CHAPTER 3

METHODOLOGY

3.1 Population

This paper concerned the oily sludge from rolling mills. Each represent-rolling mill is the famous rolling mill in Thailand. There are 2 factories in each rolling type.

The represent hot rolling mills were:

1. Sahaviriya Steel Industry PCL. (SSI)
2. Siam Strip Mill PCL. (SSM)

The represent cold rolling mills were:

1. Thai Cold Roll Steel Sheet Co.,Ltd. (TCRSS)
2. Siam United Steel Co.,Ltd. (SUS)

Each source was taken 4 times in every 10 batches of dewatered process before landfilling.

3.2 Apparatus and reagent

3.2.1 Apparatus for determining moisture content of sample

1. Evaporation dish
2. Analytical balance (Capable to weighing to 0.0001 g.)
3. Dessicator

3.2.2 Apparatus and reagent for sample preparation and Microwave extraction

- Apparatus

1. Mortar

2. Sieve (size 20 mesh)
3. Drying oven
4. Dessicator
5. Analytical balance (Capable to weighing to 0.0001 g.)
6. Suction flask
7. Bushner
8. Suction pump
9. Vial or glass bottle for collect sample, obtained material.
10. Microwave extraction set and also software set (Milestone Inc.)

- Reagent
  1. Acetone
  2. N-Hexane
  3. Petroleum ether

3.2.3 Apparatus for determining fine iron oxide property

1. X-ray fluorescence spectrophotometer
2. Computer and software

3.2.4 Apparatus and reagent for determining oil property

3.2.4.1 Heating value

- Apparatus
  1. Bomb calorimeter and accessory (CBA-305-010 M Automatic adiabatic Bomb calorimeter, Gallenkamp, HAAKE Refrigerated Baths and Circulators, Model D1-G)
  2. Oxygen bomb (Model 350 K , Gallenkamp)
  3. Weighing scale
  4. Hot plate

- Reagent , glass wear and others
  1. Benzoic acid. Standard
  2. Sodium Carbonate solution
  3. Distillation water

4. Phenolphthalein indicator
5. Burette
6. 250 ml. Beaker
7. Firing wire
8. Thread

3.2.4.2 Sulfur content

- Apparatus
  1. Muffle furnace (Temp. 800 – 1,000 ^{o}C)
  2. Hot plate

- Reagent , Glassware and Others
  1. Hydrochloric acid (1:9 v/v)
  2. Barium chloride 10%
  3. Silver nitrate solution
  4. Distillation water
  5. Porcelain crucible 30 ml.
  6. Beaker 250 and 600 ml.
  7. Glass cone
  8. Pipet 1 and 10 ml.
  9. Filter paper (Whatman No.40)
  10. Dessicator

3.2.4.3 Chloride content

- Reagent , Glassware and Others
  1. Potassium Chlomate Solution
  2. Silver Nitrate Standard Solution 0.0141 N
  3. Sodium Chloride Standard 0.0141 N
  4. Aluminum Hydroxide Suspension
  5. Distillation water
  6. Flask 250ml.
  7. Burette
  8. Pipette

3.2.4.4 Heavy metal

- Apparatus: Induced couple plasma spectrophotometer (ICP)

3.3 Data collection

Further to the population part, there were four represented rolling mills. The represent sludge samples in each factory would be collected after dewatered process in ready to disposed form for four times in every 10 batches.

First step, the initial sludge had to be grinding and drying in order to be dried sludge. Then, it would be extracted by microwave extraction. In the extraction part, % oil content was the main collected data that was calculated from weighing in each step (detail as in Appendix B) and tried to select suitable extraction condition for this sample. Method blank and calculating on dry weight basis was necessary. Considered factors of the microwave extraction were three temperature (at 90, 120 and 150 °C) programs and four solvent types (acetone, N-hexane, Petroleum ether and Mixture of Acetone and N-hexane). Other factors such as pressure, extracted time, cooling time, etc. would follow manufacturer's application. Cost estimation or cost of operation with microwave extraction was also considered

After extraction step, solid phase and oil phase were separated by filtration and then each phase would be studied in term of utility. Oil phase would removed solvent out to purify and then it was analyzed important parameters for all fuel properties using in cement kiln which included heat value, heavy metal (Chromium, Vanadium, Nickel, Zinc, Copper, Tellurium, Arsenic, Lead, Mercury and Cadmium), Sulfur and Chloride content. The results would be compared with secondary fuel and lignite properties of Siam Cement (Kaeng Koi) Co.,Ltd. Anothter phase, the obtained solid would be analyzed percentage of iron using as raw material in steel-remelting industry.

All results of this study would be calculated in percentage of sample dry weight. However, each composition of raw sludge would be converted in sample wet weight unit in section 4.2.4 as well.