CHAPTER 3

MATERIALS AND METHODS

In this chapter are presented the Portland cement preparation, crystal structure analysis, microstructure analysis, thermoelectric properties measurement, thermoelectric module fabrication and measurement. The schematic diagram of methodology as shown in Figure 12.

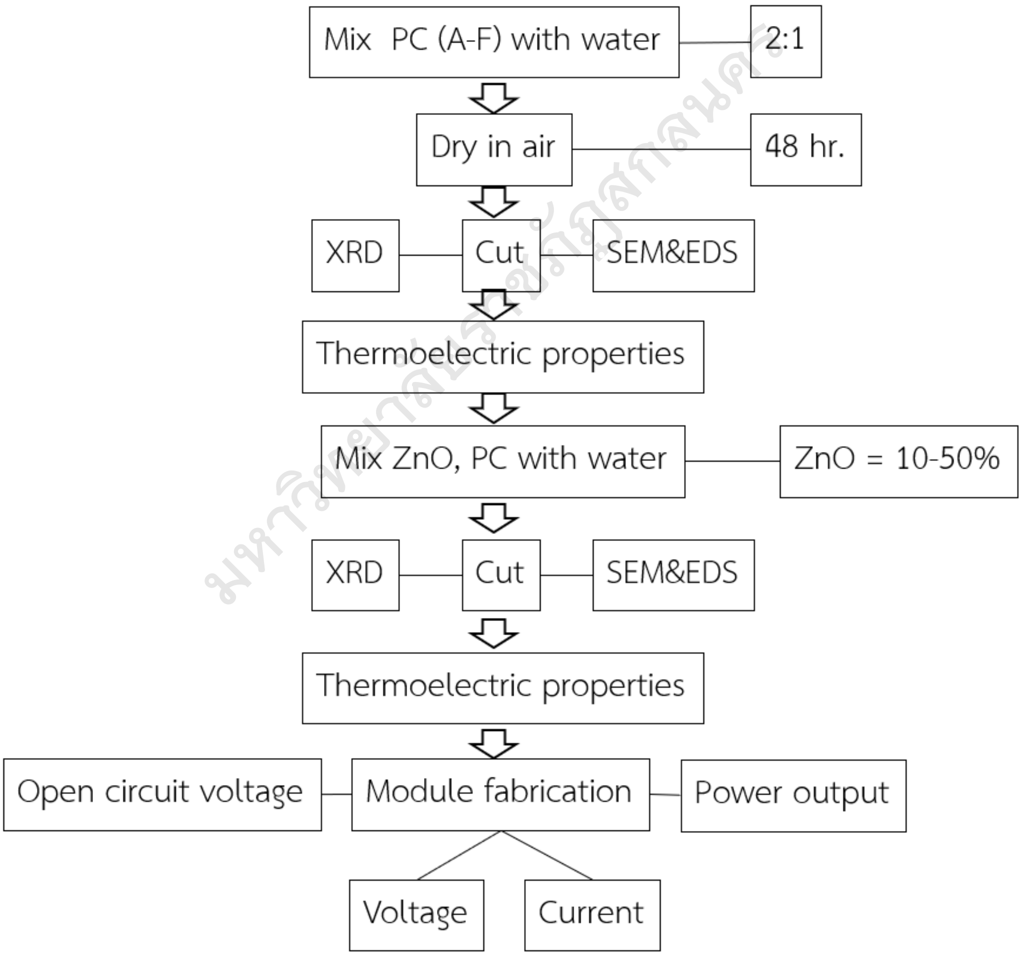


Figure 12 The schematic diagram of methodology

Materials preparation

Preparation of Portland cement samples

6 brands of Portland cement of SCG (PC–A), TPIL (PC–B), TPIL299 (PC–C), Lion (PC–D), Lotus (PC–E) and Eagle (PC–F) were used raw materials as shown in Figure 13 (a–f), respectively.

(a) (b) (c) (d) (e) (f)

Figure 13 6 brands of Portland cement (a) PC–A, (b) PC–B, (c) PC–C, (d) PC–D, (e) PC–E and (f) PC–F

The PC–A, PC–B, PC–C, PC–D, PC–E and PC–F were mixed with water in ratio 2:1 by weight on square shape block and dry in air for 48 hours. All samples were cut in size of 2×2×2 cm^3 for crystal structure analysis, morphology and elementary observation, thermoelectric properties including Seebeck coefficient and electrical resistivity measurement.

