CHAPTER 3

METHODOLOGY

3.1 Materials

3.1.1 MSWI Fly Ash Sample

In the experiment, approximately 500 kg of MSWI fly ash was sampled during the normal plant operation in March of 2001. The sample was collected in double plastic bags and kept in closed drums to prevent moisture in the air. Before carried out any tests, the fly ash sample was again sampled from each drum and sifted pass through a standard sieve No. 200 (75-micron openings).

3.1.2 Portland Cement

The Elephant brand ASTM Type-I Portland cement according to ASTM C150-95 manufactured by the Siam Cement Company Ltd., Bangkok, Thailand is used throughout the experiments.

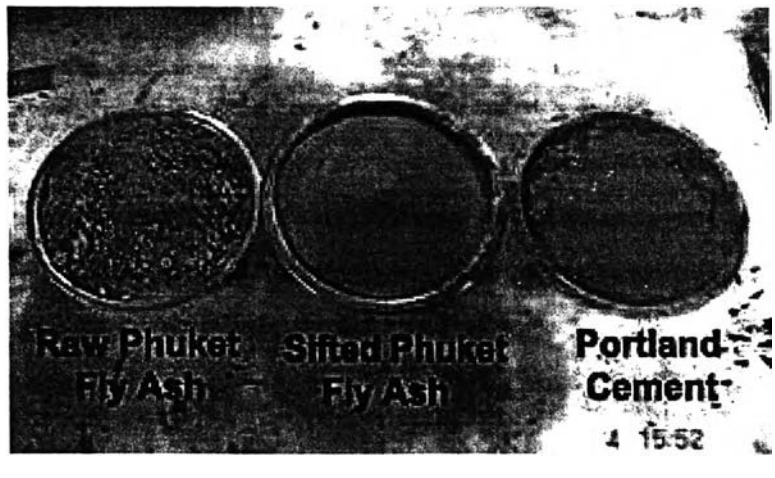


Figure 3.1 Raw MSWI Phuket Fly Ash, Sifted MSWI Fly Ash, and Portland Cement

3.1.3 Sand

River sand used for making specimens was natural silica sand that conforms to the requirements for graded sand as specified by ASTM C778-92.

3.1.4 Water

Ordinary tap water was used for all mixed.

3.2 Experimental Programs

3.2.1 Characterization of MSWI Fly Ash

3.2.1.1 Sieve Analysis

ASTM C136-95 is designed to determine grain size distribution of material larger than 75 microns. As-received MSWI fly ash was first dried to constant mass at a temperature of 110±5°C so as to avoid lumps of fine particles being classified as large particles and also to prevent clogging of the finer sieves. With the numbers of 4, 8, 16, 30, 50, 100, and 200 mesh sizes, the sieves were nested in order of decreasing sizes of openings from the top to bottom. About 500 grams of dried sample was placed on the top sieve and constantly sifted for a sufficient period as described in this standard test method.

3.2.1.2 Particle Size and Specific Surface

As cement replacement material in the subsequent testing, MSWI fly ash particles that passed though a standard sieve No. 200 were next studied the size distribution of fine textures. The ash was subjected to particle size analysis by Malvern Particle Size Analyzer model Mastersizer S that can measure particle size ranging from 0.05 to 880 microns. In this experiment, water was used as a medium with dispersing refractive index of 1.33.

According to ASTM C204-96, the standard test method was used to determine fineness of sifted MSWI fly ash by Blaine air-permeability apparatus in view of specific surface. Specific surface is expressed as total surface area in terms of square centimeters per gram of ash sample by measuring the consuming time for a fixed quantity of air flows through a compacted fly ash bed of specified dimensions and porosity. This method generally requires calibration of the apparatus with standard cement. The weight of sample used for the test was the same as that used in the calibration test, in this case, 2.14 grams. The weighed sample was placed and compacted in a permeability cell, to which the manometer tube for airflow measurement was attached.

