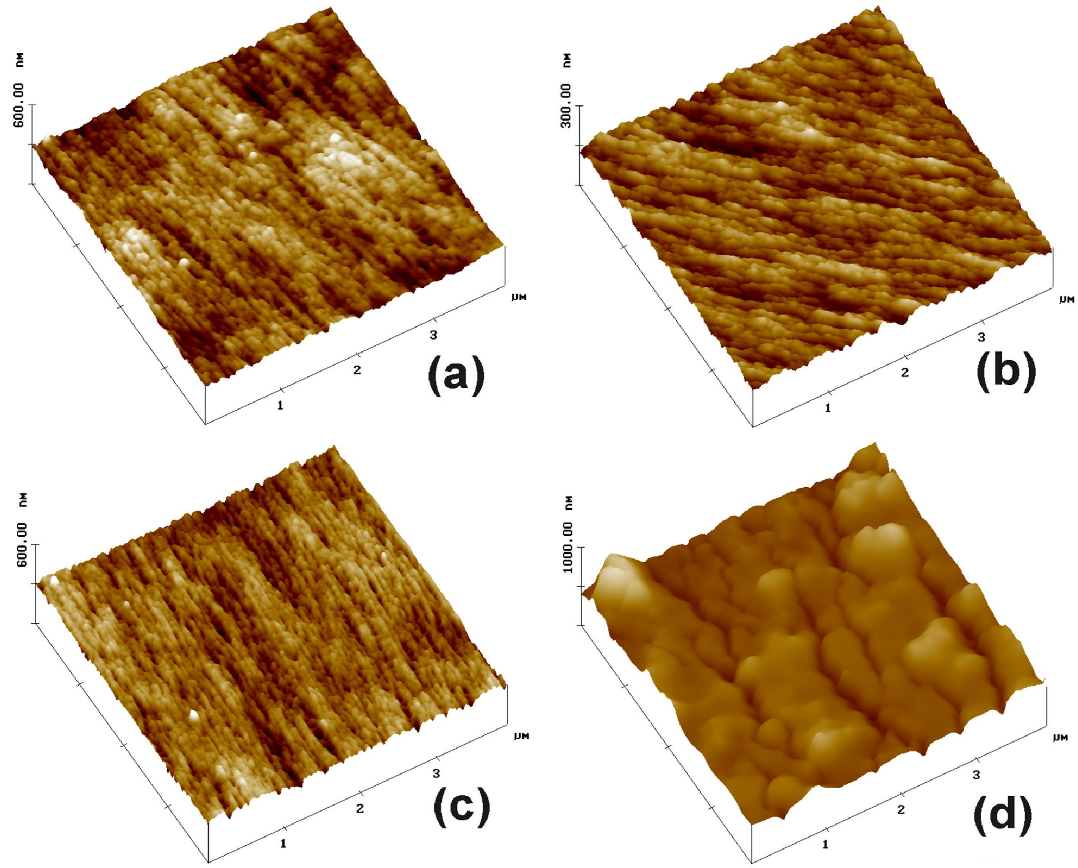


Fig.3. AFM images of the membrane surfaces at the inner fiber part, image size 3.9 μm x 3.9 μm.: (a) zero uses (control); (b) single trail; (c) after 10 times of use; (d) after 23 times of use.

To analyze inner part of the fiber was much more difficult. Some of pores have been just 1 to 3 nm of diameters, like as we suppose it is a natural kidney structure. Majority pores have diameter from 8 to 12 nm, and we found to be organized in a regular manner, giving some smooth and fine surface, (Fig.3.a.). After a single use, the most probably due to hemodialysis pressure, we observed some raptures along the nanometric lines of pores. (Fig.2.b). It seems to be some kind of irreversible damage of the membrane surface. Furthermore it will be the same, and after 23 uses, a drastic change on the membrane surface could be observed. So far it is not clear what is showing the AFM image at the Fig.2.d.: is it a new layer (contamination) deposited on the top of the fine membrane structure or the membrane finally lost such fine features and converts into surface with large parches separated with deep raptures. However we could say that observed features poses significant stability and consistence, since obtained images are very clear,sharp. In the case of the soft material (deposit or membrane decomposition), the AFM tip will move material along the scanning directions and pull apart the images quality. For sure cartridges with such structure could not be used for patient treatments, since completely lost ability of filtration.

