CHAPTER III

METHODOLOGY

3.1 Model Apparatus

3.1.1 pH meter

3.1.2 Rotary agitator

3.1.3 Magnetic stirrer

3.1.4 Inductively Coupled Plasma (ICP) Spectrometer

3.1.5 Analytical balance 4 digit

3.1.6 X-ray Fluorescence (XRF) Spectroscopy

3.1.7 X-ray Diffraction (XRD) Spectroscopy

3.1.8 Refrigerator

3.1.9 Oven

3.1.10 Vacuum pump

3.1.11 TOC Analyzer

3.1.12 Microwave Digestion

3.1.13 Mechanical Mixer

3.1.14 Shaker

3.1.15 Atomic Absorption (AA) Spectroscopy.

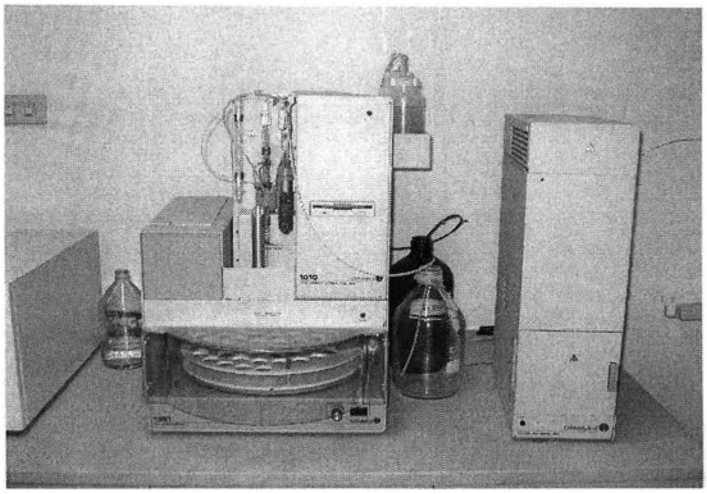

Figure 3.1 TOC Analyzer

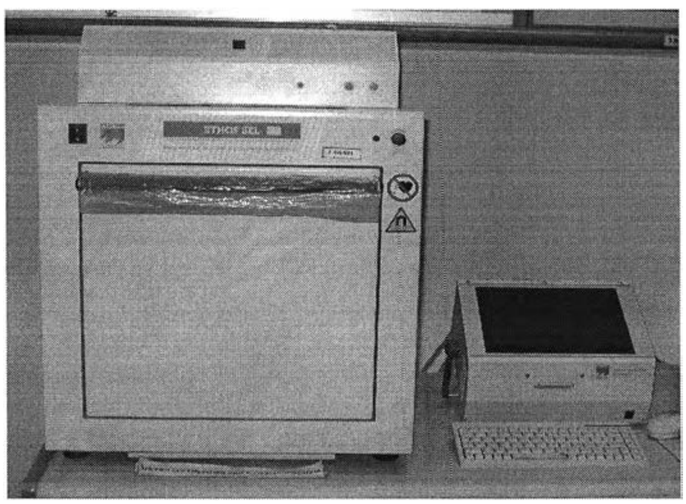

Figure 3.2 Microwave Digestion

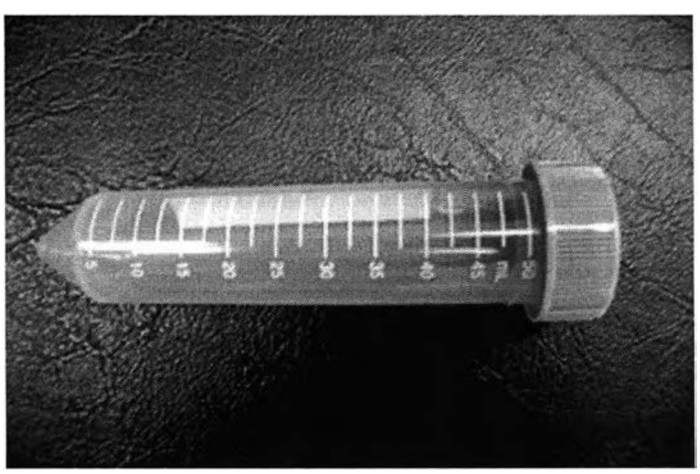

Figure 3.3 Centrifuging Teflon® Bottle

3.2 Analytical Apparatus

The apparatus for analyzing particle size characteristics, specific gravity of solids, total iron content, iron concentrations, total organic carbon, Concentration of Zinc and oxide content are shown in Table 3.1 below.

