

CHAPTER 3

EXPERIMENTAL

3.1 Materials, chemicals, apparatus and instruments

3.1.1 Materials and chemicals

- 1) Bottom ash (BA), Mae Moh Power Plant, Lampang, Thailand
- 2) Flue gas desulfurization (FGD) gypsum, Mae Moh Power Plant, Lampang, Thailand
- 3) Paddy clay (PC), Paradornbricks Co., Ltd., Thailand
- 4) Sawdust (SD), Thaweephan Wood Flooring Co., Ltd., Thailand
- 5) Methyl orange (MO), $C_{14}H_{14}N_3Na_3S$, AR, Merck, Germany
- 6) Iodine, I_2 , AR, Ajex, Australia
- 7) Postassium Iodide, KI, AR, Ajex, Australia

3.1.2 Apparatus and instruments

- 1) Autosorb[®]-1-MP analyzer micropore measurements, Quantachrome Instruments, USA
- 2) Mastersizer particle size analyzers, Melvern Instrument Ltd, UK
- 3) Hot air oven, model UNE 400, Memmert company, Germany
- 4) Digital precise shaking water bath, model WSB-45, DAIHAN, Korea.
- 5) UV-Vis spectrophotometer, model 6400, Jenway, England

- 6) X-ray fluorescence spectrometer, model X'Pert Pro MPD, Panalytica, Netherland

3.2 Preparation of raw materials and solutions

3.2.1 Preparation of raw materials

Each material (BA, FGD gypsum, C and SD) was dried sequentially at 850 °C for 7 hours. Then the materials were crushed and sieved to different sizes (BA 100 mesh, FGD gypsum 100 mesh, PS 80 mesh and SD 80 mesh) by hammer mill and sieving machine, respectively.

3.2.2 Preparation of solutions

1) Stock standard 1000 ppm methyl orange solution

1.0000 g of MO was dissolved in water in 1-L volumetric flask and then diluted to 1000 cm³ with deionized water.

2) 0.10 M KI

1000 cm³ of 0.10 M KI was prepared by weighing 16.6 g of KI, dissolving and making up the volume to 1000 cm³ in 1-L volumetric flask with deionized water.

3) 0.01000 M I₂

1000 cm³ of 0.01000 M I₂ was prepared by weighing 2.538 g of I₂, dissolving and making up the volume to 1000 cm³ in 1-L volumetric flask with 0.10 M KI.

3.3 Production of adsorbent material

The three raw materials (BA, FGD gypsum and PC) were mixed with sawdust at a ratio of 22:7:45:26 percent by weight. From each mixture, wet powder was adding about distilled water to 10%. The wet powders were compressed into metallic mould to form granules. These granules were fired in a cubic meter furnace, using liquid petroleum gas (LPG) as a fuel, at 850 °C with heating rate of about 3 °C/minute. The fired granules were then ground to powder and passed through a 100 mesh screen for determinations of particle and surface area, and for adsorption studies.

3.4 Study of physical and chemical properties of adsorbent material (AM)

3.4.1 Particle size and surface area determinations

Distribution of particle size (μm) and specific surface area (m^2/g) of fired adsorbent powders were measured by particle size analyzers by Quantachrome Autosorb automated analyser with nitrogen gas (version 2.46), respectively.

3.4.2 Chemical composition determination

The chemical compositions of fired adsorbent powders were measured by X-ray fluorescence spectrometry (XRF). Material compositions were analyzed on a Horiba/MESA-500 W X-ray spectrometer using Rhodium (Rh) tube at Mae Fah Luang University, Chiangrai Province.

3.4.3 Slake investigation

The produced AM samples were soaked in a beaker with 25 cm³ water covering the material. After 30 days, the shape of the AM samples was investigated visually.

