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Applications of Low Voltage Field Emission Scanning Electron Microscopy (FE-SEM) for Characterization of Polyethersulfone/ Polyvinylpyrillidone (PES/PVP) Based Materials for Membrane Separations

Pooja Bajaj, Albin Berzinis, Rachel Giessert, and Carl Strom

Sabic, 1 Noryl Avenue, Selkirk, New York 12158 USA

Polymer blending offer an interesting route to modify the material properties with the goal to combine the unique key attributes that the individual constituents have to offer for improved performance. Polyethersulfone/polyvinylpyrrolidone (PES/PVP) presents unique properties when combined owing to their mechanical and chemical stabilities for preparing ultrafiltration membranes with a low molecular weight cut-off for biotechnology applications [1]. The morphology and structure often assumes a key role in governing the polymer properties. These membranes were cast using PES (BASF E6020P) at 14 weight% and polyvinylpyrrolidone (PVP) K90 at 2 weight %, PVP K30 at 5 weight %, using phase inversion process from n-methylpyrrolidone (NMP) into water [1]. The blending of low and high K-value PVP is shown to be important to controlling the surface pore structure and tendency towards macro void formation [1]. For a functional membrane, surface provides insight into the roughness and selective porous structure, while the cross sectional morphology reveal information about the mechanical stability of the membrane. In this work, low voltage field emission scanning electron microscopy characterization using Carl Zeiss Supra 40 VP FE-SEM of PES/PVP based flat membranes will be discussed.

At low voltage operation in FE-SEM (acceleration voltage ≤ 5keV), the beam interaction with the specimen is confined to regions very close to the surface, improving the lateral spatial resolution, and when combined with in-lens detection provides surface sensitive details. These developments in FE-SEM are attributed to incorporation of a Schottky field emission source allowing for high electron optic brightness, low beam noise, and near atomic resolution [2-4]. The Kanaya and Okayama expression* and the Monte Carlo electron trajectory simulations illustrate the direct dependence of interaction volume and the depth of electron penetration on the beam acceleration voltage [5]. Several parameters were optimized including the acceleration voltage, aperture size, detection mode and inert metal coating thickness for high resolution surface details.

$$*R\ (\mu m) = \frac{0.0276\, A\, E^{1.67}}{Z^{0.89}\, \rho}$$

R– Depth Penetration
A– Atomic Weight (g/mole)
E– Beam Energy (keV)
Z= Atomic number
ρ = density (g/cm)^3

FE-SEM characterization revealed the asymmetric PES/PVP based membranes with nanoporous surface layer (Figure 1). Statistical analysis of pore size distribution and the effect of acceleration voltage on surface porosity analysis will be discussed. The cross section (Figure 2) provides information about the mechanical stability of the membrane. Figure 2 shows the cross sectional morphology with three distinct regions, the dense nano-porous skin layer, nano-to-micro porous sub layer with lower layer showing micro and macro-voids. In summary, using low voltage FE-SEM, high resolution structural characterization for surface porosity and cross sectional morphology was developed. Pore size analysis

and cross sectional morphology characterization provide valuable insight for probing the effect of factors including the composition, molecular weight, miscibility between the different components, viscosity, and additives among others. This structural characterization can be gainful in understanding the mechanical performance and tailoring of new materials.

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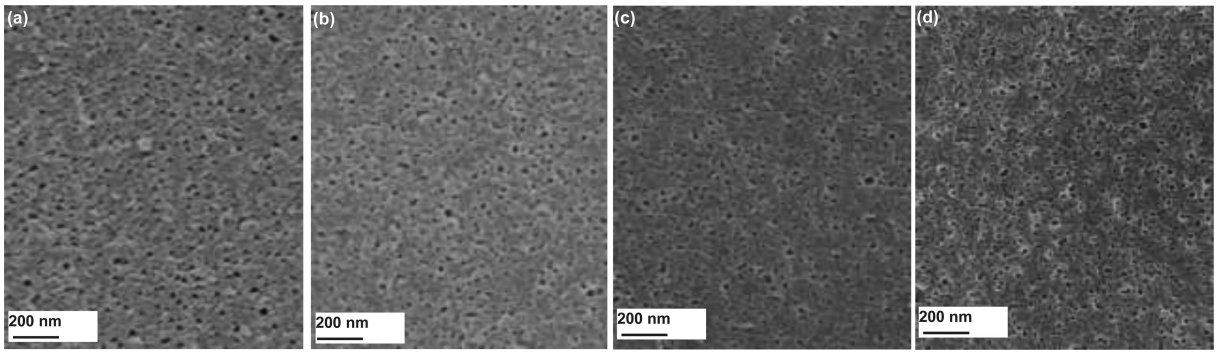

Figure 1. Effect of increasing beam energy (a) 0.5 keV, (b) 1 keV, (C) 5 keV (d) 20 keV on surface pore structure characterization of PES/PVP based flat membranes. At low voltage (≤ 5 keV), true surface pore structure is revealed. At 20 keV, apparent beam damage to the surface features is demonstrated.

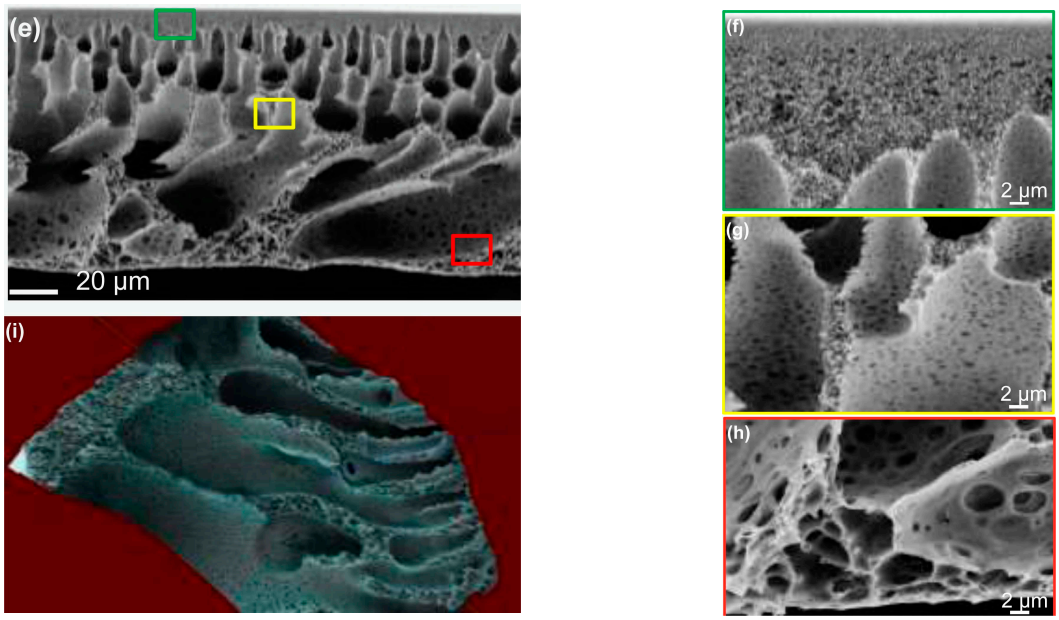

Figure 2. Cross-sectional morphology of PES/PVP based membranes (e). The membranes were soaked in ethanol for 5 minutes and cryo-fractured using liquid nitrogen. (f), (g), (h) zoom in on the three areas of the cross section showing the dense skin layer, porous sub layer and macro-voids (i) 3-D representation of the membrane in cross section.

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