CHAPTER 3

METHODOLOGY

3.1 Wastewater

The wastewater used in the experiment was collected from the metal plating industry in Samutprakarn province, Thailand.

3.2. Iron scrap preparation.

The iron used as absorbent was waste iron scrap, which had been recovered from industrial manufacturing process. The iron scrap is shown in Figure 3.1. The size of iron scrap was cut to about 4.4 mm x 6.5 mm x 0.1 mm as shown in Figure 3.2 (a). The iron scrap was pretreated with 0.01 N-HCl solution to remove oil from its surface. It was then dried for about 4 weeks in room temperature to form iron oxide (Figure 3.2 (b)).

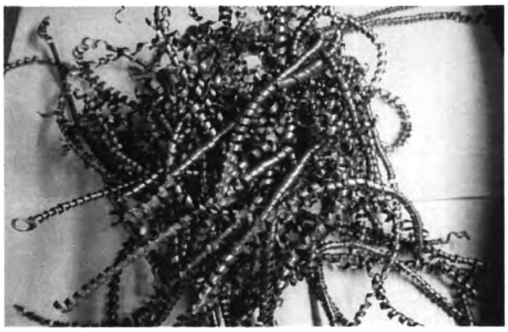
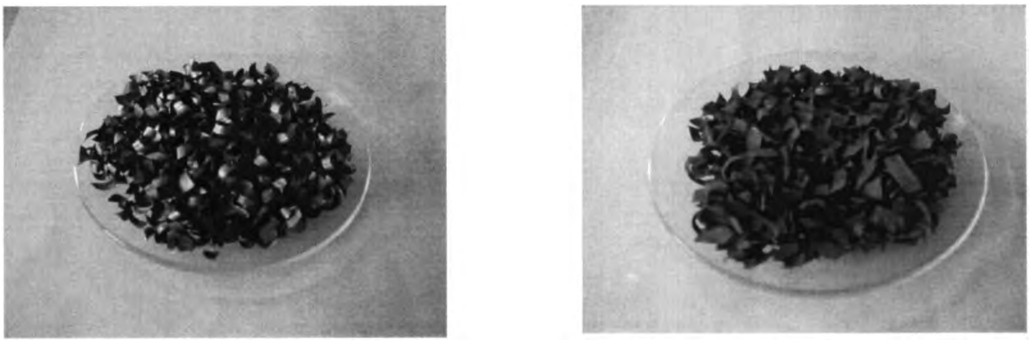


Figure 3.1 The iron scrap from industrial manufacturing process



(a) (b)

Figure 3.2 (a) The size of iron scrap was about 4.4 mm x 6.5 mm x 0.1 mm
(b) Iron oxide

3.3 Experimental Methodology

3.3.1 Characteristics of Wastewater

Metals in wastewater were analyzed with an inductive coupled plasma mass spectrometer (ICP-MS, Varian Ultra Mass 700). The pH was measured with a pH meter (HACH, Model sension 3) and the conductivity was measured with a conductivity meter (HACH, Model sension 5). The concentration of Ni in the wastewter was 565 mg/L with ICP analysis.

3.3.2 Nickel adsorption on hydrous ferric oxide (HFO)

In the batch experiment, 2 grams of iron scrap were added to each reactor. Each reactor contained 200 ml wastewater. The concentration of nickel in the wastewater was 6.9 mg/L, which had been diluted from the wastewater collected from the industry. The reactors were shaken (GFL Gesellschaft fur Labortechnik mbH, GFL model 3071) in room temperature for a preset reaction time. The experimental apparatus for the study nickel adsorption with HFO is shown in Figure 3.3. The nickel concentrations in the supernatant were analyzed with an atomic absorption spectrometer (Perkin Elmer, AA Model 2380) at the reaction times 10, 15, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 150, 160, 170, 180, 300, 420, 540, 660, 900, 1020, 1140, 1260, 1380, and 1500 minutes, respectively. The nickel percentage removal was then plotted versus reaction time.

