

# Chapter 2

## Experimental Section



## 2.1 Experimental Section

### 2.1.1 Materials

All the reagents used in this study, including graphite plate, SDS, APTES, hydrochloric acid and PVA (with  $M_w \approx 72,000$ ) were provided by Merck and Sigma Aldrich Companies. Deionized water was also used for the preparation of solutions and washing.

### 2.1.2 Synthesize of the Graphene Sheets

All experiments were conducted in a two-electrode electrochemical cell with working volume of 50mL at 25 °C. A platinum plate and a pure graphite electrode (polished to a mirror finish, washed with acetone and distilled water) were used as the cathode and anode, respectively. The geometric area of the anode and cathode were  $1.5 \times 1 \text{ cm}^2$  and  $1.5 \times 2 \text{ cm}^2$ , respectively. The aqueous electrolyte consisted of 0.1 M SDS with a pH of 8.4 (The reason why we used this concentration is related to the surface tension of liquid, refer to the appendix A). The advantage of SDS, compared to other intercalator such as fluoride [88], nitrate [89], sulfate [90] and lithium [91], is its effective inhibitory role in prevention of re-stacking and agglomeration of the produced layers. However, identification of the role of applied potential in electrochemical intercalation/exfoliation of graphite is a prerequisite for the effective synthesis of graphene sheets [100]. This is especially true when this method have to compete with different chemical/physical based synthesis routes.

The intercalation and exfoliation steps of the graphite plate were performed by applying different potentials of 5, 10, and 15 V for 10 hours. Then, the

obtained stable graphene/SDS suspensions were centrifuged at low speed (5000 rpm) for 20 min. The final samples were exposed to high frequency ultrasonic waves for improving the chemical bonds between graphite layers and SDS; furthermore, the shocks applied by the waves lead to a complete exfoliation of graphite surface [92]. The obtained suspensions were collected with a 200 nm porous filter, washed by DI water and ethanol and then the powders were dried in vacuum oven for 5 hours.

In order to determine SWGSs content of the samples and process efficiency, 20mL of 0.1 M  $\text{Ca}(\text{NO}_3)_2$  solution was added to 20mL graphene/SDS suspension which results in  $\text{Ca}(\text{DS})_2$  and SWGSs precipitation. By weighting the filtered samples, SWGSs content and efficiency were estimated [79].

### **2.1.3 Preparation of SWGSs/PVA Composites**

The SWGSs/PVA composites were synthesized in a simple liquid phase blending method. Since, the SWGSs are stable in water and PVA is soluble in water too, this method will result a homogenous mixture simply. A composite film of SWGSs and PVA in different percentage of SWGSs (0.06, 0.1, 0.2, and 0.3 Vol%) was made by drop casting route on a plexiglass substrate and keeping in the vacuum oven at 45 and 60 °C for 24 hours, separately. Consequently, after drying completely, the films were peeled off from the substrate.

### **2.1.4 Methods of Characterization**

In order to preparation of the samples for HRTEM, TEM (JEOL, JEM-2100, operating at 200 kV), FESEM (Mira\\Tescan) and AFM (in tapping mode, Dualscope/Rasterscope C26, DME, Denmark) analyses, APTES was mixed with water and one drop of hydrochloric acid was added; then, Si wafers were entered

into the solution for 30 min and then washed thoroughly with DI water. Afterward, the substrate was dipped in SWGSs solution for 10s and subsequently introduced to the DI water and dried [93]. During the synthesis process, the CTT curves were recorded at different applied potentials. Additionally, in order to study the functionalization of the SWGSs, FT-IR spectra of the graphene dispersion were collected. XRD of the graphene sheets were carried out with Equinox 3000/INEL, France diffractometer with Cu  $k_\alpha$  radiation ( $\lambda = 1.541874\text{\AA}$ ). In order to study the density of defect and number of SWGSs layers, Raman spectra were recorded with Raman Microprobe Nicolet ALMEGA with 532 nm excitation source under 100mW power.

To investigate the effect of process temperature on the polymer films, TGA was performed by using of TGA 50 Shimadzu Tokyo Japan with heating rate of 10 °C/min in N<sub>2</sub> atmosphere. Furthermore, DSC was done by Perkin Elmer in a non-isothermal 10 °C/min cooling ramp. The mechanical properties of the nanocomposite sheets with a width of 10 mm were studied with a 5566 U.S.A Instron at a strain rate of 5 mm/min in ambient situation; in addition, the gauge length in all experiments was 20 mm.

