CHAPTER V
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Lithium ferrite was successfully prepared from Fe_2O_3 and Li_2CO_3 by a solid state reaction at 1100°C in furnace. The flux agent ($LiBO_2$) was used to lower its melting point so that it could be coated onto a platinum wire. The cubic phase crystal structure of $LiFe_5O_8$ was determined by X-ray Diffraction while an average grain size of 100 micrometers of $LiFe_5O_8$ was determined by Scanning Electron Microscopy.

Potential measurements of $LiFe_5O_8$ were made against a standard calomel electrode at different lithium concentrations at room temperature. At 10^{-3}, 10^{-4} and 10^{-5} M LiOH, the potential for a high ratio (2:1 of $LiBO_2$: $LiFe_5O_8$) of $LiFe_5O_8$ were 28±2 mV_{SCE}, 52±2 mV_{SCE} and 95±1 mV_{SCE} respectively, while of the equimolar ratio (1:1 of $LiBO_2$:$LiFe_5O_8$) were 18±2 mV_{SCE}, 78±4 mV_{SCE} 110±3 mV_{SCE} respectively. The fluctuation of measured potentials was in an acceptable range (< ±5 mV). For Li_2CO_3 solution, the potentials were inconsistent for both the high ratio and the equimolar ratio at different concentrations.

After several potentiometry runs, a red brownish compound was formed on the electrode surface. This compound was confirmed to be maghemite (γ-Fe_2O_3) as analyzed by XRD. However, due to the similarity of the phase structure between $LiFe_5O_8$ and γ-Fe_2O_3, Laser Raman was used to confirm the electrode phase transformation. Laser Raman indicated most of the peaks match maghemite spectra, however a few peaks are slightly offset.

The electrode equilibrium reaction was proposed as follows:

$$LiFe_5O_8 + e^- + 2H^+ \rightarrow Li^+ + Fe_2O_3 + Fe_3O_4 + H_2O$$

It seemed that this was confirmed by the calculation of the Gibbs free energy and the reaction products were a combination of magnetite, maghemite and

hematite. However, when cyclic voltammetry (CV) was applied to validate the electrode equilibrium reaction, the CV results and potential OCP measurements indicated more than one electron transfer reaction. This contradicts to the proposed reaction. This probably due to a mixed potential was established instead of an equilibrium potential when conducting those experiments. The voltammetry of this material indicated a more complex redox behavior and it is therefore somewhat ambiguous.

The $LiFe_5O_8$ showed an inability to withstand the high temperatures and pressures achieved in the autoclave. After several high temperature runs, the $LiFe_5O_8$ was more than half consumed.

The $LiFe_5O_8$ produced in this work cannot be used as a reference electrode due to its irreversible behavior. Further work and characterization is required to determine the electrochemical reaction taking place under reactor coolant conditions.

5.2 Recommendations

- Different lithium ferrite synthesis methods should be used such as high energy ball milling or mechanical solidification.
- Other flux agents should be tried apart from $LiBO_2$ since it is in doubt whether $LiBO_2$ is reacting with the testing solution. Dilithium tetraborate ($Li_2B_4O_7$) could be another flux agent to try. Since it is typically used for refractories with a high metal content, it is often mixed with alkali metal carbonate. Different coating or solidification techniques should be introduced.
- Some other potentiometry techniques can be used such as step potential, AC voltammetry and polarography.