2 Experimental

2.1 Apparatus and chemicals

2.1.1 Apparatus

1. UV-visible Recording Spectrophotometer, UV-2000, Hitachi, Japan
2. Atomic Absorption Spectrophotometer, AAS-275, Varian Techtron, Australia
3. pH meter with combined electrode and temperature probe, Model F-16, Horiba, Japan
4. Fourial Transfer Infrared spectrophotometer, FTIR 510, Nicolet, United States of America
5. Diffuse Reflectance Spectrophotometry, UVP -8452A, Hewlett packard, Germany

2.1.2 Chemicals

- **E. Merck, Darmstadt, Germany**
  1. Butanoic acid, G.R., $C_4H_8O_2$
  2. Calcium chloride, A.R., $CaCl_2$
  3. Cobalt chloride, Standard solution for Atomic Absorption spectroscopy, $CoCl_2$
  4. Nitric acid, A.R., HNO_3
  5. Potassium bromide, for spectroscopy, KBr
  6. Sodium chloride, Lap grade, NaCl
  7. Nickel chloride, Standard solution for Atomic Absorption spectroscopy, $NiCl_2$

- **BDH Chemical Ltd., England**
  1. Ammonium chloride, L.R., NH_4Cl
  2. Dimethylglyoxime, L.R., $CH_3C(=NOH)C(=NOH)CH_3$

- **Fluka AG, Buchs SG., Switzerland**
  1. Bipyridyl, A.R., 
  2. Copper chloride, A.R., $CuCl_2$
  3. Ethylenediamine, Purum, $NH_2CH_2CH_2NH_2$

- **Farmitalia Carlo Erba SRL AR., Italy**
  1. Ammonium hydroxide, A.R., NH_4OH
  2. Hydrochloric acid, A.R., HCl
  3. Nickel chloride, A.R., $NiCl_2$

- **Pflatz & Bauer, inc. Stanford, Conn.**
  1. Cobalt chloride, Lab grade, $CoCl_2$

- **Research Center of Ceramic Technology, Thailand**
  1. Bentonite clay

- **Deionized water**

2.2 Method to study about properties of compound

2.2.1 Physical properties

Observe the surface characteries and colour of samples

2.2.2 Infrared spectrophotometry

Infrared spectrometre was used in the range 4000 - 100 cm^{-3}(wave number) by mix with KBr disc technique.

2.2.3 Electronic spectrum

UV-vis was used with quartz cell in the range 190 - 1100 nm(wavelenght).

2.2.4 Diffuse reflectance spectrophometry

Spectra were recorded in the regions 190-820 nm of the sample

2.2.5 Atomic Absorption Spectrophotometer(AAS)

Quantitative analysis of Ni and Co were analysed by spectroscopy method. For sample solution was directly analysis. But Ni and Co in clay samples were transfer to solution by using nitric aqua region digestion.

2.2.5.1 Procedure of digestion

1 g of bentonite was put in a clean dried test tube, it was then placed in an oil bath (a 1000 ml beaker containing oil), 3 ml of HNO_3 was added into a test tube, the reaction test tube was left for 0.5-1.5 h until the reaction was complete. HCl(4 ml) was added to the test tube, the temperature of the oil bath was adjusted to 120 °C, the reaction was left to cool to room temperature and 1.5 ml HCl was added, the volume of the solution was adjusted to 25 ml by adding deionized water. Other samples were digested by the same method

2.2.5.2 Procedure of spectroscopy

1. Preparation of standard calibration curve of Ni and Co

Nickel standard solution concentration 0.2-1.0 ppm was prepared by dilute standard stock solution concentration 1000 ppm with deionized water. as followed:

1. Nickel standard solutions were prepared by pipetting 1.0 ml of nickel stock standard solution into a 100 ml volumetric flask and adding deionized water to the mark. This was 10 ppm standard soluton.

2. 1, 2, 3, 4, and 5 ml of this nickel standard solution(10 ppm) were transfered to 5 other volumetric flask, with the water added to 50 ml mark, the concentration was now 0.2, 0.4, 0.6, 0.8 and 1.0 ppm, respectively.

3.The absorbance corresponding to various concentration of nickel shown in Table 2.1-2.2. A plot of absorbance against concentration of nickel is shown in Fig. 2.1-2.2. It can be seen that the response is considered to be linear.

