

CHAPTER III EXPERIMENTAL

3.1 Materials and Chemicals

Stainless steel with 13%Chromium (AISI 420) and carbon steel type API 5CT L80 supplied by Chevron Energy Technology Company were used to study corrosion behavior in the production well. The composition of the L80 carbon steel and 13%Cr stainless steel is given in Table 3.1. An electrolyte or a corrosive solution is the produced water which is simulated using analytical grade reagents, 99.0% purity of sodium chloride (NaCl), 99.7% purity of sodium bicarbonate (NaHCO₃), 99.8% purity of potassium chloride (KCl), 95.0% purity of calcium chloride anhydrous (CaCl₂), and 99.0% purity of magnesium chloride (MgCl₂.6H₂O). All chemicals are supplied by Labscan Asia Co.,Ltd, Thailand. The solution is saturated by the corrosive gas, carbon dioxide (CO₂) with a purity of 99.9% and nitrogen (N₂) with a purity of 99.99%, supplied by Praxair (Thailand) Co.,Ltd. Hydrochloric acid (HCl) is used for adjusting pH of solution and also as a cleaning solution for a counter electrode. Three and a half grams of hexamethylene tetramine (HMT, supplied by Nicho Co.,Ltd, Thailand) in 50%(v) HCl solution is used for cleaning scale on L80 carbon steel while 10%(v) HNO₃ solution is used for cleaning the scale formed on 13%Cr steel following ASTM G1.

Table 3.1 The composition of material L80 (carbon steel) and 13%Cr (stainless steel)

| Steel | Chemical composition (%wt) | | | | | | | | | | | | |
|-------|----------------------------|-------|------|-------|------|------|------|------|-------|-------|------|------|------|
| | Al | B | C | Cr | Cu | Mn | Mo | Ni | P | S | Si | Sn | Fe |
| L80 | 0.027 | 0.002 | 0.27 | 0.27 | 0.03 | 1.24 | 0.09 | 0.02 | 0.01 | 0.011 | 0.01 | 0 | Bal. |
| 13%Cr | 0.01 | 0 | 0.35 | 12.94 | 0.08 | 0.42 | 0.04 | 0.24 | 0.019 | 0.022 | 0.25 | 0.01 | Bal. |

3.2 Equipments and Procedure

3.2.1 Potentiodynamic Polarization Technique

The experiments were carried out by using a conventional 3 electrodes system (working, reference and counter electrodes) as shown in Figure 3.1 with the potential controlled by an Autolab PGSTAT20 potentiostat (Figure 3.2) and the experimental procedures are shown in Figure 3.3.

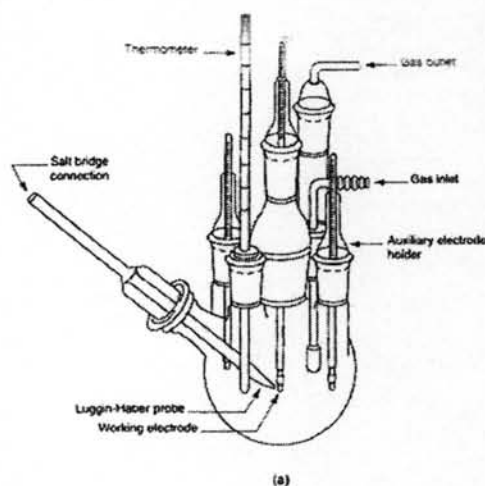


Figure 2.13 Designs of working electrodes .(© 1984 EG&G Princeton Applied Research Inc.) (Fontana, 1986).



Figure 3.1 Autolab PGSTAT20 potentiostat.

