

## **Experimental part 1 (E1):**

### ***Preparation of citric acid-treated melamine:***

35 g melamine and 1 g citric acid were mixed in 150 mL absolute ethanol, stirred at room temperature for 20 min, sonicated in an ice-bath for 15 min, and finally dried at 60 °C. The citric acid-treated melamine was mixed with the composited precursor by grinding and calcination.

## **Experimental part 1 (E2):**

### ***Analysis of Mo content in the products by UV-vis spectroscopy:***

#### 1) Solutions

- a) Solution A: 250 mL concentrated H<sub>2</sub>SO<sub>4</sub> diluted with 250 mL of water
- b) Solution B: 250 mg CuSO<sub>4</sub> dissolved in 450 mL (1+1) H<sub>2</sub>SO<sub>4</sub> and filled to 500 mL
- c) 20% KSCN solution: 62.5 g KSCN dissolved in water and filled to 250 mL
- d) 70% thiourea solution: 12.5 g thiourea dissolved in water and filled to 250 mL

#### 2) Preparation of standard solution of Mo:

5.54 mg of molybdic acid was dissolved in 5 mL of 6 M NaOH, which was diluted to 250 mL of water. Then, 1 mL, 2 mL, 3 mL, 4 mL, and 5 mL of the diluted solution was transferred into 50 mL culture tubes. One drop each of phenolphthalein indicator and solution A was added, yielding colorless solutions. The solutions were diluted to 25 mL, and 10 mL of solution B was added. After the solutions cooled down to room temperature, 5 mL of KSCN solution and 5 mL of thiourea solution were added, and the resulting solutions were diluted with H<sub>2</sub>O to a final volume of 50 mL.

#### 3) Preparation of the sample solution

5 mg of the samples were dissolved in 5 mL of concentrated nitric acid at 200 °C and filled to a volume of 250 mL by adding water. 10 mL of this solution was then transferred to a 50 mL culture tube, diluted with 15 mL of H<sub>2</sub>O, and then combined with 10 mL of solution B. The resulting solution was cooled to room temperature, and 5 mL of KSCN and 5 mL of thiourea solution were added. The mixture was then diluted with H<sub>2</sub>O to a final volume of 50 mL. One solution without KSCN was used as a control sample. UV-vis spectra were recorded at 460 nm in 30 min after preparing the testing solutions.

### ***Calculation of ESCA***

Based on the linear fitting of the current density at 0.25 V vs RHE with the increase in the scan rates for the cyclic voltammetry of different samples, the specific capacitance can be calculated as follows:  $C = (k/2) \cdot (1/m)$ , where C is the specific capacitance of the measured samples, k is the fitting slope, m is the catalyst real loading of 0.28 mgcm<sup>-2</sup>. Then, the ECSA can be calculated by assuming a standard value of 60 μF/cm<sup>2</sup>, namely,  $ECSA = C/60 \mu\text{F}/\text{cm}^2$ .