Table 3.1 Analytical Apparatus

Parameter	Method/ Apparatus
Particle size characteristics	Sieve Analysis
Total iron content	USEPA method 3051
Iron concentrations	USEPA method 7000 A
Total organic carbon	TOC analyzer
Specific gravity of solid	ASTM D854
Concentration of Zinc	USEPA 7950
Chemical composition	XRF

3.3 Materials

3.3.1 Test Materials:

3.3.1.1 Spent foundry sands: Four spent foundry sands were used in this study. The spent foundry sands were obtained from foundries in Saraburi, Thailand. Type of manufacture and sand binder is shown in Table 3.2.

Table 3.2 Type of manufactures and sand binder

Foundry Sand	Type of Manufacture	Type of Binder
Sand 1	Manufacture of fasteners (Bolts and Nuts)	Clay
Sand 2	Manufacture of seal door moulding inner and outer waist, front and rear windshield, roof moulding	Clay
Sand 3	Manufacture of cast iron parts for automotive, industry agricultural, diesel engine industry, compressor industry	Clay
Sand 4	Manufacture of automotive parts	Chemical

3.3.1.2 Zero valence iron: Commercially available iron filing granular was also used as an adsorbent for these series experiments. The purity of the iron ranged from 60 – 80 % by weight.

3.3.1.3 Bentonites: Two calcium bentonites were obtained from Sud-Chemie and Thai Nippon Chemical Industry Co., Ltd. were also used for comparative test. The physical properties of these bentonites given by supplier are summarized in Table 3.3.3

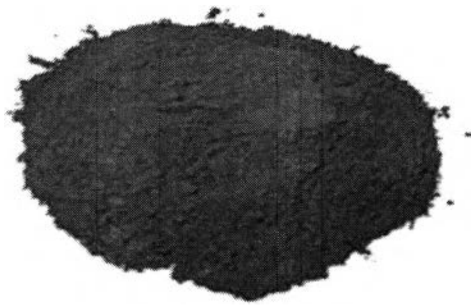

Figure 3.4 Sand 1

Figure 3.5 Sand 2

Figure 3.6 Sand 3

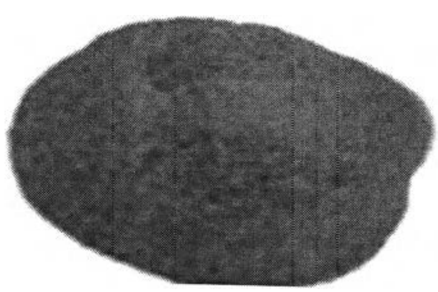

Figure 3.7 Sand 4

Figure 3.8 Iron filing

Figure 3.9 Bentonite 1

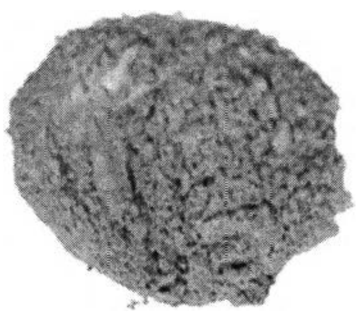

Figure 3.10 Bentonite 2

Figure 3.11 Coal dust

Table 3.3.3 Physical properties of bentonites

Typical physical properties	Bentonite 1	Bentonite 2
1.Moisture Content, %	9.90	11.6
2.pH	10.18	9.9
3.Swelling Index, ml/2g	13	20
4.Montmorillonite Content, %	82	76.3
5.Dry Particle Passing 200 Mesh, %	77.30	81.1
6.Compression Strength, N/cm^2	15.74	10.8

3.3.2 Synthetic bonded sand mixtures

Synthetic bonded sand mixtures were prepared in a mechanical mixer following method of synthetically bonded sand mixtures that available in American Foundrymen's Society handbook. Dry 2000 g of sand at a temperature that the minimum requirements of the mechanical mixer were met, which should not lower than 105°C and not higher than 110°C. After drying and cooling to room temperature, weight the correct amount of sand, bentonite, coal dust and other component as used in foundry mixture. Place the dried sand and then dried bonding materials in the mixer and mix for 2 min.

