CHAPTER 3 MATERIALS AND METHODS

3.1 Materials Preparation

Six rectangular shaped glass samples of composition xBaO:(80-x)B_2O_3:20RHA (x= 45, 50, 55, 60, 65 and 70 wt%) have been prepared by using the melt quenching technique. The oxides of barium and boron used in this work were of analytical reagent grade, and the oxide of silica was of rice husk ash procured from Nakorn Pathom Province, Thailand. The rice husks were placed in porcelain crucibles and then calcined at 400, 600, 800, 1,000 and 1,100 ^oC for 5 hours in a muffle electrical furnace. The chemical composition of the rice husk ash whose composition is not known was analyzed with an energy dispersive x-ray fluorescence (EDXRF) instrument of type Panalytical, Minipal 4 spectrometer (PW 4030/45B) with Rh X-ray tube operation. For preparation of a glass sample an appropriate amounts of BaO, B_2O_3 and rice husk ash (at highest silica content) were weighed using an electronic balance having accuracy of 0.0001 g. The chemicals were mixed and contained in the crucible to place in an electric furnace at 1,200 ^oC for an hour. The melt was poured into a preheated stainless steel mold and then slowly cooled to room temperature.

Figure 3.1 Energy dispersive x-ray fluorescence (Panalytical, Minipal 4 spectrometer: PW 4030/45B).

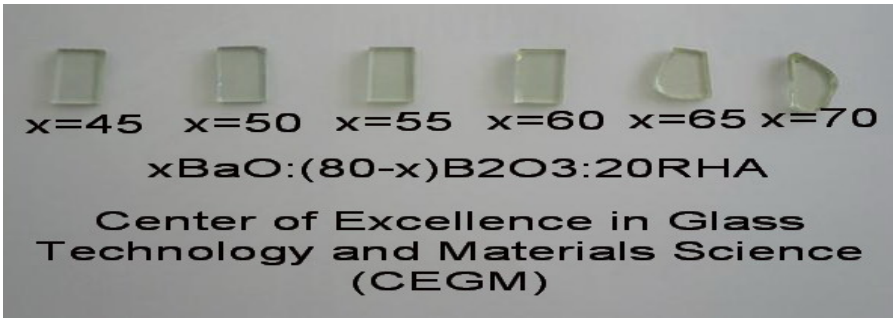

Figure 3.2 The samples are prepared.

3.2 Materials Characterization

The properties of samples were characterized using the various techniques, i.e. density measurement, refractive index, UV-Visible spectrophotometer and gamma-ray absorption; the procedures are shown in Figure3.3.

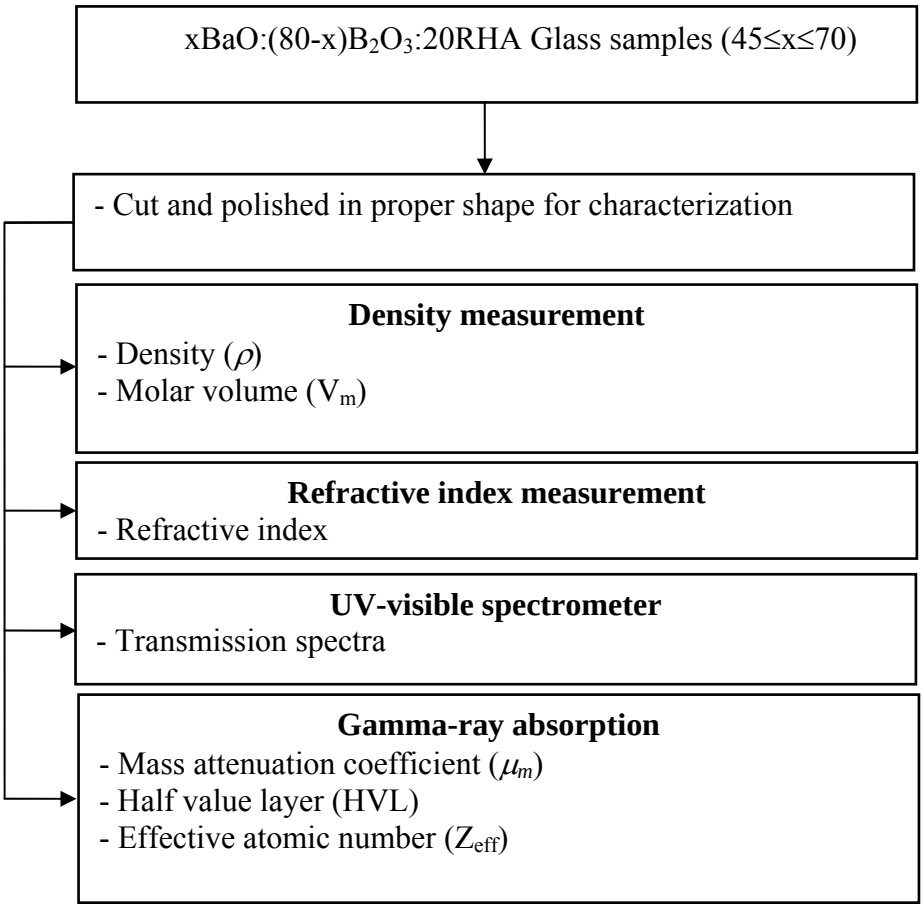

Figure 3.3 The diagram shows the steps of preparation and characterization of glasses sample.

3.2.1 Thickness Measurement

The thicknesses of the glass samples were measured by vernier caliper, which can measure down to 0.05 mm.

