CHAPTER V
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

A Ag/AgCl electrode was developed for potentiometric measurements at temperatures up to 300°C in which an innovative method of using titanium/titanium oxide was chosen as the electrode body. The rutile phase ,TiO_2, was identified by XRD and EDAX techniques to be the major composition of the titanium oxide film. The maximum efficiency of the cooling system allowed the KCl filling solution to maintain the AgCl electroactive element at 45 °C at a maximum operating temperature of 300°C. At room temperature, the Ag/AgCl electrode tested against the SCE at concentrations of 0.1, 1, 3.5 and saturated KCl produced a potential reproducibility of ±3 mV. The performance of the electrode at high temperatures was determined by means of autoclave testing. Measuring its potential versus a Pt electrode, representing the reversible hydrogen reaction, the Ag/AgCl electrode potentials were reproducible within ±25 mV at 0.01, 0.1 and 0.5 M KCl.

The deviation of the Ag/AgCl electrode potential, E^*, was observed by comparing the measured potential to the theoretically calculated potential using the Nernst equation. An empirical potential correlation was formulated based on the measured potential being the sum of all potentials generated in system. The correction potential, E^*, was modelled by assuming a contribution of three potentials, the liquid junction potential (LJP), the thermal liquid junction potential (TLJP) and the titanium dioxide reaction potential on the electrode body (E_{TiO_2}). With the idea of parallel electrolyte bridges, diffusing out of chloride anions and potassium cations at the bottom plug and oxygen self-diffusion through the Ti/TiO_2 electrode body, a compensating term for parallel conduction paths, δ, was determined as a function of temperature for each concentration of KCl solution.

The correlation for converting the measured potential using the Ag/AgCl reference electrode onto the SHE scale was purposed by addition of the theoretical Ag/AgCl electrode potential as well as the correction potentials. The correction terms were validated by means of potential measurement of carbon steel against the Ag/AgCl reference electrode in which concentrations of 0.1 and 0.5 M KCl were used. A comparison was made between the two sets of corrected carbon steel potentials. The empirical correction can be applied and the agreement of the corrected potentials was obtained within ± 17 mV, Table 4.10. The carbon steel potentials are consistent with previously reported values.

5.2 Recommendations

- The temperature at which Ag/AgCl used was estimated by measuring the tube surface temperature. Measuring directly the temperature of solution would give a more accurate calculated potential by the Nernst equation.
- The potential measurement of the Ag/AgCl electrode should be performed on various test solutions to validate the possible dependence of electrode potential in any solution in which it is immersed.
- The potential measurement of Ag/AgCl electrode should be performed on more concentrations of filling solutions and higher temperatures to validate the application of the empirical potential correlation.
- The potential measurement of the Ag/AgCl electrode should be performed in dynamic system to investigate the durability of the titanium dioxide film under flow conditions.
- The purposed correction potential was based on LJP, TLJP, and E_{TiO_2}. More work is required to characterize the effect of diffusion through the Ti/TiO_2 electrode body on potential measurement.
- Other possible contributing potentials on measured potential should be considered for different operating conditions.

- The Ag/AgCl electrode should be checked on a regular basis. The potential difference of ± 5 mV from the Ag/AgCl glass electrode is acceptable at room temperature.
- The electrode body should be fabricated from alternative Zr and Ti alloys to increase the maximum operating temperature without loosing strength.
- Other reference electrodes such as Ag/AgBr, $Ag/AgBO_3$, $Ag/AgSO_4$ should be tried to avoid Cl^- corrosive species in system.