# Characterization of Nanofiltration Membranes

**Michael Donnell Chestnut II, Computer & Electrical Engineering, North Carolina State University**
***NNIN REU Site: Howard Nanoscale Science & Engineering Facility, Howard University***

*NNIN REU Principal Investigator: Dr. Kimberly Jones, Department of Civil Engineering, Howard University*
*NNIN REU Mentor: Dr. Jermey Matthews, Department of Civil Engineering, Howard University*
*Contact: mdchestn@ncsu.edu, kjones@scs.howard.edu*

**Abstract:**

The goal of this research was to characterize the surface properties of nanofiltration membranes. The processes that are used to characterize these membranes are standard but the membranes are synthesized in the laboratory and the applications for these membranes are novel. Several properties will be investigated in detail. These include membrane charge, the surface roughness, and the membrane pore size. The membrane flux and rejection levels are also examined, but are not reported in this paper.

The particular focus of this paper is surface charge and roughness of the membrane as well as possible pore imaging and identification. The equipment employed to study the charge of the membrane included an Electro Kinetic Analyzer, used to measure zeta potential (proportional to surface charge) and an Atomic Force Microscope (AFM) with a liquid cell, used to image the morphology of the membrane. This paper also provides background information on the applications and systems which use these membranes. There is good agreement between theoretical and experimental results for such things as the zeta potential.

## Introduction:

Howard University, the Nanobiotechnology Center at Cornell University, and the Wadsworth Center in Albany, NY, were assigned the task of speeding up the process of separating DNA from hemoglobin coming from blood extracted from newborn babies. This DNA is to be analyzed for any birth defects so that they can be altered before any serious illnesses develop. Much of the performance of the separation process is related to the specific properties of the membranes that will be characterized.

There are several different classifications for membranes. The more commonly used membranes are for microfiltration, ultrafiltration, nanofiltration, and reverse osmosis. Microfiltration has the largest size pores and reverse osmosis contains the smallest sizes pores. As the pores decrease so does the range of solutes able to pass through the membranes [1]. Pressure is typically used to drive the transport through the membranes and it increases as the pores become smaller. Membranes used in this process are either inorganic or polymeric.

Nanofiltration is a process in which membranes with nano-size pores are used to separate solutes or salts based on size and/or charge [2]. This mechanism underscores the importance of the zeta potential, which is directly proportional in sign and magnitude to the surface charge density [3], and the surface morphology, which can give the surface properties and porosity, and is most accurately observed by AFM imaging (Figure 1). The surface charge is responsible for the rejection of ions of similar charge and the surface roughness

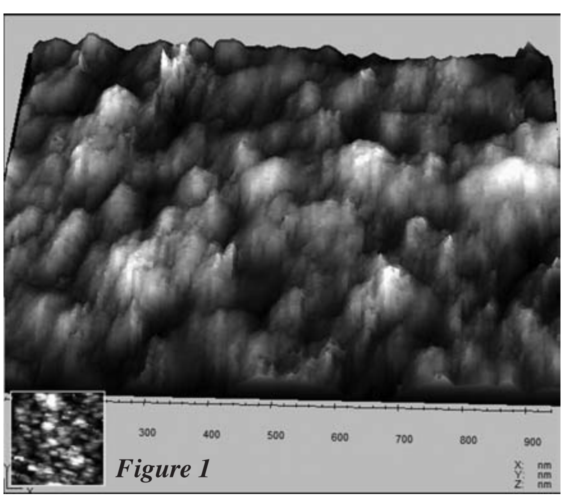

*Figure 1*

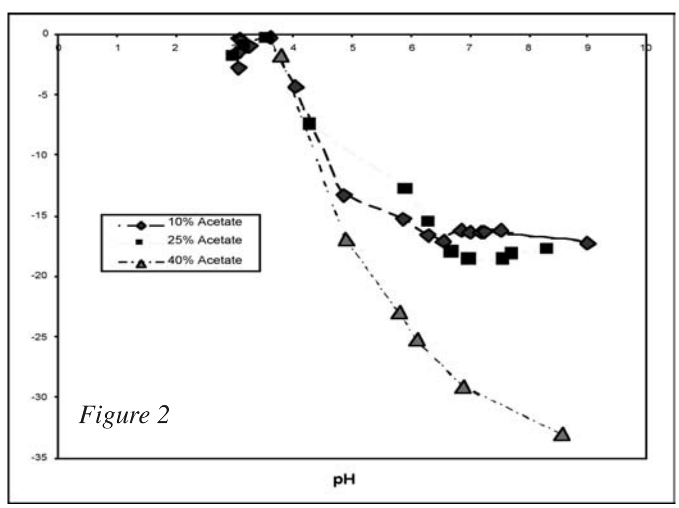

*Figure 2*

has been shown to improve solute flux while also increasing the rate of membrane fouling (Hirose, 1997).

The main use for the membranes is the removal of bacteria, viruses, salts, and other organic and inorganic material from water. This process is found at wastewater plants. The processed water can not only be used for city purposes but also beverage products such as soft drinks and coffee. Other applications can be found within the medical field where membranes are attached to silicon wafers as a means of separating biomolecules [4].

