



CHAPTER III EXPERIMENTAL

3.1 Materials

The surfactant used in this study, Triton X-100[®], a nonionic surfactant with 100% active ingredient, was purchased from Union Carbide Chemicals and Plastics Company Inc. (Danbury, CT). Tetraethyl Orthosilicate (TEOS) with a purity of >99% was purchased from Fluka (Milwaukee, WI). Styrene, 99% purity, was purchased from Sigma Chemical (St. Louis, MO). 2,2'-azobisisobutyronitrile (99% purity) was obtained from Aldrich (Milwaukee, WI). Methanol, HPLC grade, was purchased from Fisher Scientific (Fair Lawn, NJ). All materials were used as received. Muscovite mica discs, 9.9-mm diameter, were purchased from Ted Pella, Inc. (Redding, CA) and cleaved on both sides at ambient condition and quickly immersed in the solution. Water was deionized through Barnstead E-pure water system consisting of charcoal filters, ion-exchange media, and filter. The resulting de-ionized water had a resistivity of 18.3 M Ω .cm⁻¹.

3.2 Synthesis of the Formed Polystyrene/Silica Composite Film on Mica

3.2.1 Synthesis of the Formed Polystyrene/Silica Composite Film in the Presence of Surfactant on Mica

The concentration of surfactant (Triton X-100[®]) carried out in this study was 0.2 mM which is below the CMC of surfactant. The surfactant was adsorbed on the surface of the freshly cleaved mica by immersing the mica in the surfactant solution for 24 hours before use. The formed polystyrene/silica composite film on mica was prepared through the introduction of styrene monomer in the presence of surfactant and subsequently the addition of AIBN into the solution. Amount of AIBN used were in the ratio of 1 mole of AIBN to 6 mole of styrene. Immediately after preparation, freshly cleaved mica which prior to the adsorption of surfactant was immersed in the solution. Following 2 days, TEOS was mixed with the solution and allowed to stand for another 2 days. Polymerization was conducted

at 80°C in a water bath for 2 hours. After polymerization, the as-synthesized polystyrene/silica composite film on mica was washed with methanol and de-ionized water and was left to dry in a dessicator for at least 1 day under ambient condition.

3.2.2 Synthesis of the Formed Polystyrene/Silica Composite Film in the Absence of Surfactant on Mica

In the case of without surfactant, mica was immersed in de-ionized water instead of surfactant for 24 hours before use. The formed polystyrene/silica composite film on mica was prepared through the introduction of styrene monomer and AIBN in de-ionized water. Amount of AIBN used was in the ratio of 1 mole of AIBN to 6 mole of styrene. Following 2 days, TEOS was mixed with the solution and allowed to stand for another 2 days. Polymerization was conducted at 80°C in a water bath for 2 hours. After polymerization, the as-synthesized formed polystyrene/silica composite on mica was washed with methanol and de-ionized water and was left to dry in a dessicator for at least 1 day under ambient condition.

3.3 Modified Surface Characterization by Atomic Force Microscopy

The morphology of the modified surfaces was imaged with a Nanoscope IIIa, Multimode Scanning Probe Microscope from Digital Instrument, Inc. (Santa Barbara, CA) equipped with AS-130V (“J” vertical) scanner with the maximum lateral and vertical scan ranges of 125µm x 125µm and 5.0µm, respectively. AFM images were obtained under ambient conditions while operating the instrument in tapping mode. Tapping Mode Etched Silicon Probe (TESP), purchased from DI (Santa Barbara, CA), were used, which posses spring constant of 20-100 N/m, a nominal tip radius of curvature of 5-10 nm, and a resonance frequency of 200-400 KHz. The mica on which the adsorbed layer of polystyrene/silica composite material was fabricated was adhered to a metal disc for measurement. The images were captured simultaneously in height and phase modes with a resolution of 256x256 pixel at scan speed at 1 Hz on all images. and typical resonant frequency around 300 kHz. were used to obtained the image. The obtained images were flattened and analyzed using the NanoScope IIIa software.