The electrode preparation is the first step. The working electrode, or material being studied, was ground with silicon carbide paper up to 1200 grit and cleaned with ethyl alcohol in an ultrasonic bath, rinsed with de-ionized water, dried with hot air, and stored in a desiccator. The specimen holder constantly provides exposed area of 1 cm^2 . The tip of reference electrode or saturated calomel electrode was rinsed with de-ionized water and avoided air to get inside. The Luggin probe was filled with saturated potassium chloride solution and then connected to the reference electrode. Last electrode was the counter or platinum electrode. To remove any precipitate formed on platinum during the experiment, 50% by volume of hydrochloric solution and de-ionized water are used as a cleaning and rinsing solution, respectively.

The sample solution was deoxygenated by purging nitrogen gas for an hour and saturated by stream of acid gas, carbon dioxide (CO_2) or gas mixture between carbon dioxide and nitrogen.

Finally, polarization curves are measured potentiodynamically in both cathodic and anodic parts by Autolab PGSTAT20 potentiostat with a scan rate of $1,000 \text{ mV/hour}$. after the polarization curve was obtained, the tafel extrapolation technique was then applied to measure the corrosion current density or i_{corr} to further calculate the corrosion rate.

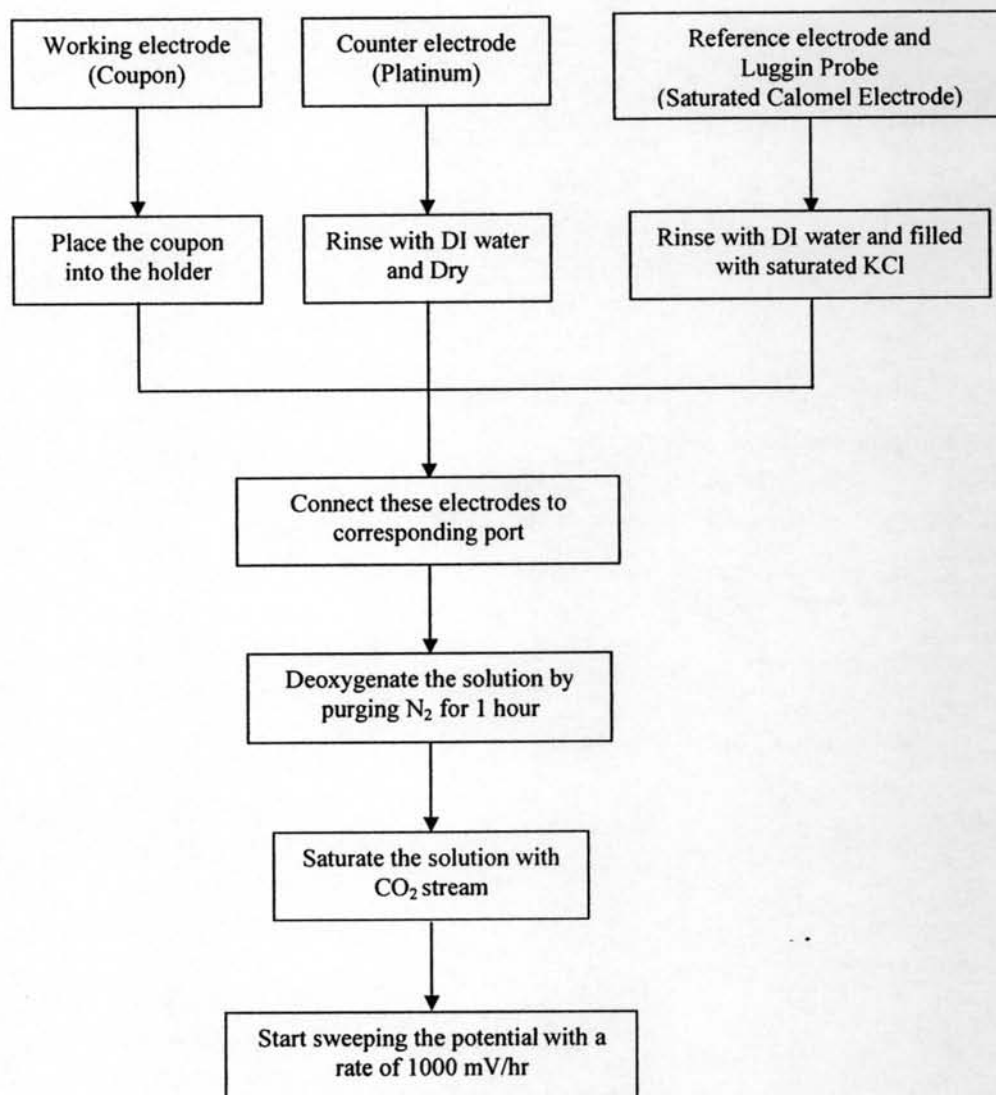


Figure 3.2 Schematic of experimental procedure for potentiodynamic polarization technique.

3.2.2. Immersion Technique

The rectangular coupons were degreased with ethanol, rinsed with de-ionized water and dried. After that, coupons were measured for the dimension and weighed. Finally, they were immersed for 2 weeks in the deoxygenated and CO₂ saturated solution for 2 weeks (carbon dioxide being bubbled continuously).

After 2 weeks, the corroded coupons removed from the immersing solution were rinsed with ethyl alcohol. The corroded coupons were evaluated by weight loss and surface examination (an optical polarizing microscope with 100X magnifications). For weight loss determination, the corroded coupons were cleaned in the cleaning solution following ASTM G1 method in order to remove corrosion scale.

3.3 Corrosive Solution Preparation