3.2.1.3 Bulk Specific Gravity

Bulk specific gravity is defined as the ratio of the weight of a given volume of sample, including voids between the grains, to the weight of an equal volume of water. The procedure described in ASTM C188-95 was used to determine bulk specific gravity of Portland cement and adapted to calculate that of the MSWI fly ash. About 50 grams of fly ash sample was used instead of 64 grams of cement as recommended in ASTM C311-96. In this test, kerosene was used in place of water to ensure that all grains of the ash were wetted by the liquid and hydration of the sample was minimized.

3.2.1.4 Moisture Content and Loss on Ignition (LOI)

The technique to determine moisture content is described in ASTM C311-96. The sample was first dried to a constant weight at 105-110°C in a ceramic crucible. Then, it was further cooled to room temperature in a dessicator to prevent moisture absorption. The weight loss is assumed to be absorbed water.

Loss on ignition (LOI) is also defined in ASTM C311-96 and ASTM C114-94 as the weight fraction of material that is lost by heating in a muffle furnace at 750°C. The residue left from moisture content determination shall be ignited to a constant weight in an uncovered porcelain crucible at 750±50°C. LOI is a measurement of unburned carbon remaining in the ash. This value is perhaps the single most critical

characteristics of the fly ash. Higher carbon contents can result in air-entrainment problems and can adversely affect the performance of fly ash concrete. LOI can also be used as an indicator of the degree of burnout in fly ash or defined the combustion efficiency.

3.2.1.5 pH and Conductivity

The guidance to determine pH and conductivity of MSWI fly ash is expressed in the U.S. EPA SW-846 Method 9045C. The aqueous phase sample was prepared by adding 100 ml of deionized (DI) water in a 150-ml beaker, in which approximately 5 grams of fly ash was first placed. The sample was regularly stirred at least 5 minutes and let to gradually stand and settle out for about 15 minutes from the suspension. After complete segregation, the SensIon-1 pH meter and the SensIon-5 conductivity meter were employed to determine the pH and conductivity of the sorted aqueous phase sample.

3.2.1.6 Morphology

The MSWI fly ash features were investigated by a JEOL JSM-6400 scanning electron microscope (SEM). The ash was initially glued on an aluminum stub and coated with goal-palladium alloy inasmuch as it was not electrically conductive. This method examines only a tiny area of fly ash particles. It is however believed to be representative of reaction process of fly ash since the result can indicate not only the performance of fly ash-cement concrete but the potential for utilization as well.

3.2.1.7 Bulk Chemical Compositions

ASTM C618-96 lists the required chemical compositions for coal fly ash used in concrete. These limitations are based on oxides of silica, aluminum, iron, calcium, and sulfur. X-ray fluorescence (XRF) spectroscopy was carried out to measure bulk chemical compositions of Phuket fly ash. This method provides useful information on the overall compositions of the analyzed materials. To obtain a good representative, the sample was initially ground in a ceramic mortar to achieve a homogeneous size of below 45 microns since the X-ray only penetrates up to a few

millimeters from surface of a sample. With 1.5 grams of H_3BO_3 (2.5% by weight) binder, around 4.5 grams of ground sample was pressed into a pellet for convenient handling and measurement. Before running, the pilled sample was put in a sample cup and loaded on a feeder tray of the Philips XRF Spectrometer model 2400.

3.2.1.8 Mineralogical Compositions

The powder X-ray diffraction (XRD) spectrometer, Bruker model D8 Advance, was used to identify mineralogical compositions of Phuket fly ash in terms of crystalline phases as shown in Figure 3.2. XRD patterns were obtained with a computer-controlled diffractometer equipped with a copper X-ray tube and a scintillation detector. A graphite monochromator was used to produce diffracted lines according to a single X-ray wavelength with low background. A sample was prepared by dehydrating in an oven and grinding in a ceramic mortar until homogeneous with particle size of below 45 microns. Operating conditions of the instrument were set at 40 kV accelerating voltage, 40 mA current, 0.015 step time, and 10° to 70° 2θ scanning range. The measured patterns were matched against a powder diffraction file database (PDF) from International Center for Diffraction Data (ICDD) that contains patterns for a large number of compounds.

