

## CHAPTER II

### EXPERIMENTAL

#### Materials

The materials used were bentonite supplied by East Asiatic Company (Thailand), kaolinite from Ban Bang Rin, Ranong, Thailand. The kaolinite was X-ray pure with irregular morphology (43). The X-ray diffraction patterns of the Bang Rin kaolinite and bentonite are shown in Figure 2.1. Geological data, index properties, and chemical analysis and clay mineral content of this kaolinite are shown in Appendix I.

Marl (bought from Banglumpoo and Lopburi) was wash 3 times with distilled water, and made into powder form by grinding and heating at 200°C for 12 hours. The X-ray diffraction pattern of marl is shown in Figure 2.1.

All inorganic compounds used were of analytical grade from Merck:

- $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$
- KCl
- NaCl
- $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
- $\text{CaCl}_2$
- $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$
- $\text{NH}_4\text{NO}_3$
- $\text{CH}_3\text{COOK}$

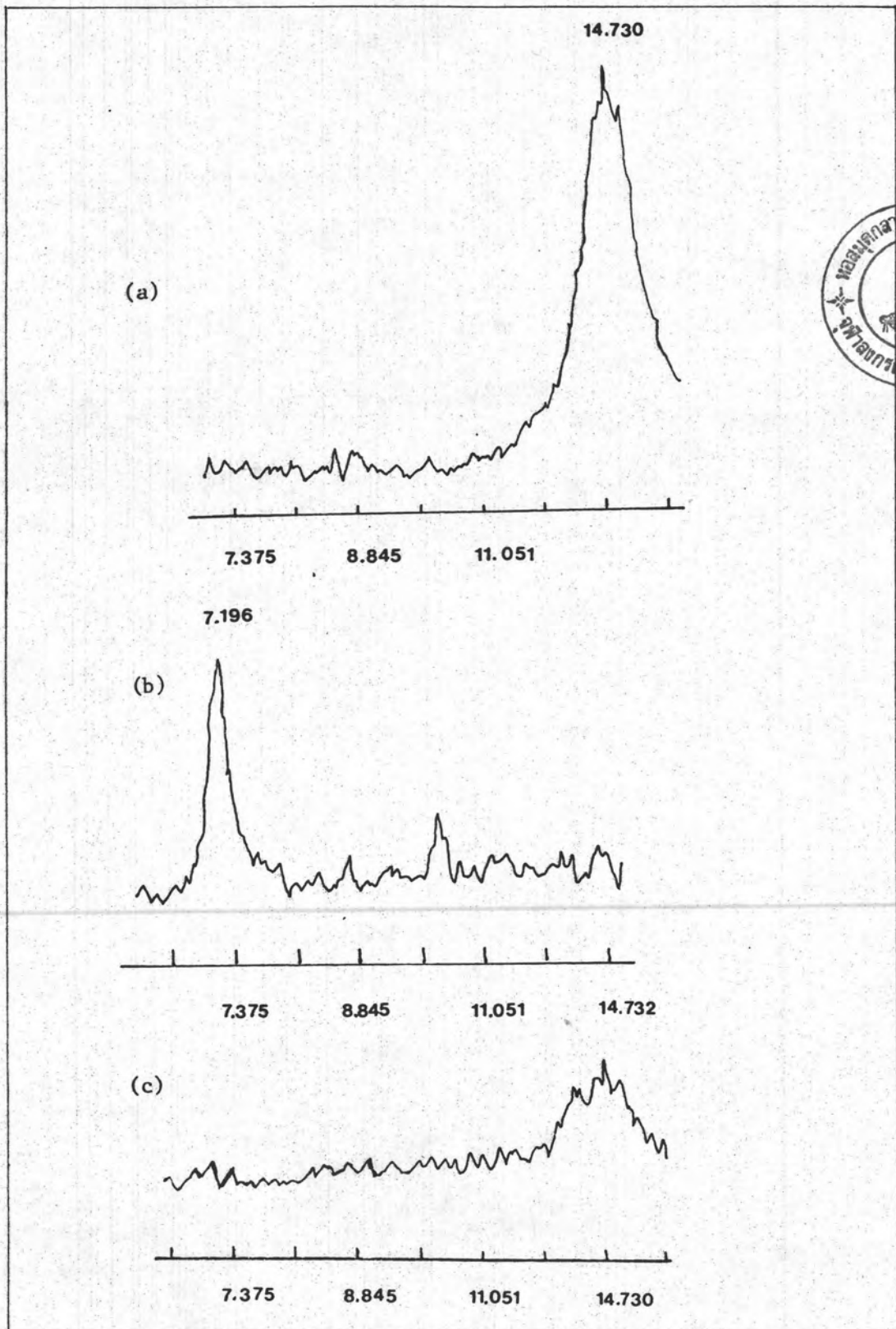


Figure 2.1 X-ray diffraction patterns of (a) bentonite, (b) kaolinite, (c) marl

#### Organic compounds used were:

- paraquat (used in the present study as a dichloride salt (1,1'-dimethyl-4,4'-bipyridinium dichloride) 27.6% W/V sold as an aqueous solution under the trade name 'Gramoxone').
- Dimethyl formamide (DMF)
- Dimethyl sulfoxide (DMSO)

#### Preparation of Intercalation Complexes

All complexes were prepared by soaking approximately 2 g of clay in a concentrated salt solution ( $.5 \text{ mol dm}^{-3}$ ) or in the organic compounds (paraquat, DMF, DMSO). All samples were shaken for one hour. The suspensions were centrifuged and the supernatant liquid discarded.

#### X-ray Diffraction Measurements

The samples from section 2.2 were spread on glass slides and air dried at room temperature for X-ray analysis.

X-ray diffraction patterns were attained from a Philips Pw 1730/10 X-ray diffractometer, operating at 40 kV and 30 mA. Scanning began at  $5^\circ(2\theta)$  at a rate of  $1^\circ/\text{min}$ ;  $2\theta$  values corresponding to the diffraction peaks were then converted into interplanar spacing,  $d(hkl)$ , by diffraction Tables.

