#### CHAPTER II

## EXPERIMENTS

## 2.1 Materials

# 2.1.1 Characteristics of the Soils under Investigation

Three red soils used were the Chok Chai, the Pak Chong and the Tha Mai series. These soils can generally be classified by 2 systems as shown in Table 2.1. The Chok Chai and the Tha Mai series are in the same order of the Oxisols but the Pak Chong series is in the order of the Ultisols. In different series, soils are different in parent materials, time of development of soils and other properties. In general the red soil has a large degree of Fe and Al hydrous oxides. For simplicity, the hydrous oxides are often shown as actual iron and aluminum hydroxides,  $Fe(OH)_3$  and  $Al(OH)_3$ . Probably more correct general formula are as follows:  $Fe_2O_3$ .  $xH_2O$  and  $Al_2O_3$ .  $xH_2O$ . The x indicates that the quantity of associated water of hydration is different in minerals; as example, gibbsite  $(Al_2O_3, 3H_2O)$ , goethite  $(Fe_2O_3, H_2O)$ . The general characteristics of these soils (41) can be described as follows :

The Chok Chai series. The profile examined is located about 30 metres south of Chok Chai - Dejudom Road (at km. 18.5), Amphoe Chok Chai, Nakorn Ratchasima Province. Most of the area is occupied by fine-textured soil which has been developed from tertiary basalt. The Chok Chai series represents a typical soil in basalt terrain. The dominant clay mineral is kaolinite and it has low active iron (free iron oxide) about 2.7-3.6 %, soluble aluminum about 0.01-0.02 %.Originally, native vegetation is mixed

Table 2.1 Classification of the Soils under Investigation

Soil Series	Soil Taxonomy <sup>1</sup> (USDA)	<sup>2</sup> National
The Chok Chai	Clayey, kaolinitic,	Reddish - Brown
	iso-hyperthermic, Typic <sup>3</sup> Haplustoxs	Laterític
The Pak Chong	Clayey, kaolinitic,	Reddish - Brown
	iso-hyperthermic, oxic <sup>4</sup> Paleustults	Lateritic
The Tha Mai	Clayey, kaolinitic,	Reddish - Brown
	iso-hyperthermic, Typic <sup>5</sup> Haplorthox	Latosols

<sup>2</sup>Dudal and Moormann classification

<sup>3</sup> Haplustoxs	=	Hapl + ust + ox
<sup>4</sup> Paleustults	=	Pale + ust + ult
5 Haplorthoxs	=	Hapl + orth + ox
Hapl	-	Gk. haplous, simple, minimum horizon
Pale	-	Gk. paleos, old, excessive development
Ust	- 3	L. ustus, burnt, dry climate, usually hot in summer,
		ustic moisture regime
Orthos	-	Gk. orthos, true, the common ones
Ult	-	L. Ultimus (last), ultimate
ox	-	F. Oxide (oxide)

diciduous forest, however, much of it has been cleared for cultivation. Chili, corn, cotton, cassava, castor bean, pine-apples, jackfruits, bananas and mangoes are grown successfully on the Chok Chai soils. It is believed that this is one of the most productive soils in the northeast plateau.

The Pak Chong series. The taken profile is located about 154 kilometres northeast of Bangkok on Mitraparp Highway, Ban Pang Sok, Amphoe Pak Chong, Nakorn Ratchasima Province. It is an area of red clayey soil developed from shale associated with Carboniferous and/or Permain limestone. These soils generally occur on erosion surfaces. This particular surface is covered by a deep weathering profile. Kaolinite is the dominant clay minerals and the percentage of free iron oxide is 7.5-9.6, and soluble aluminum is 0.03-0.05%. Corn and cassava are grown on these soils. The Pak Chong series is widespread mostly in the central highlands as well as in the northwest region.

The Tha Mai series. The profile used in this study is located about 100 metres east of Tha Mai-Chanthaburi Road (at km. 5.1), Amphoe Tha Mai, Chanthaburi Province. This is a dark reddish brown, fine textured soil developed from tertiary basalt. The dominant clay mineral is kaolinite and it has high free iron oxides about 9.2-11.0 %, low soluble Al about 0.002-0.007%. The native vegetation is tropical evergreen forest but most of it is now replaced by pepper, rubber and various kinds of fruit trees (such as rambutan, durian and citrus). Gemstone minings are obviously seen in this area.

The samples for this study were selected at the depth of 86-156, 90-137 and 95-125 cm. for the Chok Chai, the Pak Chong and the Tha Mai soils, respectively. Chemical composition and general soil characteristics have been described in Tables 2.2, 2.3 and 2.4 (41).