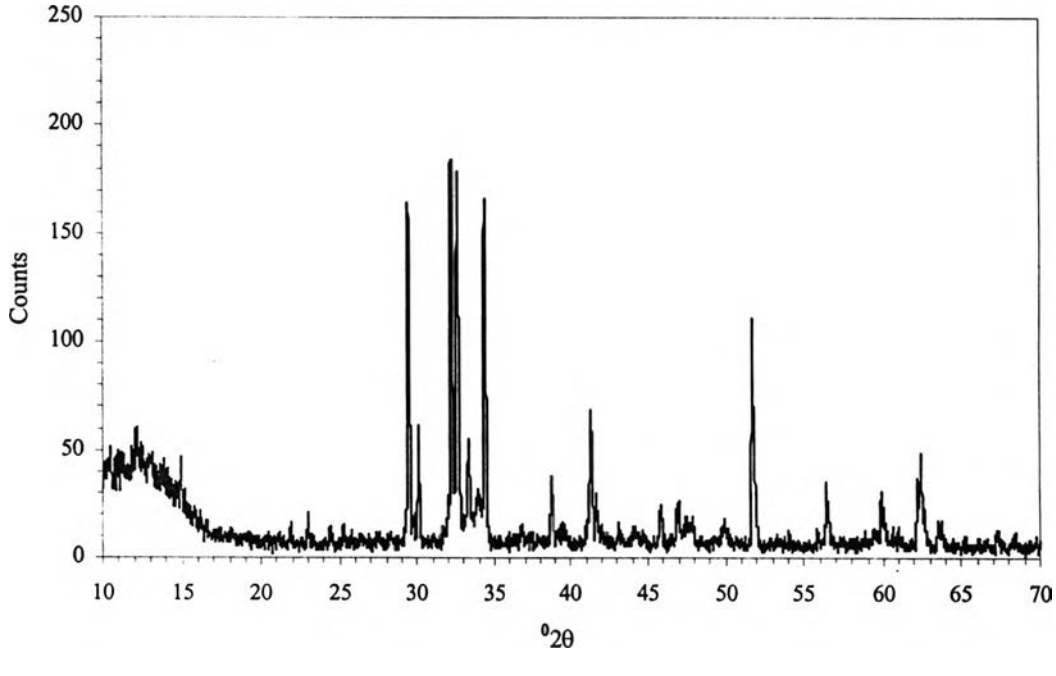


Figure 3.2 Example of Diffraction Pattern Obtained from XRD Spectrometer

3.2.2 Properties of Portland Cement with MSWI Fly Ash

3.2.2.1 Normal Consistency and Setting Time

The standard test method for normal consistency (ASTM C187-86 reapproved 1991) and time of setting (ASTM C191-92) of hydraulic cement were used to analyzed MSWI fly ash-cement pastes by vicat apparatus. About 650 grams of binder, or cement and fly ash (c+fa), were mixed with water to form quickly in to ball shape. The ball was put and pressed in a vicat ring and then leveled carefully within 30 seconds after complete mixing. The amount of water in the paste shall be normal consistency when the 10-mm plunger end of rod can penetrate to a point 10±mm from the paste surface in 30 seconds after being released. Initial setting time is the time when a penetration of 1-mm vicat needle is 25 mm after molding and remaining in the moist cabinet for 30 minutes.