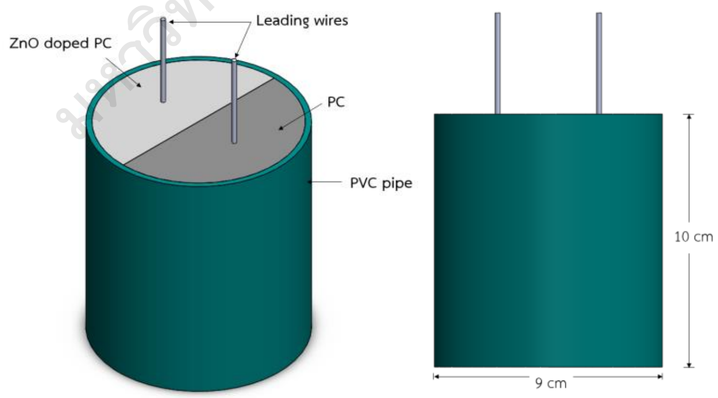
Preparation of Portland cement added with ZnO (10%, 20%, 30%, 40% and 50% by wiegth) samples

In this study we used the PC-A for added with nano ZnO (10%, 20%, 30%, 40% and 50% by weight). The PC-A and nano ZnO were mixed with water in ratio 2:1 by weight on square shape block and dry in air for 48 hours. All samples were cut in size of 2 × 2 × 2 cm^3 for crystal structure analysis, morphology and elementary observation, thermoelectric properties including Seebeck coefficient and electrical resistivity measurement.

PC Thermoelectric device fabrication

PC Thermoelectric devices design

Thermoelectric devices were designed by solid works program. The PC thermoelectric device was designed by cylindrical shape in 9 cm of dimeter and 10 cm of high. The PC thermoelectric device design model shows in figure 14 (a and b).



(a) (b)

Figure 14 The model of PC thermoelecric device design (a) material and (b) size

PC Thermoelectric devices fabrication

The thermoelectric devices fabrication was used highest power factor of p-type PC (A-F) and n-type PC-A added with nano ZnO (10%, 20%, 30%, 40% and 50%) for thermoelectric element. The fluid of p-PC-C + nano ZnO 40% and n-PC-F were prepared and poured into the cylindrical block by half each side. The 2 steel bars were impaled in the n and p fluid before drying for electrode. The PC thermoelectric device shows in figure 15.

Figure 15 The PC thermoelectric device

Crystal structure analysis

The crystal structure was analyzed by X-ray diffractometor (Shimadzu, XRD-6100) in scan rang 20-80 degree of 2θ for 2 deg/min of scan speed with 0.02 of sampling pitch. All electromagnetic radiation is characterized by its wave character λ or by means of photon energy E. The relationship between these quantities can be presented by:

$$v = \frac{c}{\lambda} \tag{5}$$

$$E = hv \tag{6}$$

$$E = \frac{hc}{\lambda} \tag{7}$$

Where c is speed of light, v is frequency h is Planck's constant.

$$E = \frac{12.398}{\lambda} \tag{8}$$

For $CuK\alpha_1$ doublet has an energy of 8.046 keV, the wavelength will be equal to 12.398/8.046 = 1.541 Å. The oscillating electric field of a light wave will interact with the electrons in matter to cause coherent scattering knowing since Christian Huygens (1629–1695) that each scattering point may be treated as a new source of spherical waves. If a periodic array of objects each scatter radiation coherently, the concerted constructive interference at specifies angles is called diffraction. Max von Laue first developed the description of the diffraction of X-ray by a crystal. William Henry Bragg and William Lawrence Bragg developed a much simpler way of understanding and predicting diffraction as show in Eq. 9.

$$2d\sin\theta = n\lambda \tag{9}$$

Where n is a positive integer, λ is the wavelength of the incident wave and d is the interplanar distance.

Microstructure analysis

The microstructure of bulk samples was analyzed by scanning electron microscope (JEOL, JSM-7600F Prime). The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS).