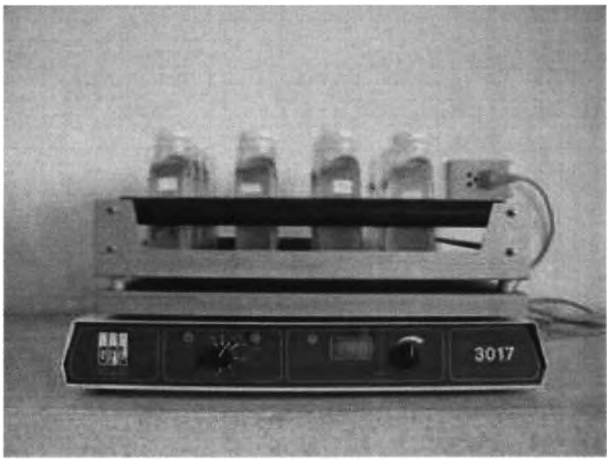


Figure 3.3 The experimental apparatus for the study nickel adsorption with HFO

3.3.3 Effect of pH on nickel removal

Another batch experiment was carried out to study the effect of pH on iron oxide sorption of nickel. The concentration of nickel was 6.9 mg/L and the experimental set up was identical to that of the previous experiment. The pH of the tested samples was adjusted to 6, 7, 8, 9, and 10, respectively, by adding 0.1 N HCl or 0.1 N NaOH. The sample bottles were shaken in room temperature for a preset time. The nickel concentrations in the supernatant were analyzed with an atomic absorption spectrometer at the reaction times 10, 15, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 150, 160, 170, 180, 300, 420, 540, 660, 900, 1020, 1140, 1260, 1380, and 1500 minutes, respectively. The nickel percentage removal was then plotted versus pH. The optimum pH obtained in this experiment was then used in the experiments described Section 3.3.4, 3.3.5 and 3.3.6.

3.3.4 Effect of column height on nickel removal efficiency

Bench-scale adsorption columns were fabricated for continuous flow experiments. The column is 2.5 cm in diameter and it is filled with waste iron scrap. The iron packing density is 2.8 g/cm. Three columns were put in series. Three column heights, namely, 30cm, 40cm, and 50cm, were tested. The testing apparatus is shown in Figure 3.4. Wastewater was diluted to have a nickel concentration of 10 mg/L. It was found in the previous experiment that the optimum pH for nickel adsorption was 7 and the pH was maintained at 7 by adding 0.1 N HCl or 0.1 N NaOH. The wastewater was pumped to an elevated water tank, which was placed on the top of a rack. The wastewater was fed to the top of the first column, then it went through the columns by gravity. The flow rate was controlled at 10 liter per day by using a valve. The effluent from the last iron scrap column was sampled and nickel concentrations were analyzed with an atomic absorption spectrometer. The nickel percentage removal was then plotted versus column height. The column height which would have the highest nickel removal would be used for the experiments conducted in Section 3.3.5 and 3.3.6.