Following the 2 min mixing period, allow 2 min for dust to settle before removing the mixer cover. Add the amount of water gradually within 30 sec to give the required moisture content as used in foundry, plus sufficient additional moisture to allow for evaporation during mixing and mix for the period of 10 min remove the sand from the mixture to container as quickly as possible.

3.3.3 Synthetic Groundwater

Groundwater contaminated with zinc was synthesized for experiments in laboratory scale by dissolving zinc chloride in DI water to yield an expected zinc concentration and mixed with sodium azide (0.1% by weight) to prevent biological activity. The initial pH of solution was adjusted with 1.0 M nitric acid or 1.0 M sodium hydroxide.

3.3.4 Chemicals

All chemicals are reagent grade. The chemicals included tetrasodium pyrophosphate ($Na_4P_2O_7$), sulfuric acid (H_2SO_4), nitric acid (HNO_3), sodium hydroxide (NaOH), sodium azide (NaN_3), methylene blue, and zinc chloride ($ZnCl_2$).

3.3.5 Glassware

All glassware used in this study such as Beaker, glass bottle, volumetric flasks, suction flasks, buret, pipet and Centrifuging Teflon® Bottles were specially cleaned to minimize contamination. Glassware was washed with water and was soaked overnight with 10% HNO_3. Then, it was washed with water and dried in an oven before use.

3.4 Analytical Methods

3.4.1 Leaching ability

The leachate extraction procedure outlined in the 6^{th} Notification of the Ministry of Industry (1997) was conducted to determine the leaching ability. A 100 g of spent foundry sand that has particle size pass a 9.5 mm sieve was placed in beaker and add leachant or synthetic acid rain extraction fluid until the pH of mixture is 5.0. Adjust the volume of mixture to the ratio leachant is 20 times of the weigh of waste. Rotate at 30 rpm for 18 hours at ambient temperature. Solids and suspended materials were removed through a glass fiber filter.

3.4.2 Total iron content

An acid digestion was conducted following USEPA Method 3051 to measure the total iron content. A 0.5 g sample of green sand was digested in 10 ml of concentrated nitric acid for 10 min using a microwave oven. Fluorocarbon digestion vessels were used. Groups of six vessels were evenly located on the turntable in the microwave oven. The vessels were diluted to 1: 1 using distillated water, and solids and suspended materials were removed using a glass fiber filter.

Iron concentrations from digestions were measured by atomic absorption using a Varian SpectrAA 800 following USEPA Method 7000A. Calibration standards were prepared by diluting stock standard solutions. The calibration standards were acidified using nitric acid to simulate the condition of the digestion solution. Interference effects were compensated by using standard additions as described in Lee and Benson (2002).

3.4.3 Total organic carbon

Total organic carbon (TOC) content of each foundry sands was measured by using a solid TOC analyzer. Inorganic carbon existing in the form of calcite or dolomite was removed beforehand by adding 4 M HCl as described in Methods of Soil Anlaysis (Nelson and Sommers, 1982).

3.4.4 Clay content

The methylene blue titration was conducted on each foundry sand following ASTM C 837-99 to measure total amount of clay content. A 2.00 g sample of green sand has been dried in accordance with the procedure in test method C 324. Add 300 ml of distilled water to the beaker and stir with the mixer until the clay is uniformly dispersed. Determine the pH of the slurry and add sufficient sulfuric acid to bring the pH within the range from 2.5 to 3.8. With the slurry still under the mixer, fill the buret with the methylene blue solution and observe the appearance of a light blue halo around the drop on the filter paper.

3.5 Experimental procedures:

3.5.1 Series of batch kinetics tests (Lee et al. 2004) were initially conducted to determine the sorption kinetics for zinc in the presence of spent foundry sand. The initial zinc concentration is 50 mg/l. Two grams of spent foundry sand, bentonite or zero valent iron were placed in 50 ml centrifuging Teflon® bottles containing 50 mg/l of zinc solution. Then, the bottles were shaken for various reaction times and concentrations were measured at designated sampling interval. Blanks without green sand were used as controls to estimate losses of zinc. The test also repeated for sorption characteristics on bentonites and iron filing in the same condition to determine applicable trend.