Thermoelectric properties measurement

The Seebeck coefficient and electrical resistivity were measured by two point prob method. A prism sample is set in a vertical position between the upper and lower blocks in the heating furnace. While the sample is heated, and held, at a specified temperature, it is heated by the heater in the lower block to provide a temperature gradient. Seebeck coefficient is measured by measuring the upper and lower temperatures T_1 and T_2 with the thermocouples pressed against the side of the sample, followed by measurement of thermal electromotive force dE between the same wires on one side of the thermocouple. Electrical resistivity is measured by the dc four-terminal method, in which a constant current I is applied to both ends of the sample to measure and determine voltage drop dV between the same wires of the thermocouple by subtracting the thermo-electromotive force between leads.
The samples were connected to Cu plat by silver paint for thermoelectric properties measurement as shown in figure 16 (a). The schematic diagram of thermoelectric properties measurement shows in figure 16 (b).

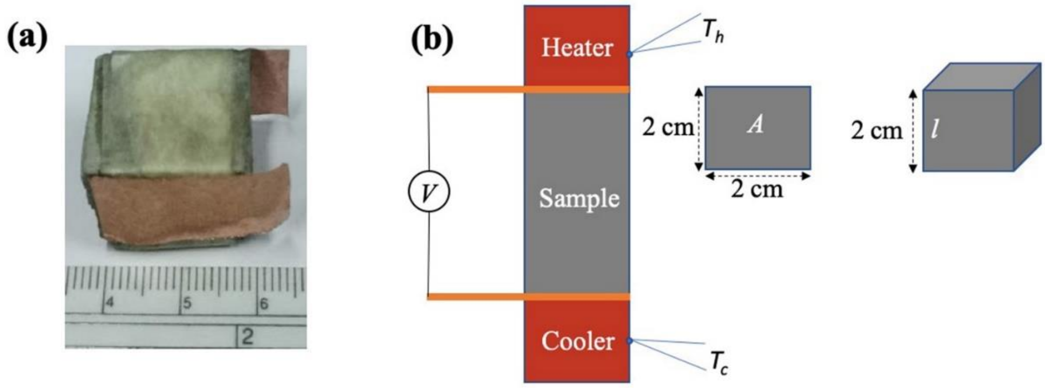


Figure 16 Portland cement bulk samples of (a) Cu electrode connecting and (b) diagram of electrical resistivity and Seebeck coefficient measurement

The Seebeck coefficient and electrical resistivity is found by using the following Eq. 12 and 13, respectively.

Seebeck coefficient (S)

$$S = \frac{\Delta V}{Temp1 - Temp2} \tag{12}$$

Where ΔV is Voltage, $Temp1$ is lower probe temperature and $Temp2$ is upper probe temperature.

$$\rho = \frac{RA}{l} \tag{13}$$

Where R is resistance value (R=V/I), A is cross section area of sample and l is distance between probes.

Thermoelectric power generation

The power generation was measured using heat source by coil heater on bottom of thermoelectric device. The electrical voltage and electrical current were measured by Picotest (M3500A 61/2 Digit Multimeter). The schematic diagram of thermoelectric device power generation measurement is shown in figure 17.

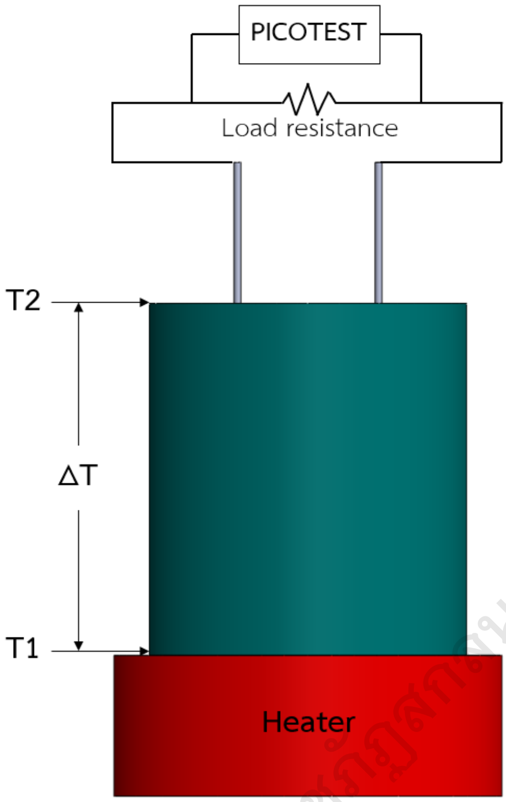


Figure 17 Schematic diagram of power generation measurement of thermoelectric