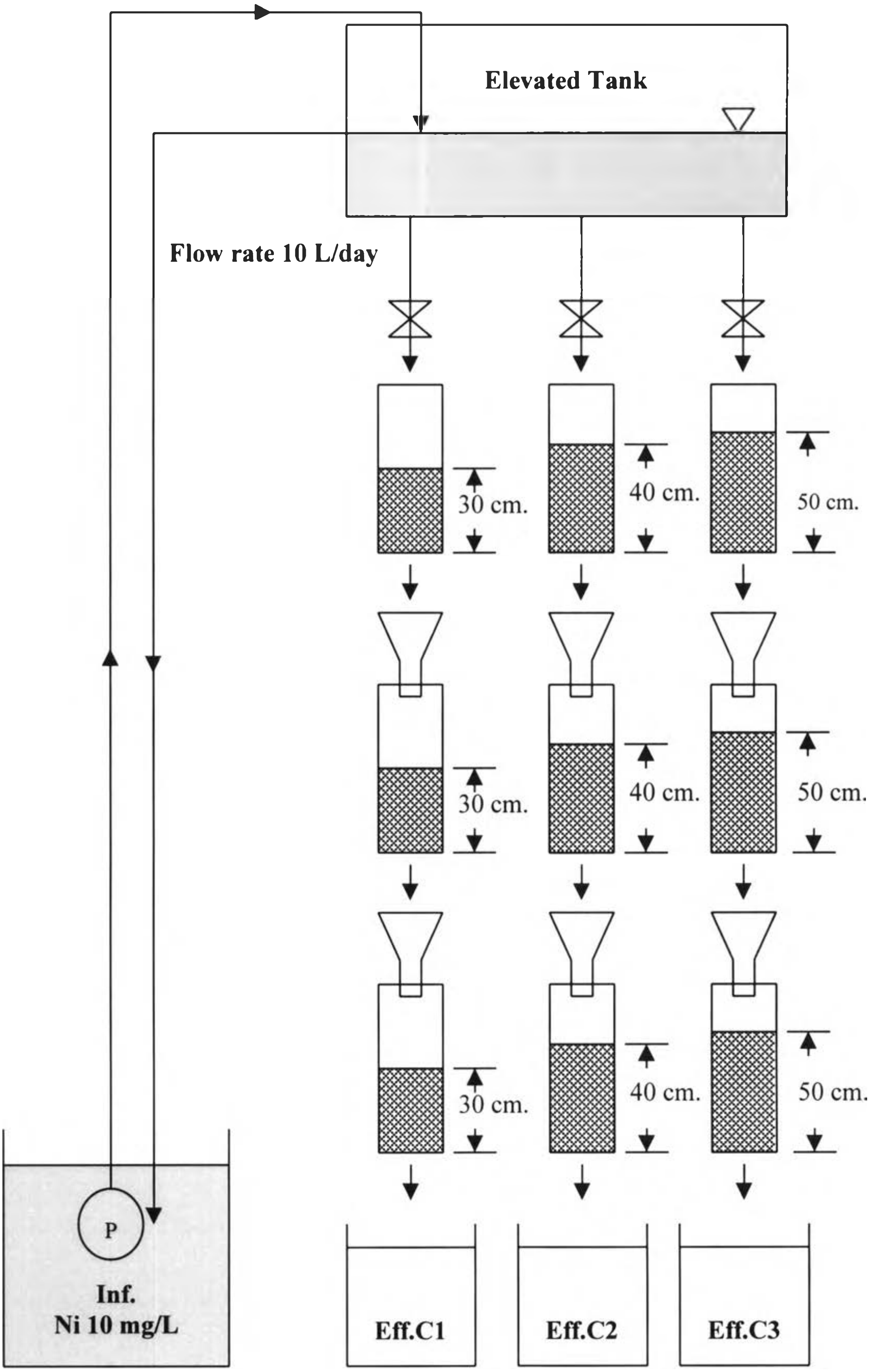


Figure 3.4 Experimental apparatus for the study of the effect of column height on nickel removal

3.3.5 Effect of influent flow rate on nickel removal efficiency

The purpose of this experiment was to determine how the influent flow rate would affect iron removal in the continuous flow conditions. Bench-scale adsorption columns were fabricated. The column is 2.5 cm in diameter and it is filled with waste iron scrap. The iron packing density is 2.8 g/cm. Two units were used for this part of experiment. Each unit has 3 columns in series. The column height, which achieved the best nickel removal in Section 3.3.4, was used here. The wastewater was diluted to have a nickel concentration of 10 mg/L and the pH was maintained at 7. The wastewater was again pumped to an elevated water tank and then fed to the top of the first column and went through all three columns by gravity. Two flow rate were tested, 5 liter per day and 15 liter per day, as shown in Figure 3.5. The effluent from the last column was sampled and nickel concentrations were analyzed with an atomic absorption spectrometer. The nickel percentage removal was then plotted versus flow rate. Three flow rates were compared, 5 liter per day, 10 liter per day, and 15 liter per day. Which the flow rate data 10-liter per day obtained from Section 3.3.4. The flow rate that removed the most nickel would be used in the experiment in Section 3.3.6.