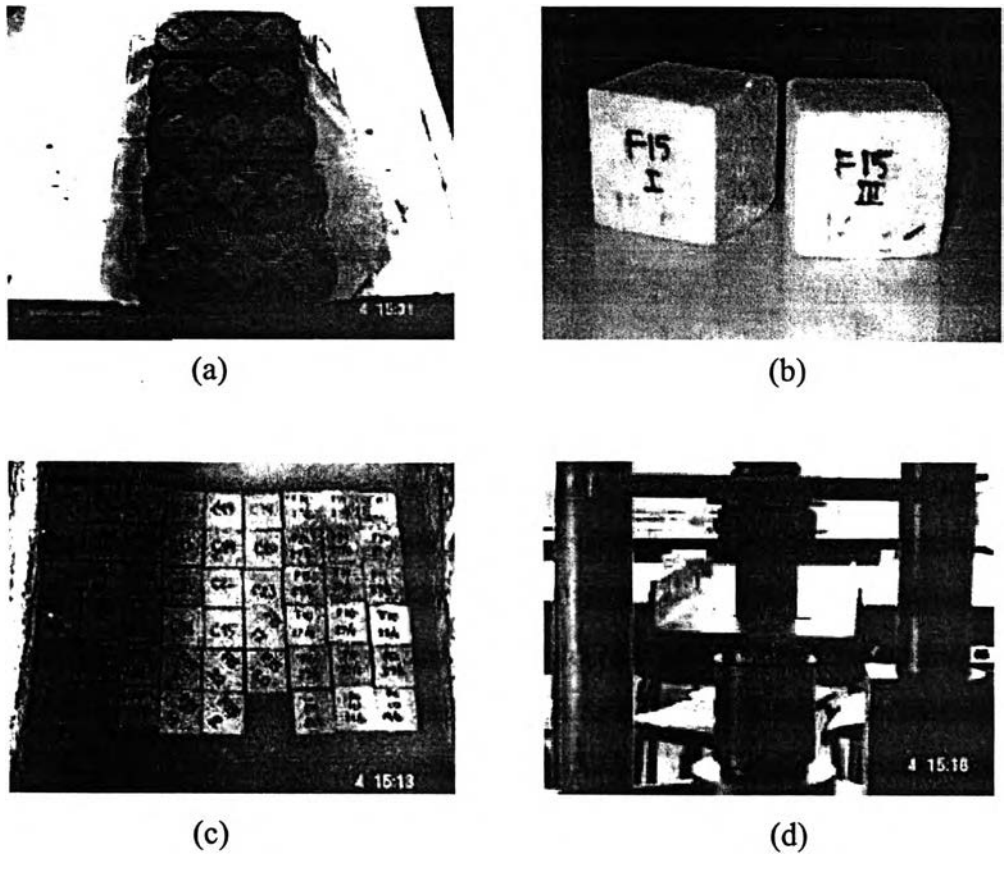
3.2.2.2 Compressive Strength Development of MSWI Fly Ash-Cement Mortars

The procedures to measure compressive strength of hydraulic cement mortars are described in ASTM C109-95 that specifies a flow of sample at 100±5. The method must be criticized to vary water-to-binder ratio (w/(c+fa)) in the mixes in order to achieve the same flow. The variation in water-to-binder ratio could affect compressive strengths of fly ash mortars and reaction rate in the mixes. Therefore, the test is kept the constant water-to-binder ratio with corresponding to various flows.

Phuket fly ash directly replaced cement in the mixes with 0%, 10%, 15%, and 25% by weight. According to the requirement in ASTM C109-95, the weight ratio of cement to sand is 1.00:2.75 and water to binder ratio is 0.485. Two-inch or 50-mm tested cubes were prepared. The cubes were cured one day in the molds and then stripped and immersed in lime water until tested. Unconfined compressive strength tests were carried through mortar cubes at the age of 1, 3, 7, 14, 28, 45, and 60 days. For each different levels of cement replacement, three specimens were prepared and tested so that the average would be reported. The amounts of each ingredient required to make the mortar mixes for twenty-four cubes in each mix were given in Table 3.1.