In order to evaluate changes in the membrane surface in a quantitative manner, we measure the surface roughness for each sample after each use and for external and inner part of fiber, separately. Results are presented at Fig.4.a-d., for external and Fig.5.a-d., for inner part of the membrane surface, respectively.

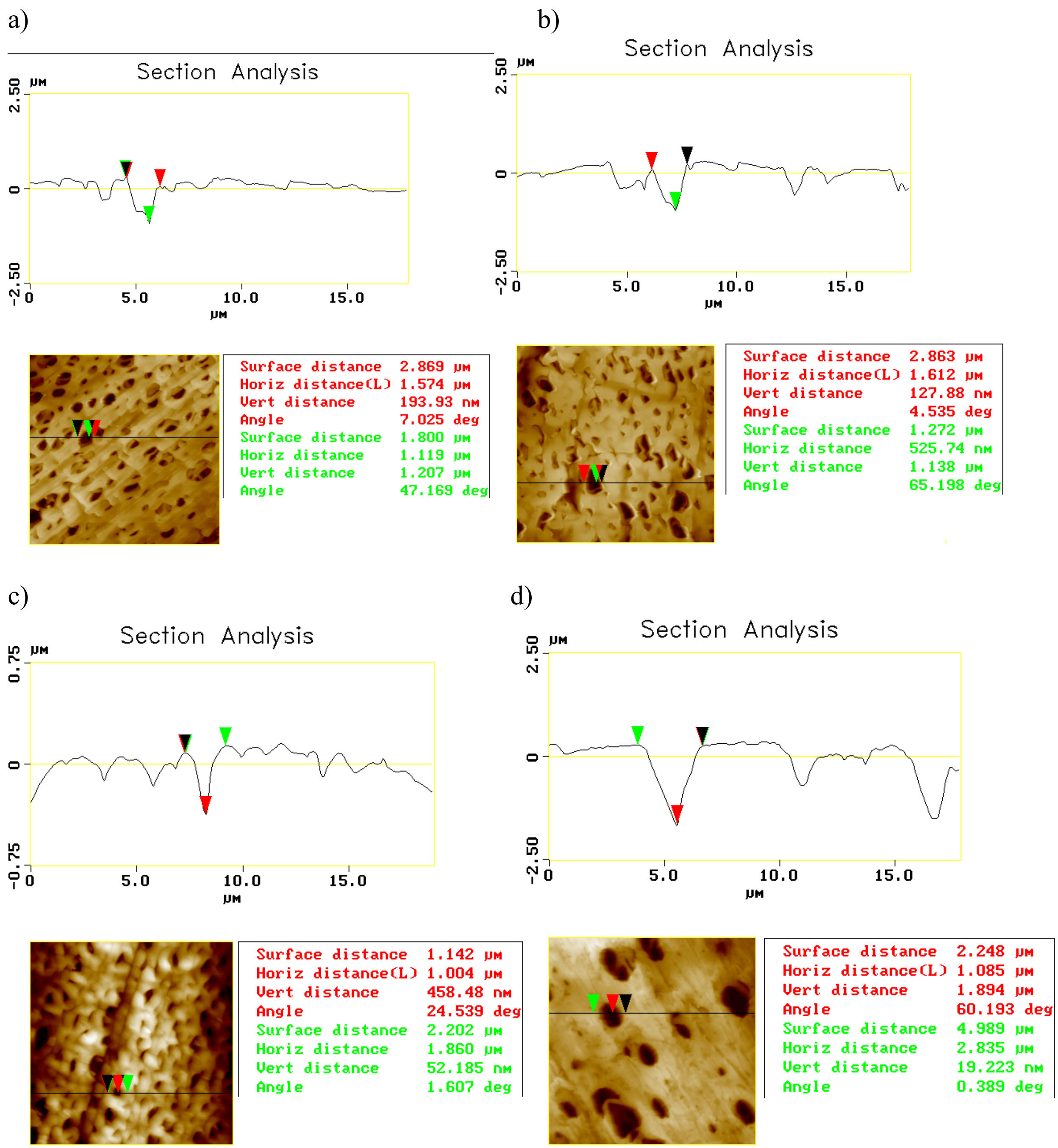


Fig.4. Cross section analysis of AFM images of the external (outer) membrane surface: (a) zero uses (control); (b) single trail; (c) after 10 time of use; (d) after 23 times of use.