3.2.2 Density Measurement

In this work, the density were measured by applying Archimedes principle , the weight of prepared glass samples were measured in air and in xylene using a sensitive 4-digit microbalance (AND, HR200). Then, the density was determined from the relation.

$$\rho = \frac{W_a}{W_a - W_b} \times \rho_b$$

where W_a is the weight in air, W_b is the weight in xylene, and ρ_b is the density of xylene $(\rho_b = 0.863 g/cm^3)$. The corresponding molar volume (V_m) was calculated using the relation $V_m = M_T / \rho$ where M_T is the total molecular weight of the multi-component glass system

3.2.3 Refractive Index Measurement

The refractive index were measured by using an Abbe refractometer (ATAGO) with a sodium vapor lamp as the light source emitting the light at a wavelength (λ) of 589.3 nm (D line) and having mono-bromonaphthalene as the contact layer between the sample and prism of the refractometer.

3.2.4 UV-Visible Spectrometer

The transmission spectra of prepared glasses were performed using a UV-Visible spectrophotometer (Cary UV-50 model), together with a dual light source capable of emitting ultraviolet as well as visible light. Percentage transmission spectra were recorded in the wavelength 300–800 nm, using air as the reference. The rate interval 1 nm, average time 0.6 second and scan rate 100 nm/min.

3.2.5 Gamma-Ray Absorption

The block diagram of good geometry set up is shown in Figure 3.4. The source and absorber system were mounted on composite of adjustable stands. With the help of a screw arrangement the platform having material was also made capable of movement in the transverse direction to the incident beam for proper alignment. The sample detector solid angle was $< 0.5\times10^{-4}$ sr. The ^{137}Cs radioactive source of 15mCi strength was obtained from office of atomic for peace (OAP), Thailand. The incident and transmitted gamma-ray intensities were determined for a fixed preset time in each experiment by recording the corresponding counts, using the 2″×2″ NaI(Tl) detector having an energy resolution of 10.2% at 662 keV. The statistical uncertainly was kept below 0.3 % by choosing the counting time so that 10^5-10^6 counts were recorded in the full energy peak. All measurements were made using an ordinary counting system. The spectra were recorded using a PC-based multichannel analyzer, supplied by CANBERRA, USA.

All instrument in this research work are shown in Figure 3.5 to 3.11 for glass melting high temperature electrical furnace, glass anneal high temperature electrical furnace,

sensitive microbalance, Abbe refractometer, UV-Visible spectrophotometer, gamma-ray spectrometer and NaI(Tl) scintillation detector (for transmission experiment) respectively.

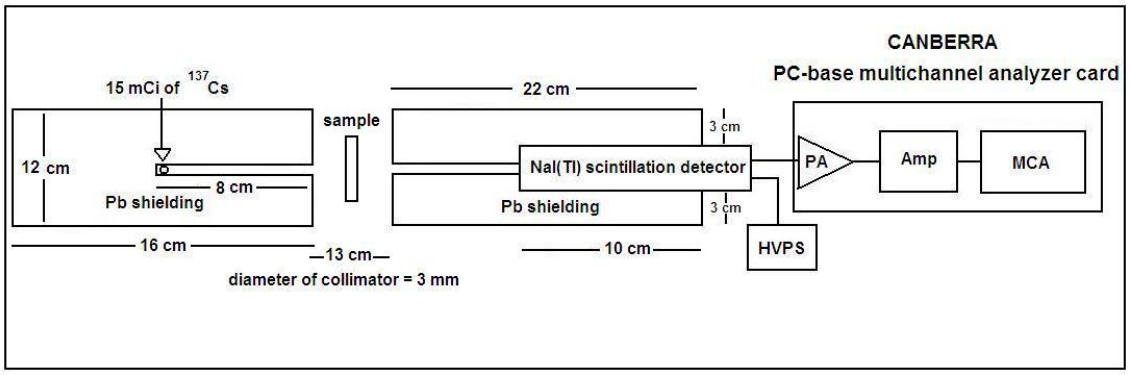

Figure 3.4 Experimental setup of transmission method.

Figure 3.5 High temperature electrical furnaces for glass melting.

Figure 3.6 High temperature electrical furnace for glass annealed.

Figure 3.7 The sensitive microbalance for density determination.

Figure 3.8 The Abbe refractometer (ATAGO).

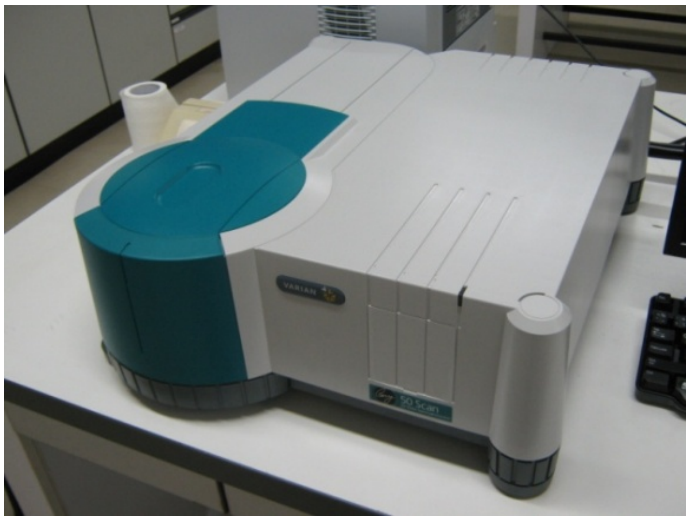

Figure 3.9 UV-Visible spectrophotometer (Cary UV-50 model).

Figure 3.10 Gamma ray spectrometer.

Figure 3.11 NaI(Tl) scintillation detector (TELEDYNE BROWN).