



CHAPTER V CONCLUSIONS

In this thesis work, we demonstrated 2 techniques to synthesize $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ nanocrystalline powder using lead, titanium and zirconium glycolate precursors. Three precursors can be easily synthesized as starting materials and they are non-sensitive toward moisture and air. The first technique, PZT preparation via the sol-gel process, we showed the effects of HNO_3/NaOH , pH value, heating temperature and time on the formation of PZT gel. The best conditions for the mixture of three precursors were to use 2.5 M of HNO_3 acid adjusted pH = 3 by adding 2.5 M of NaOH. PZT gel formed when heated at 50°C for 5-6 h. The gel time of PZT was obtained using FTIR to follow the M-O-C and M-O peaks, and the strongest and sharpest peaks occurred after 5-6 h. Beyond this time, PZT gel was reversed to be solution. Then PZT crystalline was complete when calcined PZT at $800^\circ\text{-}1100^\circ\text{C}$ to obtain the pure perovskite phase having tetragonal structure, which was thermally stable.

The second technique, PZT preparation via the sol-gel process and the microwave technique, was easier to control condition than the first one. Thermal properties using TGA/DTG indicated lower temperature ($580^\circ\text{-}650^\circ\text{C}$) needed to form perovskite phase than the first technique and the general unseeded PZT ($600^\circ\text{-}700^\circ\text{C}$). A longer heating time by microwave resulted in a higher crystallinity, higher density surface and higher grain size. XRD patterns indicated the tetragonal structure with higher intensity, high degree of crystallinity and purer perovskite phase at $800^\circ\text{-}1300^\circ\text{C}$. Moreover, PZT synthesized by this technique was stable at higher temperature than the first one and the other common techniques. If Pb glycolate precursor was replaced by Pb acetate precursor, XRD pattern indicated the rhombohedral structure. That is, different precursors gave different PZT structures.