3.3.6 Effect of nickel concentration on column performance

The same experimental apparatus used for Section 3.3.4 was used for this part of the experiment. Three units were used in this study. The column height and the flow rate obtained in Section 3.3.4. and Section 3.3.5. to have the most nickel removed was used in this part of the experiment. To study the effect of nickel loading on adsorption performance, the nickel concentration in wastewater was diluted to three concentrations in this study, 20, 50, and 100-mg/L. The pH was maintained at 7. Wastewater was pumped to an elevated water tank and then directed to the iron scrap column, as shown in Figure 3.6. Samples were taken from the columen effluent and nickel concentrations were measured.

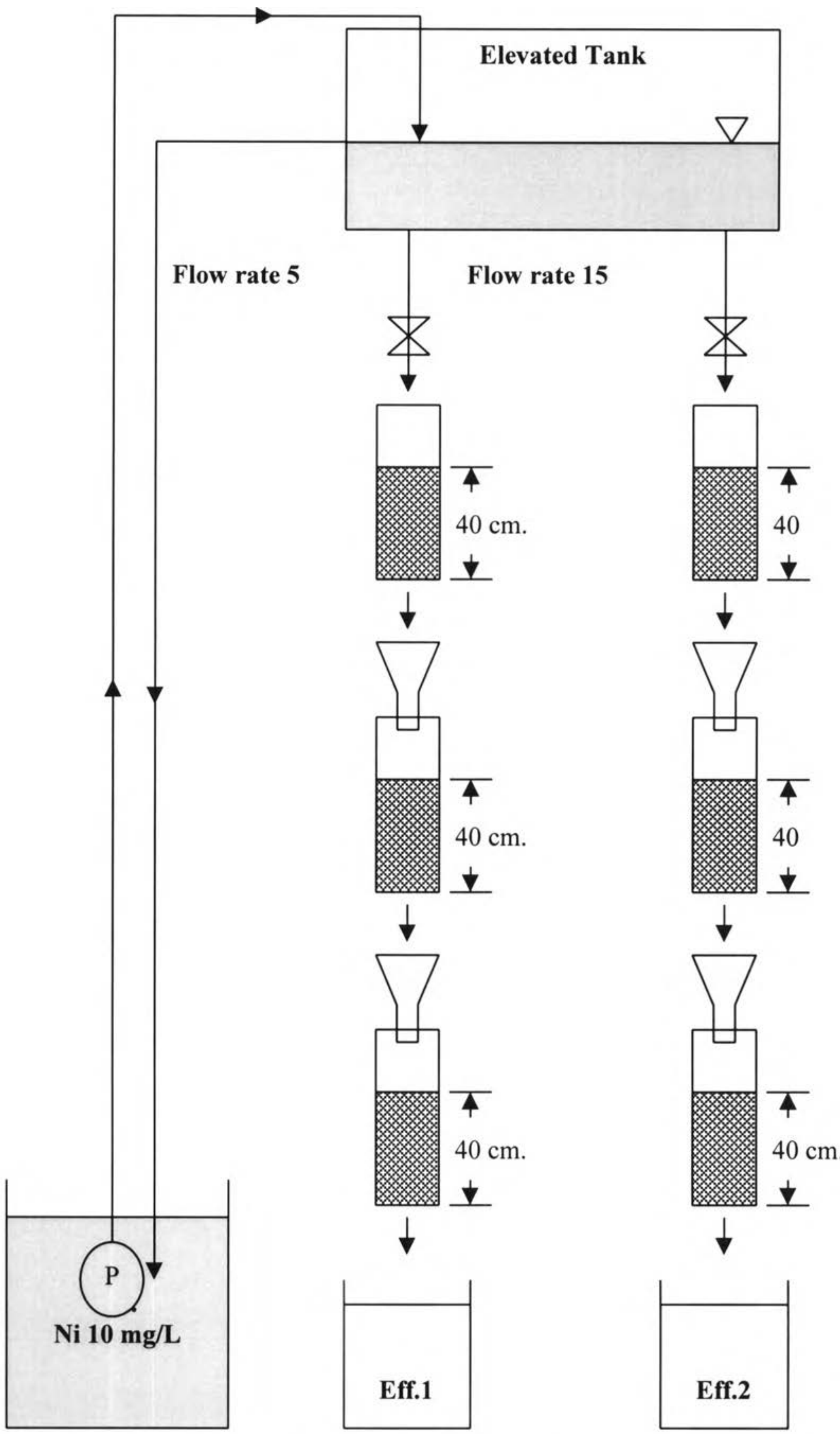


Figure 3.5 Experimental apparatus for the study of the effect of influent flow rate on nickel removal

