

CHAPTER III

EXPERIMENTAL

3.1 Materials

Octylphenoxyethoxyethanol (Triton X-100®), 100% nonionic surfactant, was obtained from Union Carbide Chemicals and Plastic Company INC. (Danbury, CT). Tetrabutyl orthotitanate (TBOT) (97% purity) was purchased from Fluka (Milwaukee, WI). Tetraisopropyl orthotitanate (TIPP) (99.999% purity) was purchased from Fluka (Milwaukee, WI). HPLC grade isopropanol, 99.9% purity, was purchased from Fisher Scientific (Fair Lawn, NJ). Mica discs with a 9.9 mm diameter were obtained from Ted Pella (Redding, CA). De-ionized water was obtained from a Branstead E-pure water system with a resistivity of 18.3 mega ohm cm⁻¹.

All materials were used as received.

3.2 Surface Modification Procedure

Mica discs were cleaved in air before use and then immersed in a 0.2 mM Triton X-100® aqueous solution for 24 hours to allow the admicelle to form on the substrate. The mica disc was then transferred to 10 cm diameter petridish containing 18 mL of 0.2 mM Triton X-100 ® solution. Then 2 mL of the precursor solution diluted in isopropanol was injected into the petridish. Concentration of each component in the solution is shown in Table 3.1. The systems were allowed to sit for the desired reaction time (10 minutes, 6 hours and 24 hours). The mica disc was then removed from the solution and washed with isopropanol 3 times for 5 minutes each time. Finally, the modified mica disc was dried in a desiccator at room temperature for 24 hours before imaging by tapping mode AFM and EDS.

Table 3.1 Concentration of solutes in solution

Experiment set A1

Sample	TritonX-100 (mM)	TBOT (μ M)	2-propanol (%vol/vol)
A1-1	0.2	0.01	10
A1-2	0.2	10	10
A1-3	0.2	500	10

Experiment set A2

Sample	TritonX-100 (mM)	TBOT (μ M)	2-propanol (%vol/vol)
A2-1	-	0.01	10
A2-2	-	10	10
A2-3	-	500	10

Experiment set B1

Sample	TritonX-100 (mM)	TIPP (μ M)	2-propanol (%vol/vol)
B1-1	0.2	0.01	10
B1-2	0.2	10	10
B1-3	0.2	500	10

Experiment set B2

Sample	TritonX-100 (mM)	TIPP (μ M)	2-propanol (%vol/vol)
B2-1	-	0.01	10
B2-2	-	10	10
B2-3	-	500	10

3.3 Equipment

3.3.1 Atomic Force Microscope (AFM)

The atomic force microscope, a multimode Nanoscope III a AFM, from Digital Instruments, Inc. (Santa Babara, CA) and a J scanner was used.

Tapping Mode AFM was used to capture topographic and amplitude images of dried samples under ambient conditions. Tapping Mode Etched Silicon Probes (TESP) from Digital Instruments (Santa Babara, CA) were used. TESP has a resonant frequency of 200-400 kHz. All tapping mode images were captured at room temperature and in air with a relative humidity less than 30%. The relative humidity was monitored by a humidity probe from Cole-Parmer Instrument Company (Vernon Hills, IL)

3.3.2 Energy Dispersive X-Ray Spectroscopy (EDS)

The sample was placed on a copper stub and coated with gold at 10 mA for 7 minutes. The sample was then analysed by energy dispersive x-ray spectroscopy (Oxford), ISIS 300 model, UK.