Table 2.1 Absorbance of Ni^{2+} ion in the range 0.200-1.000 ppm for Ni-bentonite

Concentration (ppm)	Absorbance
0.200	0.014
0.400	0.029
0.600	0.036
0.800	0.050
1.000	0.064

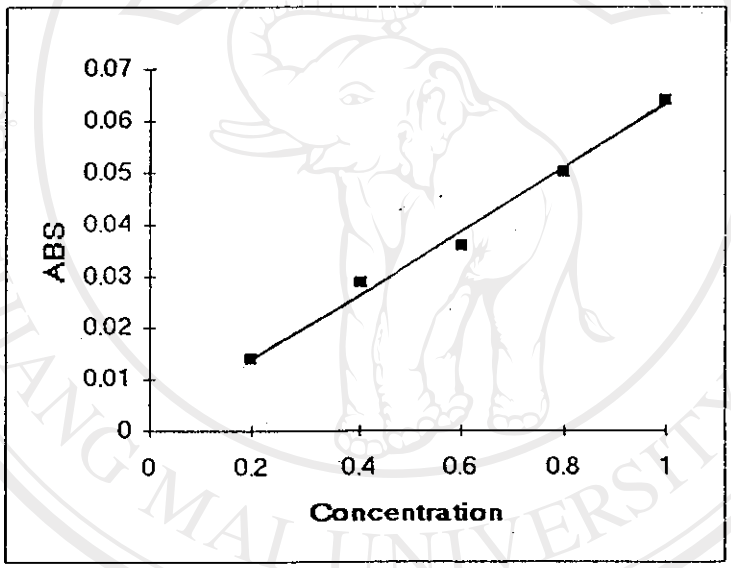

Fig 2.1 Standard calibration curve of Ni^{2+} ion in range 0.200-1.000 ppm for Ni-bentonite

Table 2.2 Absorbance of Ni^{2+} ion in the range 0.200-1.000 ppm for Ni-bentonite/1M

Concentration (ppm)	Absorbance
0.200	0.010
0.400	0.022
0.600	0.030
0.800	0.045
1.000	0.056

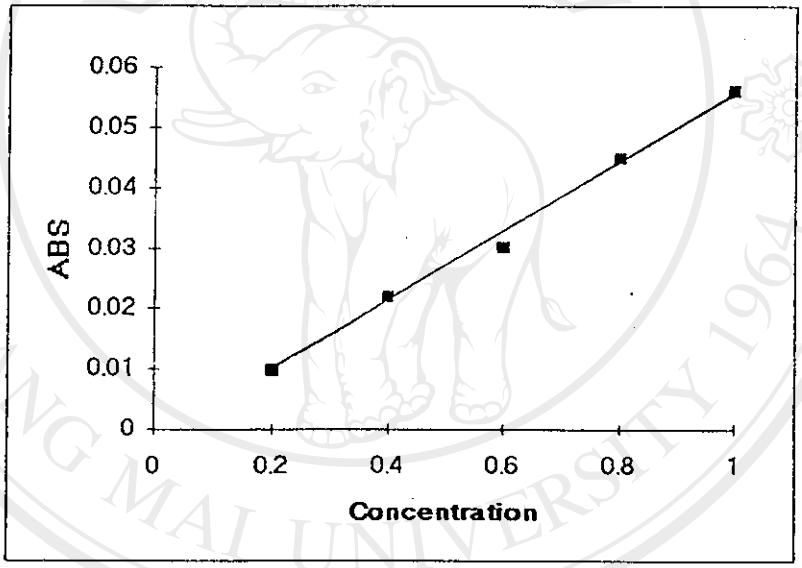

Fig 2.2 Standard calibration curve of Ni^{2+} ion in range 0.200-1.000 ppm for Ni-bentonite/1M

The standard solution of cobalt with concentration range 0.2-1.0 ppm were prepared in a similar manner with standard nickel solution.

Table 2.3 Absorbance of Co^{2+} ion in the range 0.200-1.000 ppm for Co-bentonite

Concentration (ppm)	Absorbance
0.200	0.010
0.400	0.024
0.600	0.042
0.800	0.055
1.000	0.071

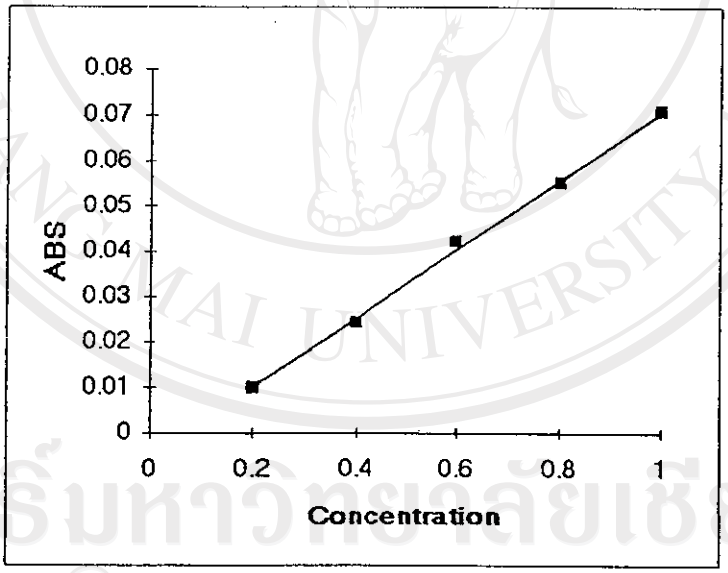

Fig 2.3 Standard calibration curve of Co^{2+} ion in range 0.200-1.000 ppm for Co-bentonite