		Particle	Size	Distri	bution	Bulk				pl	ł		Ex	tracta	ble ba	ses	KC1	Moisture
Depth	Horizon	Coarse	Sand	Silt	Clay	Density	Org.C	. N	Ext.Fe	1:1H <sub>2</sub> 0	1:1KC1	∆рН	Ca	Mg	K	Na	Ext. Al	Content
cm.			Pct -			g.cm3		-Pct						-meq/1	00g			Pct
0 -10/14	A <sub>1</sub>	0.31	22.9	25.8	51.3	1.01	1.45	0.10	3.01	4.4	4.3	-0.1	2.31	1.60	0.18	0.04	0.12	10.7
10/14- 36	B <sub>21t</sub>	0.24	17.9	20.4	61.7	1.07	0.62	0.11	2.96	4.2	3.9	-0.3	0.97	0.73	0.10	0.04	0.15	8.4
36 - 60 60 - 36	) P	10.47	15.8	24.4	59.8	], 17	0.42	0.11	2.93	3.9	3.8	-0.1	0.47	0.41	0.06	0.04	1.70	5.1
60 - 86	∫ <sup>0</sup> 22t	) 0.47	18.2	19.2	62.6	1.1/	0.26	0.03	2.66	3.8	3.7	-0.1	0.44	0.53	0.06	0.04	1.67	9.8
86 - 120 120 - 156	) <sub>p</sub>	1 02	15.4	23.0	61.6	1.2.20	0.16	tr.	3.04	3.8	3.7	-0.1	0.30	0.36	0.06	0.04	1.71	7.1
L20 - 156	∫ <sup>°</sup> 23t	1.02	16.1	20.6	63.3	] 1.20	0.14	0.04	2.73	3.9	3.8	-0.1	0.30	0.29	0.06	0.03	1.88	9.9
156 - 185 185 - 220	1	1000	17.4	23.8	58.8	]	0.26	0.02	2.75	3.9	3.9	-0.0	0.23	0.16	0.06	0.06	1.95	9.9
185 - 220	$\int ^{b}24t$	J. 0. 84	16.9	23.2	59.9	}1.1/	0.14	0.02	3.56	4.1	3.9	-0.2	0.23	0.16	0.06	0.03	1.68	9.9

Table 2.2 General Soil Characteristics and Chemical Composition of the Chok Chai Series.

Note :- Clay Fraction - Kaolinite + X-ray amorphous

		Particle	Size	Distri	bution	Bulk					pН		Ez	xtracta	able ba	ises	KC1	Moisture
Depth	Horizon	Coarse	Sand	Silt	Clay	Density	Org.C	N	Ext.Fe	1:1H20	1:1KC1	∆pH	Ca	Mg	К	Na	Ext. Al	Content
cm.			- Pct -			g. cm. 3		-Pct						- meq/	/100g -			Pct
	Ap										5.3	-0.9	7.78	2.02	0.22	0.10	tr.	6.7
12 - 30	B <sub>21t</sub>	0.18	5.3	23.5	71.2	2	1.13	0.04	8.81	5.0	4.2	-0.8	5.05	1.23	0.07	0.08	0.44	8.0
											3.8					0.05		3.9
53 - 90	B <sub>22t</sub>	10.28	3.2	12.2	84.6	11.00	0.37	0.01	8.92	4.4	3.7	-0.7	0.55	0.34	0.03	0.03	5.03	7.2
90 - 137	] <sup>2</sup> 22t	0.20	2.3	14.5	83.2	1.00	0.40	0.03	7.44	4.4	3.7	-0.7	0.45	0.42	0.03	0.04	4.31	9.8
137 - 160	) R	2010	2.4	11.0	86.8	1.00	0.40	0.03	9.30	4.1	3.7	-0.4	1.59	0.77	0.04	0.04	3.64	9.9
160 - 200	B <sub>23t</sub>	) 0.10	2.2	15.2	82.6	1.20	0.29	0.24	9.53	4.1	3.7	-0.4	1.49	0.97	0.05	0.06	3.58	9.8

Table 2.3 General Soil Characteristics and Chemical Composition of the Pak Chong Series.

Note :- Clay Fraction - Kaolinite.

Table 2.4 General Soil Characteristics and Chemical Composition of the The Mai Series.

		Particle	e Size	Distri	bution	Bulk				1	рH		Ex	tracta	ble ba	ses	KC1 Ext.	Moisture
Depth	Horizon	Coarse	Sand	Silt	Clay	Density	Org.C	N	Ext.Fe	1:1H <sub>2</sub> 0	1:1XC1	∆pH	Ca	Mg	K	Na		Content
cm.			- Pct -			g.cm.3		- Pct						- meq/	100g -			Pct
Ø - 20	A <sub>1</sub>	11.57	5.0	33.4	61.6	0.89	1.65	0.12	11.0	5.3	4.2	-1.1	2.53	0.18	0.29	0.47	0.22	6.0
20 - 50	P <sub>21</sub>	0.96	8.0	38.8	53.2	0.89	0.83	0.08	9.18	5.3	4.3	-1.0	1.96	1.24	0.20	0.31	0.31	9,8
50 - 95	B22	1.10	10.4	32.4	57.2	0.94	0.79	0.09	9.22	5.2	4.1	-1.1	1.00	0.54	0.14	0.29	0.67	2.9
95 - 125	]	1.17	4.9	29.1	66.0	1.07	0.63	0.15	10.15	5.3	4.2	-1.1	1.84	0.48	0.06	0.12	0.27	5.2
.25 - ,155	B23	0.48	3.8	26.6	69.6	1.02	0.42	0.08	10.18	5.2	4.2	-1.0	1.44	0.32	0.06	0.11	0.48	9.5
.55 - 180		-	3.9	34.9	61.2	-	0.41	0.04	10.76	5.1	4.4	-0.7	1.61	0.38	0.06	0.11	0.37	9.3