3.4.4 Water absorption determination

- 1) The weighed dried adsorbent was put into a 50 mL beaker and water was added.
- 2) The adsorbent was boiled on a hot plate for 2 hours and left to cool down at room temperature for 24 hours.
- 3) The adsorbent was weighed to determine the amount of water absorbed.
- 4) The water absorption was calculated according to:

$$\text{Water absorption (WA)} = \frac{m_2 - m_1}{m_1} \times 100 (\%)$$

where m_1 is the dry mass (g) and m_2 soaked mass (g).

3.4.5 Density determination

- 1) The dried AM was weighed.
- 2) 25 cm³ of water was added into a cylinder and the weighed dried adsorbent material was added into cylinder for volume measurement.
- 3) The density was determined as the mass (weight) per unit volume of the solid material.

3.4.6 pH determination

The pH value was measured by a pH meter using the ratio of material to distilled water 1:3 by weight.

- 1) 10 g of the sample was put into a beaker.
- 2) 30 cm³ of deionized water was added into the beaker.
- 3) The mixture in the beaker was stirred with a glass rod for 10 minutes.
- 4) The material was allowed to stand for at least 15 minutes.
- 5) The pH value was measured by pH meter.

3.5 Adsorption study of methyl orange and iodine on the adsorbent material

3.5.1 The effect of the contact time between adsorbent material and solutions

1) 0.5000 g of AM powder was put into flasks and 100 cm³ of methyl orange solution at concentrations of 2.00 and 10.00 ppm or iodine solutions at concentrations of 1×10^{-3} and 1×10^{-5} were added.

2) Each flask was closed with parafilm and shaken at 120 rpm for different time from 10 to 140 minutes at 30 °C.

3) Each solution at various concentrations and at different time intervals (10–140 minutes) was passed through 0.2 µm nylon filter and measured at the maximum wavelength.

3.5.2 Adsorption study of methyl orange on the adsorbent material

3.5.2.1 Preparation of MO solutions

To prepare solutions at concentrations of 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 and 14.00 ppm, portions of 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 and 14.00 cm³ of stock 1000 ppm MO standard solution were transferred into each 1000 mL volumetric flasks and diluted with deionized water.

3.5.2.2 Adsorption study of methyl orange

1) 0.5000 g of adsorbent material was placed in a 250 mL flask and 100 cm³ of MO solution with the following concentrations: 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 or 14.00 ppm was added.

2) The mixture was shaken at 30 °C.

3) The equilibration time used for MO adsorption was 80 minutes.

4) After adsorption was in equilibrium, the solution was taken out from flask and filtered.

3.5.2.3 Measurement of MO concentration by UV-Vis spectrometry

A blank was run together with the sample through the entire procedure and wavelength at 465 nm was used for visible absorption.

3.5.3 Adsorption study of I₂ on the adsorbent material

3.5.3.1 Preparation of I₂ solutions

To prepare iodine solutions at concentrations of 1.000×10^{-3} , 2.000×10^{-3} , 3.000×10^{-3} , 4.000×10^{-3} , 5.000×10^{-3} , 6.000×10^{-3} , 7.000×10^{-3} and 8.000×10^{-3} M, portions of 10.00, 20.00, 30.00, 40.00, 50.00, 60.00, 70.00 and 80.00 mL of stock I₂ solutions were transferred into 100 mL volumetric flask, diluted to mark with deionized water.

3.5.3.2 Adsorption study of I₂

1) 0.5000 g of adsorbent material was placed in a 250 mL flask and 100 cm³ of I₂ solution with the following concentrations: 1.000×10^{-3} , 2.000×10^{-3} , 3.000×10^{-3} , 4.000×10^{-3} , 5.000×10^{-3} , 6.000×10^{-3} , 7.000×10^{-3} or 8.000×10^{-3} M was added.

2) The mixture was shaken at 30 °C.

3) The equilibration time used for I₂ was 60 minutes.

4) After adsorption was in equilibrium, the solution was taken out from flask and filtered.

3.5.3.3 Measurement of I₂ concentration

A blank was run together with the sample through the entire procedure and wavelength at 350 nm was used for UV-Vis absorption.