Table 3.1 Mix Proportions of MSWI Fly Ash-Cement Mortars for w/(c+fa) = 0.485

Sample No.	% Replacement	Mix Proportion by Weight		
		Cement	Fly Ash	Sand
F00	0	1.00	0.00	2.75
F10	10	0.90	0.10	2.75
F15	15	0.85	0.15	2.75
F25	25	0.75	0.25	2.75



(a) (b)

(c) (d)

Figure 3.3 Preparation and Testing of MSWI Fly Ash-Cement Mortar Specimens; a) Molded Mortars, b) Demolded Mortars, c) Cured Mortars in Saturated Lime-Water, and d) Compressive Strength Testing Machine

3.2.2.3 Development of Hydration and Pozzolanic Reactions in MSWI Fly Ash-Cement Pastes

To study the process and products of hydration and pozzolanic reactions formed in the MSWI fly ash-cement paste, XRD analysis was performed on fly ash-cement pastes to eliminate interference peaks originated from sand in diffractogram patterns. The pastes with water-to-binder ratio of 0.485 and 0, 10, 15, and 25 percentage of fly ash substitutions were investigated. The mixtures were cured in airtight plastic bags at normal temperature to minimize their carbonation until tested. The cured samples were tested at 1, 3, 7, 14, 28, 45, and 60 days. Before testing with XRD spectrometer, the sample was first ground in acetone for about 5 minutes to stop its hydration reaction. The soaked sample was dried in an oven at 60°C for about 4 hours and again ground in ceramic mortar to obtain homogeneous size of approximately 45 microns. The operation conditions of the XRD instrument were as described in 3.2.1.8.

3.2.3 Leachate Characteristics of MSWI Fly Ash-Cement Products

The MSWI fly ash-cement products were analyzed for the presence of heavy metals according to the leachate extraction procedure described in the Notification of Ministry of Industry No. 6, B.E. 2540 (1997). This test does not determine the total elemental contents of the samples, but indicates the leaching potential of some elements. To follow this procedure, both MSWI fly ash and MSWI fly ash-cement products were crushed to a particle size smaller than 9.5 mm, if necessary. About 100 grams of the crushed samples was added with synthetic acid rain extraction fluid at a liquid-to-solid weight ratio of 20:1. The extraction solution was prepared by adding 80% of sulfuric acid solution and 20% of nitric acid solution in the deionized (DI) water until its pH reached 5. The weighed samples and extraction fluid in the HDPE bottles were then agitated with a rotary extractor for a period of 18 hours at 30 rpm at normal temperature. After 18 hours of agitation, the extraction solution were filtered through a 0.6 to 0.8-μm glass fiber filter.

To reduce interference by organic matters and to convert metals associated with particulate to a form (usually the free metal) that can be determined by Atomic Absorption Spectrophotometer (AAS) or Inductively-Coupled Plasma (ICP) Spectroscopy, the extract required microwave-assisted acid digestion according to U.S. EPA SW-846 Method 3015. In the standard test method, a 45-ml extract was digested in 5-ml of concentrated nitric acid in a digestion vessel for 10 minutes in order that the extract temperature reached 160°C. It was further heated until 165°C for about 10 minutes. After the digestion process, the digested solution was cooled to room temperature and kept in a HDPE bottle prior to analysis.

The solution was analyzed for contaminants according to the procedures described in the Notification of Ministry of Industry No. 2, B.E. 2539 (1996). The results were compared against the regulatory limits to ensure that the MSWI fly ash-cement products are safe to human health and environment. Except mercury (Hg), all analytes; namely, silver (Ag), arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), lead (Pb), and selenium (Se) were analyzed by Perkin-Elmer ICP Atomic Emission Spectrometer model PLASMA-1000. Mercury was tested by Varian Atomic Absorption Spectrophotometer model Spectr AA-300 with manual cold vapor generation technique.