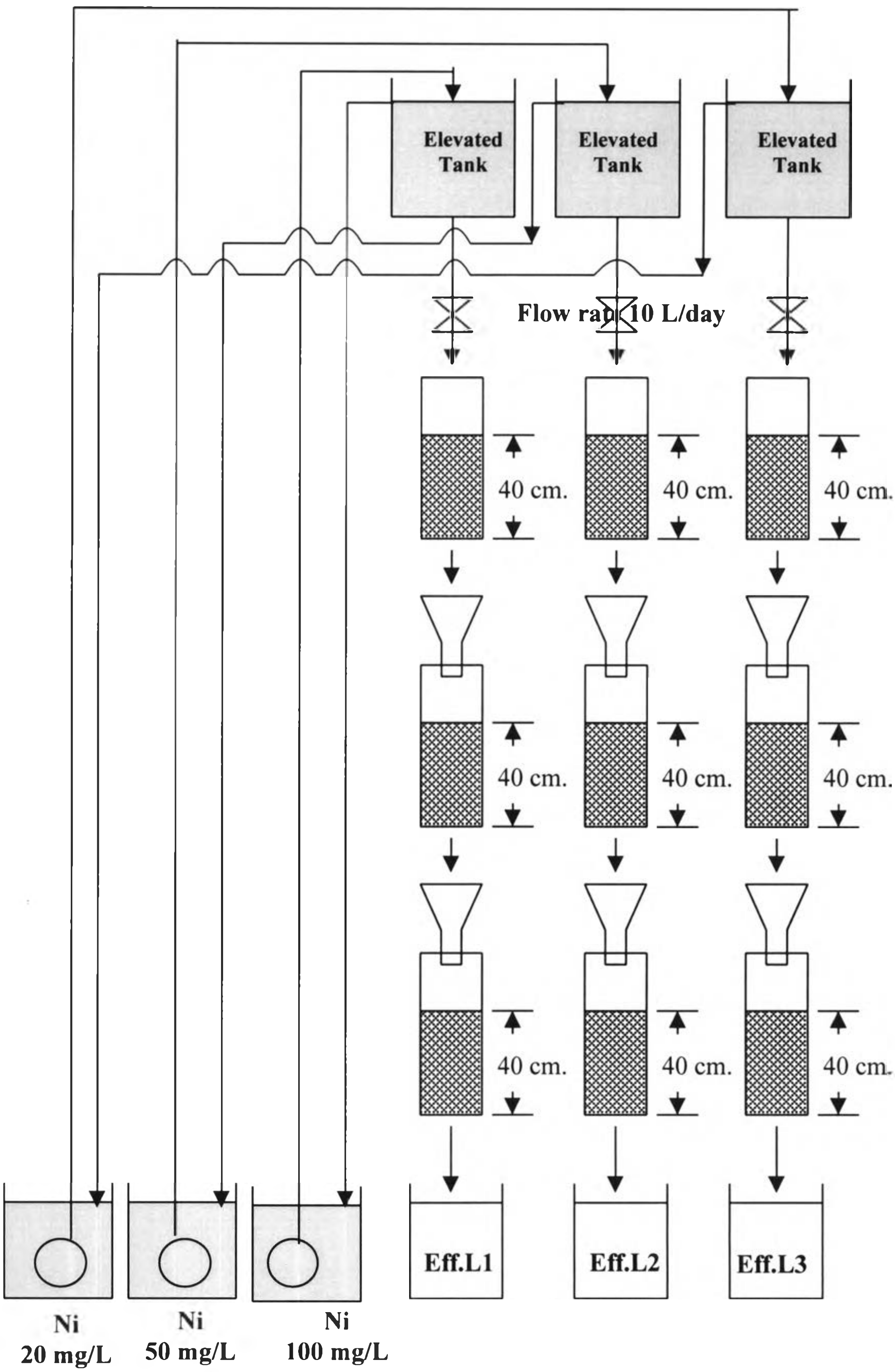


Figure 3.6 Experimental apparatus for the study of the effect of nickel concentration on column performance