Table 2.4 Absorbance of Co^{2+} ion in the range 0.200-1.000 ppm for Co-bentonite/1M

Concentration (ppm)	Absorbance
0.200	0.012
0.400	0.027
0.600	0.041
0.800	0.056
1.000	0.068

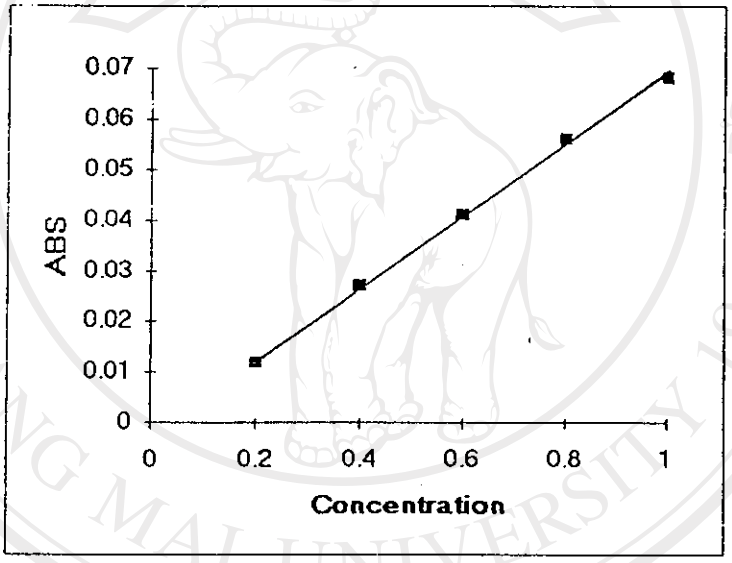

Fig 2.4 Standard calibration curve of Co^{2+} ion in range 0.200-1.000 ppm for Co-bentonite/1M

In parallel experiments, nickel stardand solution with concentration 0.4-2.0 ppm were prepared to be used in the analysis Ni released from clay as followed:

1. Nickel standard solutions were prepared by pipetting 1.0 ml of nickel stock standard solution into a 100 ml volumetric flask and adding deionized water to the mark to 10 ppm standard solution.

2. 2, 4, 6, 8 and 10 ml of this nickel standard solution(10 ppm) in the first volumetric flask were transfered into 5 other volumetric flask, with the water added to 50 ml mark to make concentration 0.4, 0.8, 1.2, 1.6 and 2.0 ppm, respectively.

3.The absorbance corresponding to various concentrations of nickel are shown in Table 2.5-2.6. A plot of absorbance against concentration of nickel is shown in Fig. 2.5-2.6. It can be seen that the response is considered to be linear.

Table 2.5 Absorbance of Ni^{2+} ion in the range 0.400-2.000 ppm

Concentration (ppm)	Absorbance
0.400	0.021
0.800	0.045
1.200	0.069
1.600	0.099
2.000	0.122

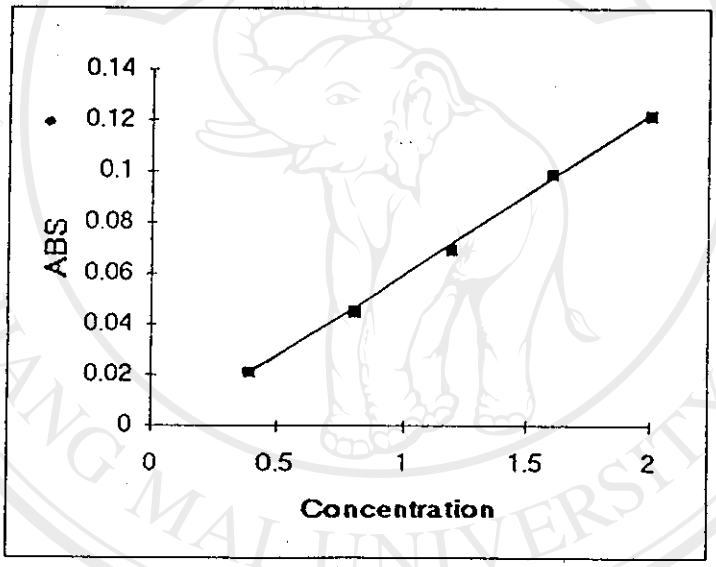

Fig. 2.5 Standard calibration curve of Ni^{2+} ion in range 0.400-2.000 ppm

Table 2.6 Absorbance of Ni^{2+} ion in the range 0.400-2.000 ppm for study lability