3.5.2 Synthesis foundry sand by using an appropriate mix of pure sand, bentonite, and other components follow the proportion of spent foundry sand that has the great sorptive capacity and also compare the removal efficiency of synthesis foundry sand and spent foundry sand.

3.5.3 Enhanced the removal efficiency of synthetic foundry sand by using a multivariate regression analysis to optimize the proportion of significant index parameters for the best removal efficiency.

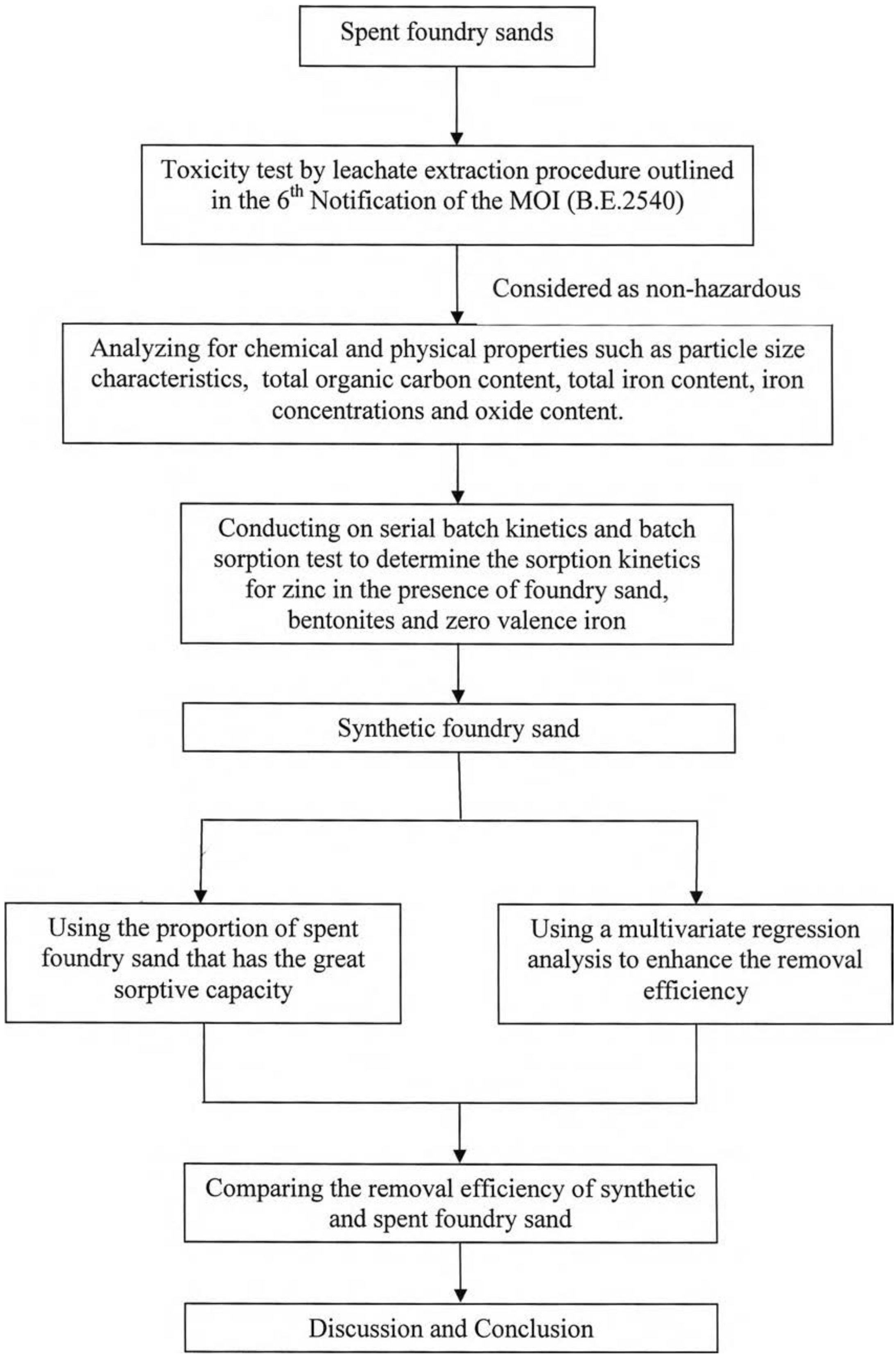

Figure 3.12 Scheme of the overall experiment procedure