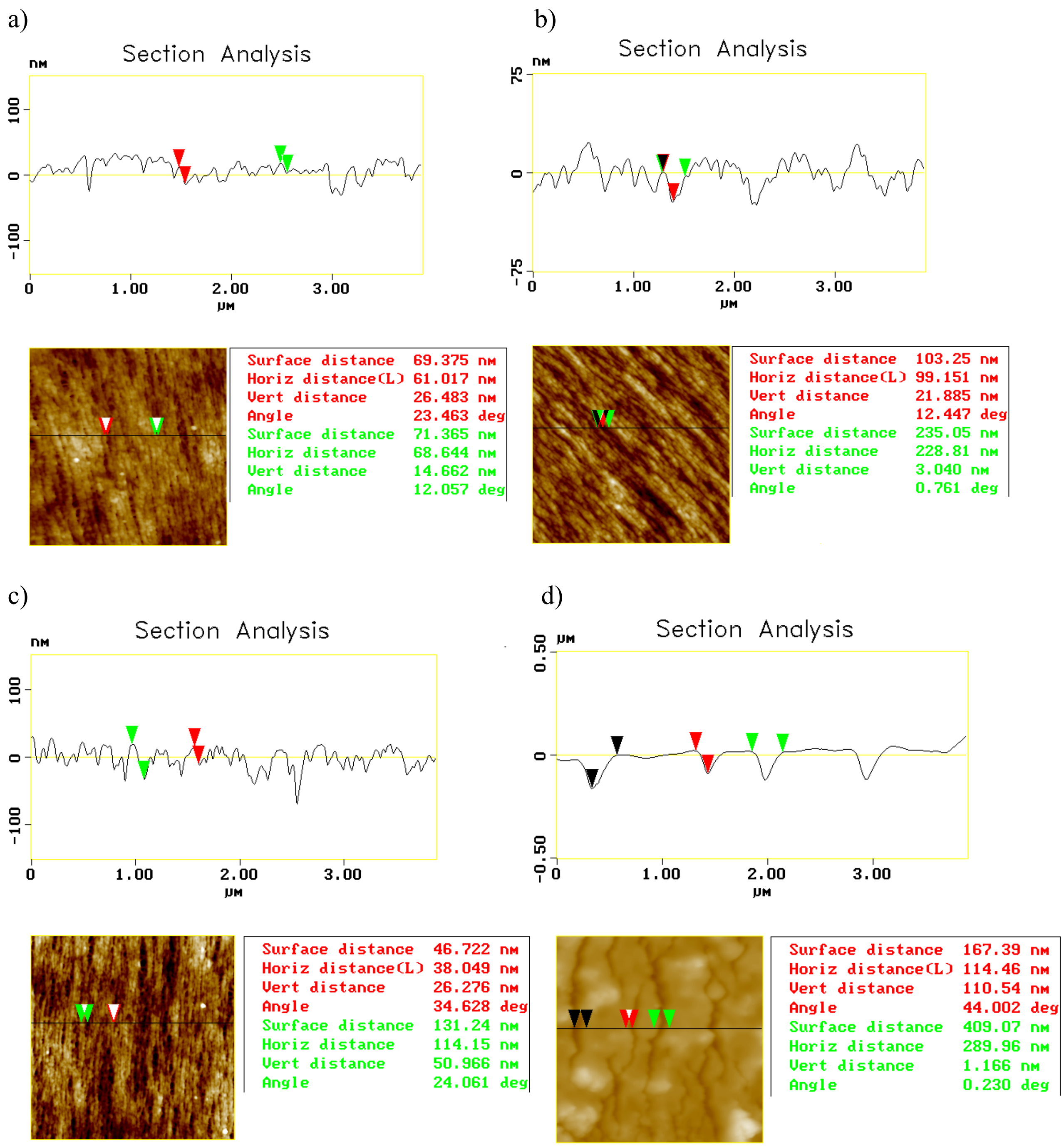
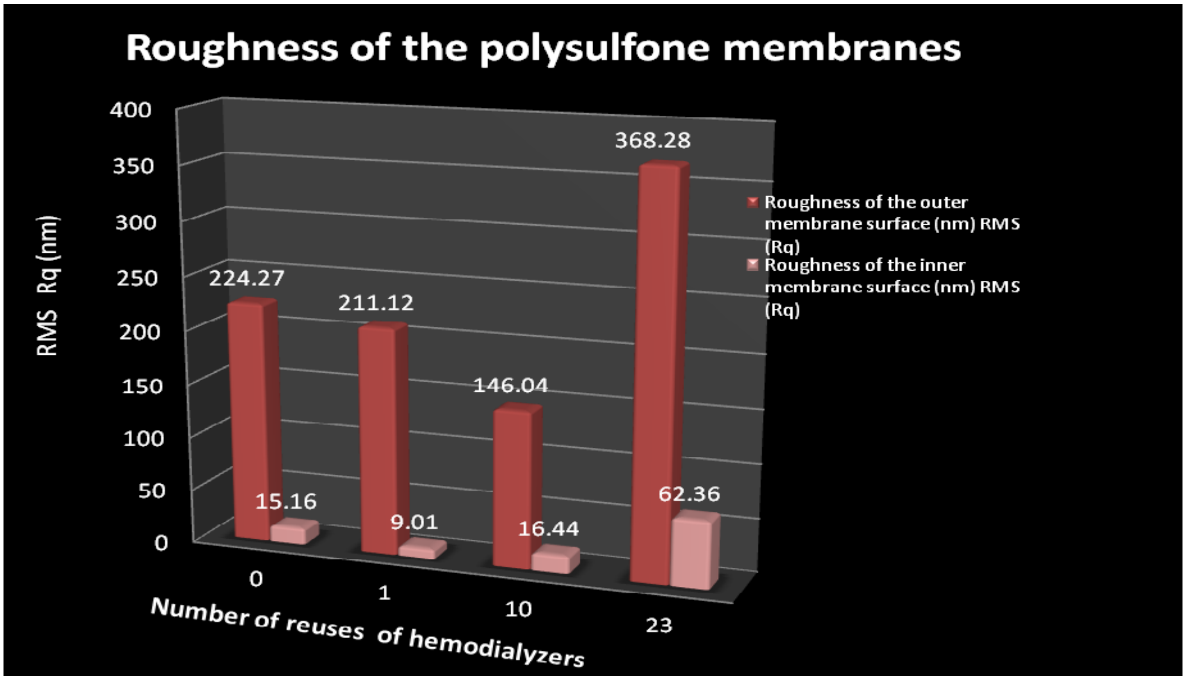