Concentration (ppm)	Absorbance
0.400	0.017
0.800	0.037
1.200	0.060
1.600	0.078
2.000	0.090

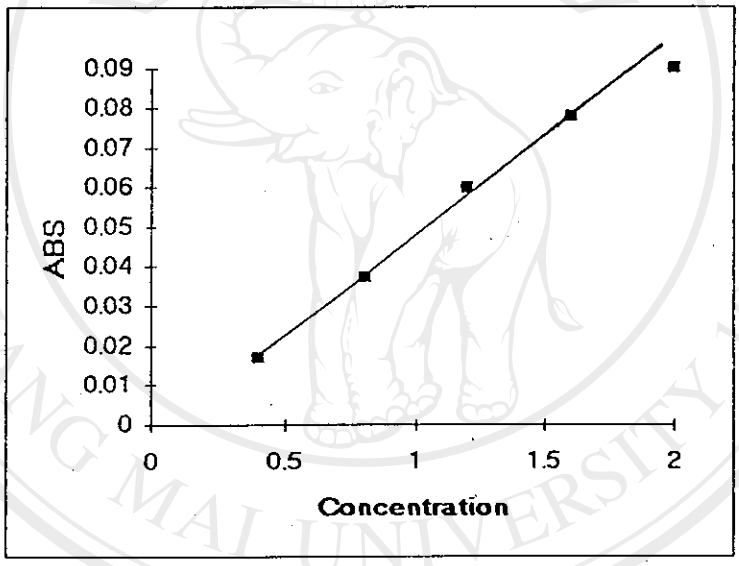

Fig. 2.6 Standard calibration curve of Ni^{2+} ion in range 0.400-2.000 ppm for study lability

The standard solution of cobalt in the concentration range of 0.4-2.0 ppm were prepared in similar manner with nickel standard solution. The absorbance corresponding to various concentrations of cobalt are shown in Table 2.7. A plot of absorbance against concentration of cobalt is shown in Fig.2.7.

Table 2.7 Absorbance of Co^{2+} ion in the range 0.400-2.000 ppm

Concentration (ppm)	Absorbance
0.400	0.030
0.800	0.063
1.200	0.101
1.600	0.142
2.000	0.175

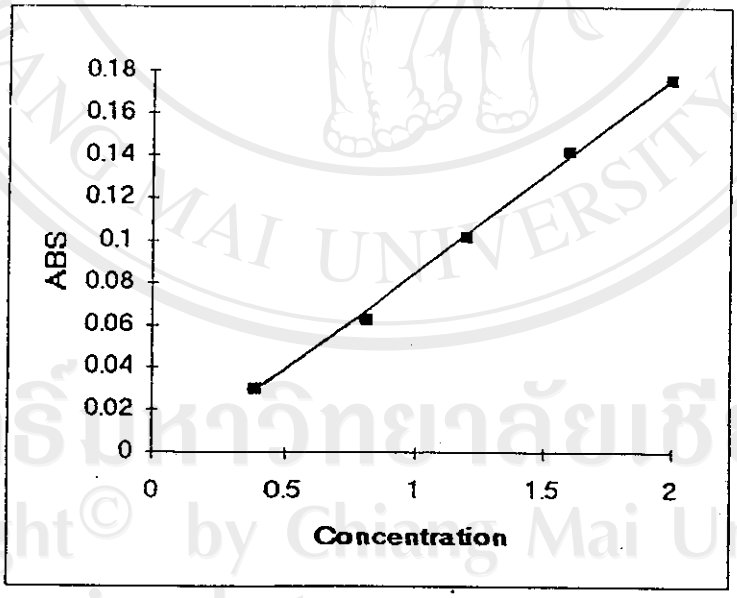

Fig. 2.7 Standard calibration curve of Co^{2+} ion in range 0.400-2.000 ppm

2. Preparation of sample solution

Clay samples were digested by nitric aqua regia digestion to change to sample solution. This sample solution were pipetted into 50 ml volumetric flask and adding 1 % HNO_3 to mark. Nickel and cobalt quantitative was analysed by AAS.

2.2.6 pH meter

pH meter was calibrated by buffer 4 and buffer 7 standard solution before using to measure sample solution.