#### IR Measurements

A small amount of air-dried complexed clay was milled with a drop of Nujol and lightly grounded in a small agate mortar. The grounded sample were then mounted between two sodium chloride IR windows.



Infrared spectra were recorded on IR-20A infrared spectrophotometer. Infrared transmittance of sample were recorded as a function of frequency (in wave number units) with the region from 4,000 to 3,000  $\text{cm}^{-1}$ .

### CEC Determination

#### 1) Single Exchangeable Cations

1.0 g of bentonite, kaolinite and marl, were soaked in 5  $\text{cm}^3$  of 1  $\text{mol dm}^{-3}$  solution of the appropriate cation, shaken for one hour and then the suspension was centrifuged and the supernatant liquid discarded. Excess cations on the clay samples were removed by washing with 1  $\times 10^{-5}$   $\text{mol dm}^{-3}$  solution of the same ion. The treatment was repeated five times and all washings were discarded.

The exchangeable ions were displaced by treating the clay with five successive 10  $\text{cm}^3$  portions of 1  $\text{mol dm}^{-3}$  ammonium nitrate solution, shaking and centrifuging. The washings were collected and the amount of displaced cation in the solution was determined by atomic absorption spectrophotometry, using an Instrumentation Laboratory Atomic Absorption Spectrophotometer model 457. Only exchanged  $\text{Ca}^{2+}$  was determined flame photometrically employing a Corning 400 flame photometer.

#### 2) Mixed Exchangeable Cations

1.0 g of bentonite, kaolinite and marl were soaked in 5  $\text{cm}^3$  of a 1  $\text{mol dm}^{-3}$  solution of chosen mixed ions and shaken for one hour then the suspensions were centrifuged and the supernatant liquid discarded. Excess cations were removed by washing with 1  $\times 10^{-5}$   $\text{mol dm}^{-3}$  solution of the mixed ions. The treatment was

repeated five times and all of washings were discarded.

The exchangeable ions were displaced by treating the clay with five successive  $10 \text{ cm}^3$  portions of  $1 \text{ mol dm}^{-3}$  ammonium nitrate solution, shaking and centrifuging. The washings were collected and the amount of exchangeable ions in the solution was determined by atomic absorption spectrophotometry.