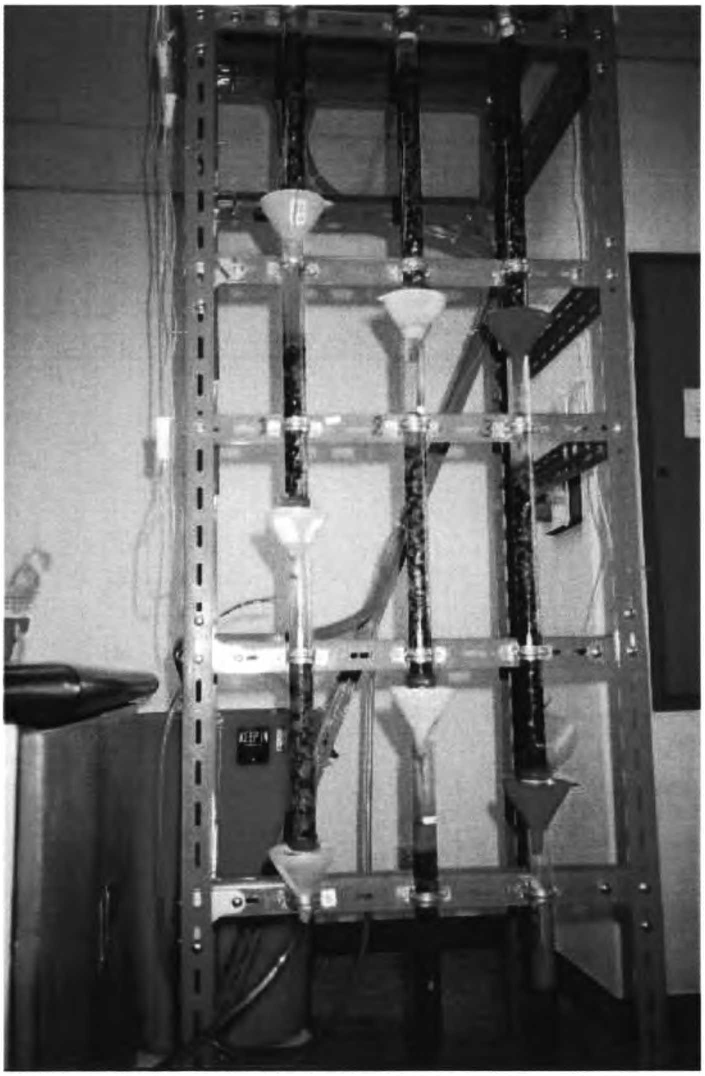


Figure 3.7 Experimental apparatus for the study of the continuous flow