

CHAPTER III METHODOLOGY

3.1 Chemicals

1-Hydroxyethylidene-1, 1-diphosphonic acid (HEDP) was selected as a scale inhibitor in this study. It has phosphonate deprotonating four hydrogens which offer four potential reacting sites. The chemical structure and physical properties of this scale inhibitor are shown in Figure 3.1 and Table 3.1.

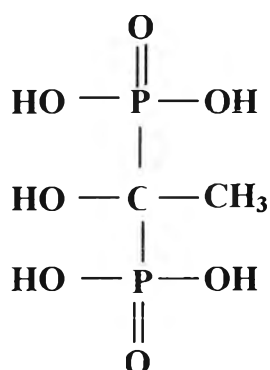


Figure 3.1 Chemical structure of 1-Hydroxyethylidene-1, 1-diphosphonic acid.

Magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) was used as coprecipitating species for synthesis of magnesium-HEDP precipitates.

Table 3.1 Chemical and physical properties of HEDP (Monsanto Technical Bulletin, Dequest 2010).

Molecular weight	206
Description	Clear solution, free form solids
Color, APHA (Hazen)	150 maximum
Total active acid content, %	58.0 – 62.0
Phosphorous acid, %	4.0 maximum
Acetic acid, %	1.0 maximum
pH of 1 % solution at 25 °C	2.0 maximum
Iron (as Fe), ppm	20 maximum
Chloride (as Cl), ppm	100 maximum
Specific gravity (20/20 °C)	1.430 – 1.471

3.2 Mg-HEDP Precipitates Synthesis

The HEDP solutions used in this study were prepared by diluting 60 % HEDP solution with deionized water. This chemical was provided by Solutia Inc. (Monsanto company). The magnesium solutions were prepared by dissolving a desire amount of magnesium chloride hexahydrate in deionized water.

The schematic of the titration apparatus used to prepare magnesium-HEDP is shown in Figure 3.2. All titrations were carried out at room temperature (about 22 °C). The magnesium solution was added into the HEDP solution until the desired molar ratio in solution was obtained. During the addition of the magnesium solution, crushed and concentrated KOH and concentrated HCl were added to maintain a constant pH. After the titration was completed, the resulting solution was left to reach equilibrium about 2 to 3

days. After that, the resulting precipitate was filtered using 0.22 micro filter membrane and washed with deionized water to eliminate interstitial precipitating solution. The precipitate was then dried at 65 °C for 24 hours.

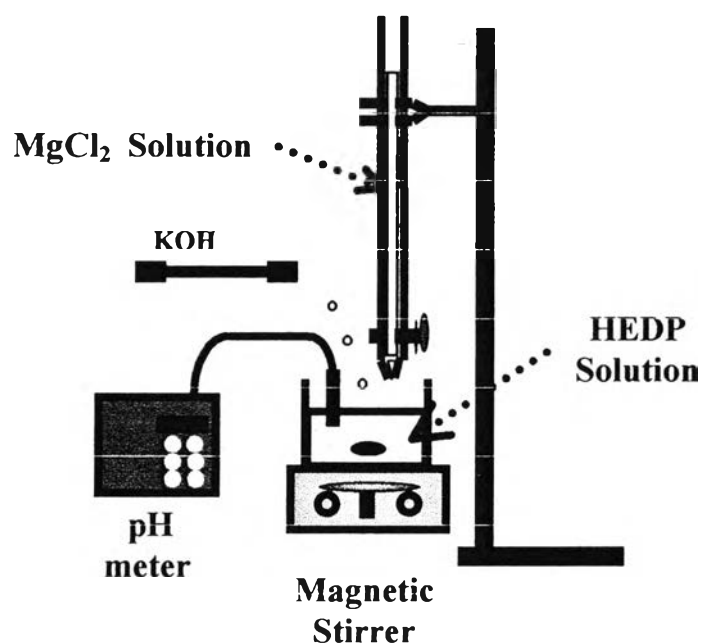


Figure 3.2 Apparatus for Mg-HEDP precipitates synthesis.

3.3 Characterization Techniques

3.3.1 Precipitate Morphology

Scanning Electron Microscope (SEM), Philips XL30, and X-ray Diffraction (XRD), Rigaku CuK_α radiation with rotating anode were used to examine the morphology and structure of precipitates synthesized in this study.

3.3.2 Chemical Composition

To determine molar ratio of magnesium to HEDP in precipitate, a known amount of precipitate was dissolved in HCl solution and prepared in a volumetric flask. Atomic Absorption Spectrophotometer (AAS) was used to analyze magnesium concentration and spectrophotometric method using Hach technique was applied to determine HEDP concentration. A 25 ml of HEDP solution was oxidized in the presence of persulfate under UV light for ten minutes to form orthophosphate. The orthophosphate formed then reacted with a molybdate reagent to yield phosphate. Finally, the phosphate concentration was measured by using an UV spectrophotometer.

3.3.3 Precipitate Solubility

Batch dissolution experiment was employed to determine the equilibrium solubility of precipitates in deionized water. Since a typical pH value of formation water is 5.5, the slurry's pH was controlled at this pH value. The excess crushed precipitate was dissolved in deionized water and stirred continuously. The HEDP concentration was monitored as a function of time during dissolution process. After this reaction was performed for a long time until equilibrium, the solubility of each precipitate was obtained.