## Procedures:

Samples were imaged by AFM in a PicoPlus fluid cell (Molecular Imaging, Inc.) under Magnetic AC (MAC) mode in 1 mM KCl electrolyte solution. The cantilever was the MAClever Type IV (Molecular Imaging, Inc.) made of silicon nitride with a resonance frequency of 15 kHz and a force constant of 0.02 N/m.

The zeta potential was measured by streaming potential with the EKA Analyzer (Brookhaven Labs) over a pH range of 3-9 using 1 mM KCl electrolyte and HCl and KOH as pH adjusters.

Figure 1 is an AFM of the 10% acetate concentration membrane.

## Results:

**EKA: Effect of pH and Acetate Concentration on Zeta Potential/Charge Density;** In Figure 2, the y axis represents the change in zeta potential which is the uncontrolled variable and the x axis represents the pH, the controlled variable. The zeta potential (and hence the surface charge) was negative throughout the entire pH range, and as the percentage of the acetate concentration within the membrane increased so did the zeta potential which should hold true in theory (Elimelech, 1999). This was observed for the 40% acetate membrane. But there seems to be no significant difference in zeta potential between the 10% acetate and 25% acetate membranes.

**AFM: A Study of the Surface Topography;** The data on the surface roughness, shown in Figures 3 and 4, did not support the assumption that roughness has a positive dependence on the acetate concentration. The roughness therefore depends more on other parameters such as how the membranes were made during the spin process, along with reactants in the process which were all held constant. The peak to valley ratio does however seem to depend on the concentration of acetate in the membrane—the greater the concentration the less the height difference from the peaks to the valley. This relationship is assumed to be based on the reactants being more aggregated with the increase of acetate.

## Conclusions:

In conclusion the general assumption and theory of the acetate playing a major role in the surface roughness and on the zeta potential held true only for a small portion of the experiment. The roughness of the membrane did not appear to vary much with a change in acetate concentration. The zeta potential had a larger negative charge for the 40% acetate membrane than 10% or 25% as expected but it is hard to differentiate any effect on the zeta potential as a result of acetate concentration between the 25% and 10% membranes.

## Acknowledgements:

The author wishes to thank Dr. Kimberly Jones, Dr. Jermey Matthews, HNF, NNIN and NSF.

## References:


[1] Membrane Technology and Applications; 2 ed.; Baker, R. W., Ed.; John Wiley & Sons, Ltd.: Chichester, 2004.

[2] Nanofiltration: Principles and Applications; A.I. Schafer, A. G. F., T.D. Waite, Ed.; Elsevier, Ltd.: Oxford, 2005.

[3] Elimelech, M.; Xiaohua Zhu; Childress, A. E.; Seungkwan Hong. Journal of Membrane Science 1997, 127, 101.

[4] Russo, A. P.; Retterer, S. T.; Spence, A. J.; Issacson, M. S.; Lepak, L. A.; Spencer, M. G.; D.L. Martin; MacColl, R.; Turner, J. N. Separation Science and Technology 2004, 39, 2515.


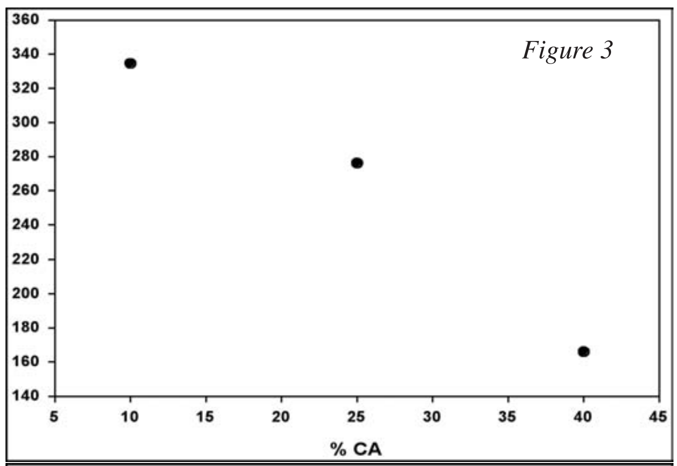


*Figure 3*

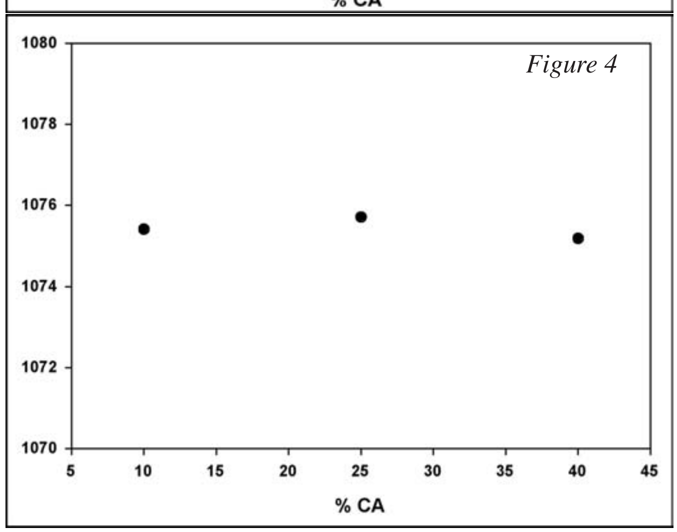


*Figure 4*