# CHAPTER 5

# CONCLUSION

La or Ba in A-site and Co or Ga in B-site of $ABO_3$ based perovskite with substitution of Sr, Ba, and Fe showed the cubic structure. The single-phase powder was obtained from lanthanum-based perovskite without calcination and Barium-based perovskite with calcination at 1,000°C. The trace amount of impurity phase was presented in cobalt containing perovskite, it might be shown that the modified citrate method is more suitable for the synthesis of Ga than Co containing perovskite.
The lattice parameter estimated from the XRD analysis was calculated for investigating the influence of A and B-site cation on the lattice parameter as shown in Table 4.2. The lattice parameter was varied, following to the size of ionic cation.

Fine particle strontium and iron substituted lanthanum gallates $La_{1-x}Sr_xGa_{1-y}Fe_yO_{3-\delta}$, where x = 0.2, 0.4, and 0.6; y = 0.2, 0.4, 0.6, and 0.8, had been synthesized by a modified citrate method. The single phase of $La_{0.8}Sr_{0.2}Ga_{0.5}Fe_{0.5}O_{3-\delta}$, $La_{0.8}Sr_{0.2}Ga_{0.4}Fe_{0.6}O_{3-\delta}$, $La_{0.8}Sr_{0.2}Ga_{0.2}Fe_{0.8}O_{3-\delta}$, $La_{0.6}Sr_{0.4}Ga_{0.2}Fe_{0.8}O_{3-\delta}$, and $La_{0.4}Sr_{0.6}Ga_{0.2}Fe_{0.8}O_{3-\delta}$ powders could be obtained both with and without calcination. The XRD analysis showed that the concentration of Sr and Fe in the matrix of LSGF powders corresponded to $x = 0.2$ for $y < 0.8$ and $x = 0.6$ for $y = 0.8$. This means that the solubility limit of each dopant depended on the concentration of the other dissolved dopant in $LaGaO_3$. The samples with $x \leq 0.2$ and $x \leq 0.6$ for Ga rich and Ga lean, respectively, belonged to single-phase $LaGaO_3$-based perovskite. If excess Sr was added, $SrLaGaO_4$ secondary phase was clearly formed in addition to the Ga-rich perovskite. While the Ga-lean perovskite showed the other unknown secondary phase when $x > 0.6$, the results indicated that in the pH range of 1.36-9.27, the single phase $La_{0.6}Sr_{0.4}Ga_{0.2}Fe_{0.8}O_{3-\delta}$ was formed without calcination and the pH had negligible effect on the structure and lattice parameter. The fine particle of these calcined powders (< 4 μm) was obtained with the average particle size 1.70 μm at pH = 1.36 and 0.56-0.60 μm at pH range between 3.39-9.27, and with a lattice parameter about 3.9 Å. The increase of the calcination temperature did not enhance

the single phase of $LaGaO_3$-based perovskite as in $LaCoO_3$ or barium-based perovskite. The mechanism of perovskite formation was postulated as in Scheme 1.

The membranes were prepared by pressing the powders using a uniaxial pressing machine to form a disc (13 mm diameter, 1 mm thickness) and sintering at 1,250-1,380°C for 10 hours. The membranes were characterized by studying the structure, densification, and microstructure. The substitution of Sr in $LaGaO_3$ decreased the sintering temperature due to the grain growth in the membranes with higher Sr content. This phenomena was not found when the amount of Fe was increased. All the different Fe containing membranes had the same average grain diameters. There was no change in the perovskite structure during the membrane preparation step. The LSGF6428 membrane exhibited the single-phase perovskite structure while the other membranes showed the presence of the secondary phase of $LaSrGaO_4$ in the form of needle shape, lying on the matrix surface and cross section of membranes.

For membrane prepared without binder, the densification of LSGF6428 samples was not complete after sintering at 1,250°C with 80% relative density and the formation of an interconnected pore structure. When the sintering temperature were increased above 1,350°C, the densification was nearly complete with the relative density over 95%. The membranes remained the cubic structure under the sintering temperature below 1,400°C.

The spring back effect was appeared on membrane without binder, therefore the use of binder was necessary. However, this caused the large pore inside the membranes which could not be sintered at high temperatures. Therefore the optimum amount of binder should be further studied.

For the preliminary characterization of the incompletely densed $La_{0.6}Sr_{0.4}Ga_{0.2}Fe_{0.8}O_{3-\delta}$ disc (0.58 mm thickness with the relative density 80%), the membrane could enrich the oxygen from air up to 45% and was stable for the long time permeation. For densed $La_{0.4}Sr_{0.6}Ga_{0.4}Fe_{0.6}O_{3-\delta}$, membrane, the oxygen permeation rate was as high as 1.5 $cm^3(STP)/cm^2.min$ at 1198K and 0.62 mm thickness.