Simulated produced water was prepared based on the information provided by Chevron Thailand Exploration and Production Ltd. as shown in Table 3.2. The amount of chemicals needed to simulate and the prepared compositions are shown in Appendix A.

Table 3.2 The analysis of produced water (Chevron Thailand Exploration and Production Ltd., 2006)

| Composition | pH | Ca ²⁺ | Mg ²⁺ | K ⁺ | Na ⁺ | HCO ₃ ⁻ | Cl ⁻ |
|----------------------|-----|------------------|------------------|----------------|-----------------|-------------------------------|-----------------|
| Concentration (mg/L) | 7.7 | 1.65 | 0.35 | 41 | 1682.5 | 2317.75 | 615.75 |

The effect of temperature was determined by varying temperature in the range of 30 to 60°C while simulated produced water was saturated with carbon dioxide. To illustrate the effect of carbon dioxide, nitrogen is used to adjust the carbon dioxide partial pressure.

The composition of produced water is studied. The corrosive anion, chloride, is considered to be very affected the corrosion behavior. The concentration of chloride was varied using sodium chloride in the range of 0.1%-3.5% by weight. The pH in the range of 5-12 is adjusted by hydrochloric acid and sodium hydroxide in the simulated produced water saturated with CO₂ at 60°C. Finally, the effect of mercury was studied by adding mercuric chloride into the simulated produced water. Mercury concentration was varied from 0 to 1000 ppm at constant temperature of

60°C and the trace mercury (0-12 ppm) at various temperature and CO₂ pressure as summarized in Table 3.3.

Table 3.3 Lists of parameters for corrosion behavior study

| Experiment | Solution | Temperature | Remarks |
|---------------------------------|---|--|---|
| Temperature | Simulated produced water saturated with CO ₂ | 30°C, 50°C and 60°C | - |
| Carbon dioxide partial pressure | Simulated produced water | 60°C | P _{CO2} = 0, 0.5 and 1.0 atmospheric |
| Chloride concentration | Solution saturated with CO ₂ | 60°C | [Cl ⁻] in the range of 0.1%-3.5% |
| Solution pH | Simulated produced water saturated with CO ₂ | 60°C | HCl and NaOH are pH adjuster |
| Mercury(II) | Simulated produced water | 60°C, P _{CO2} = 1 30°C, P _{CO2} = 1 60°C, P _{CO2} = 0 and 0.5 | 0-1000 ppm 0-12 ppm 3 ppm |