Fig.5. Cross section analysis of AFM images of the inner membrane surfaces, image size 3.9 µm x 3.9 µm.: (a) zero uses (control); (b) single trail; (c) after 10 times of use; (d) after 23 times of use.

The surface roughness changes were monitored and presented with help of the $RMS_{[Rq]}$ (root-mean-square) parameter, which is often used as a standard in such type of calculations. It measures height of each feature in the AFM images and calculate surface roughness with high precision.



The obtained results are presented in diagram at Fig.6. Note that all AFM images selected for $RMS_{[Rq]}$ evaluation were the same size, for external and inner membrane surface, respectively. Results are very interesting, since in both cases the 23 use of the same membrane leads to significant increment in the surface roughness, which clearly means, the membrane is wear out in the reuse process. Also it seems that inner (nanostructured) part of membrane is less affected during the consecutive use, which also could be clearly seen in the above presented AFM images.

4. CONCLUSION

AFM images reveal changes in the surface morphology of the dialyzer membrane during the consecutive use of the hemodialysis cartridges. Due to the high resolution ability, the AFM analysis shows changes in the structural features at the nanosize level. Beside qualitative analysis, due to use of $RMS_{[Rq]}$ parameter, the changes in the membrane surface roughness were presented at the quantitative level, too. Imaging and the surface roughness evaluation were performed at external (outer) and inner part of the hemodyalisis membrane, systematically for cartridges of different consecutive use: 0,1,10 and 23. Results are interesting clearly showing that inner and outer part differently resist to wearing process, which should be taking into account for design of new and better materials and artificial systems for the future hemodialitic processes.

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ACKNOWLEDGMENTS

This study was supported by Project of "Ciencia y Tecnología para la Capital del Conocimiento", ICyTDF: "Desarrollo de técnicas nanotecnológicas para evaluar la calidad del tratamiento médico hemodialitico" (12411497), and DCBS/DCBI-UAM, The ICyTDF-UAM Nanotechnology Project. Acknowledgements to SEP-CONACYT project (CB-61242) : "Nanotecnología para medicina y biología: estudio de caracterización de células por AFM y STM", "Técnica nanotecnológica y metabólica para valorar la calidad de los tratamientos médicos hemodialíticos" of CBI, UAM-I and UAM multidisciplinary investigation project 13/2007. G.M.C Acosta and P.A. Avalos are recipients of CONACYT scholarship for Graduate programs at UAM-I.