CHAPTER VIII
CONCLUSIONS AND RECOMMENDATIONS

The lead glycolate precursor was successfully synthesized by the "Oxide One Pot Synthesis" process with a very high yield. The product has remarkably high purity as shown by the elemental analysis data, and it is moisture stable as shown by TGA data. The FTIR result shows the Pb-O-C bonds while the NMR data give only a single peak of ethylene glycol ligand. FAB^+-MS gives the molecular peak of a trimer-[-$PbOCH_2CH_2O$-]$_3$-. Moreover, the percentages of carbon and hydrogen contents in the product are close to the theoretical values. Since the lead glycolate is more stable, it is soluble in most acid solutions, except for nitric acid. With its small particle size, lead glycolate is useful for selectively mixing with other precursors, such as titanium glycolate and zirconium glycolate, to produce lead titanate, lead zirconate and lead titanate zirconate (PZT). From the electrical properties and the microstructures at high temperature, we can summarize the lead glycolate precursor could be used as a possible starting material for ferroelectric, anti-ferroelectric, and piezoelectric materials.

The synthesis of lead titanate by the sol-gel process using lead glycolate and titanium glycolate as starting precursors gives a high purity and low sensitivity to moisture light yellow color powder. The experimental stoichiometry value between Pb and Ti is 0.995:1, close to the calculated value of 1:1. The lead titanate gel was dried and calcined below T_c of 430°C in order to inhibit the structure transformation from the tetragonal form to the cubic form which induces a change from the ferroelectric behavior to the paraelectric behavior. The highest dielectric constant of 17,470, dielectric loss tangent of 1.467, and electrical conductivity of 1.83×10^{-3} $(\Omega.m)^{-1}$ were obtained for the powder sample calcined at 300°C for 3 h. measured at room temperature and at 1000 Hz. Our synthesized material appears to be a suitable candidate for using as an electronic-grade $PbTiO_3$.

The synthesis of lead zirconate by the sol-gel process using lead glycolate and sodium tris (glycozirconate) as starting precursors gave the same results as those of lead titanate; the product is of high purity, low moisture sensitivity, and of light yellow color powder, but it has the antiferroelectric behavior. The experimental stoichiometry value between PbO and ZrO_2 is 0.9805:1.00, close to theoretically calculated theoretical value of $PbZrO_3$. The lead zirconate gel was dried and calcined below T_c (245.7°C) in order to prevent the structural change from the orthorhombic form to the cubic form of the perovskite phase. The highest dielectric constant of 2267, electrical conductivity of 3.058 $x10^{-4}$ $(\Omega.m)^{-1}$, and low dielectric loss tangent of 2.484, measured at 1000 Hz, were obtained from the $PbZrO_3$ calcined at 300°C for 1 h. Dielectric constant and conductivity decreased with calcination time and temperature when it was above the T_c. Our synthesized materials can be used as an antiferroelectric-grade $PbZrO_3$.

Moreover, the synthesis of lead zirconate titanate by the sol-gel process using lead glycolate, sodium tris (glycozirconate) and titanium glycolate as starting precursors gave a product of high purity, low moisture sensitivity, and light yellow color powder as well. The experimental stoichiometry value between PbO, ZrO_2 and TiO_2 is 1.00:0.521:0.479, close to theoretically calculated theoretical value of $Pb(Zr_{1-x}Ti_x)O_3$. The lead zirconate titanate gel was dried and calcined below limiting curve of T_c in order to prevent the structural change from the tetragonal or orthorhombic forms to the cubic form of the perovskite phase. The highest dielectric constant of 10190, electrical conductivity of 0.803 $x10^{-3}$ $(\Omega.m)^{-1}$, and low dielectric loss tangent of 1.513 measured at 1000 Hz. were obtained from the $Pb(Zr_{0.5}Ti_{0.5})O_3$ calcined at 400°C for 1 h. Dielectric constant and conductivity decreased with calcination time and temperature when it was above the limiting curve of T_c. Our synthesized materials appear to be a suitable material for many applications as an piezoelectric-grade $Pb(Zr_{0.5}Ti_{0.5})O_3$ as well.