Note :- Clay fraction - Kaolinite

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# 2.1.2 Preparation of the Soil Samples

The soil samples were air-dried after stones, gravels, fresh and dried roots, and other parts of plants were removed. Then they were crushed to a size small enough to pass 10 mesh sieve, mixed well and stored in a plastic container. The moisture content of air-dried samples were determined by taking a known amount of air-dried soil and oven-dried at 378 K for 12 hours, recorded the weight of the oven-dried soil, and calculated for the moisture content. In this study, each experiment was carried out in duplicate and the results shown were the average.

#### 2.2 Apparatus and Chemicals.

# 2.2.1 Apparatus

a) Beckman pH meter model 96 with a saturated calomel-glass electrodes pair was used to measure the pH of the solutions. The saturated calomel-Ag/AgCl electrodes pair was used in potentiometric titration of chloride.

b) Klett-Summerson photoelectric colorimetric model 800-3 was used in turbidity determination of sulphate and colorimetric determination of phosphate.

c) Rotary variable speed shaking machine was used for shaking all soil samples.

## 2.2.2 Chemicals

#### a) For Chloride Adsorption.

-Hydrochloric acid solution. Eight different concentration solutions were prepared with concentration of chloride between 0-20 meg/100g soil.

-Potassium chloride solution. Five different concentration

solutions were prepared with concentration of chloride between 0-12 meq/100g soil.

-Hydrochloric acid - Potassium chloride mixture. Two sets of the mixture were prepared, Each set comprised 4 or 5 mixture solutions of different amount of KC1 and fixed amount of HC1 within one set.

-Standard solution of 0.05 mol.dm<sup>-3</sup> AgNO3.

b) For Sulphate Adsorption.

-Sulphuric acid solution. Nine different concentration solutions were prepared with concentration of sulphate between 0-20 meq/100g soil.

-Potassium sulphate solution. Four different concentration solutions were prepared with concentration of sulphate between 0-12 meq/100g soil.

-Sulphuric acid - Potassium sulphate mixture. Two sets of the mixture were prepared. Each set comprised 4 or 5 mixture solutions of different amount of  $K_2SO_4$  and fixed amount of  $H_2SO_4$  within one set.

-Standard sulphate solution. Exactly known concentration of  $K_2SO_4$  solutions were prepared at the concentration range of sulphur 0-2 meq.dm<sup>-3</sup>

-BaCl<sub>2</sub>. 2H<sub>2</sub>O crystals.

c) For Phosphate Adsorption.

-Phosphoric acid solution. Eight different concentration solutions were prepared with concentration of phosphate between 0-20 meq/100g soil.

-Potassium dihydrogen phosphate solution. Five different concentration solutions were prepared with concentration of phosphate between 0-10 meg/100g soil. -Phosphoric acid - Potassium dihydrogen phosphate mixture. Two sets of the mixture were prepared. Each set comprised 4 or 5 mixture solutions of different amount of  $\rm KH_2PO_4$  and fixed amount of  $\rm H_3PO_4$  within one set.

-Standard phosphate solution. Exactly known concentration of  $KH_2PO_4$  solutions were prepared at the concentration range of phosphorus 0-1.0 mg  $\cdot$ .dm<sup>-3</sup>.

-Reagents for colour development were prepared according to Fox et al. (35) as given in the Appendix.

2.3 Methods.

### 2.3.1 Anions Determination

a) Chloride was determined by potentiometric titration with standardized AgNO<sub>3</sub> solution using saturated calomel-Ag/AgCl electrodes. The end point was obtained graphically.

b) Sulphate was determined by turbidimetric method using  $BaCl_2$ .  $2H_2O$  crystals. Optical density was read at  $\frac{\lambda}{420}$  nm.

c) Phosphate was determined by colorimetric method using dilute ammonium molybdate and stannous chloride reagents to develop blue colour and optical density was read at  $\lambda$  560 nm.

# 2.3.2 Equilibrium Time Study

The time required for the adsorption of each anion concerned to reach equilibrium was first determined by shaking periodically 5 g. of soils (oven-dried basis, 0.D.) with a known concentration of salt solutions, KCl or  $K_2SO_4$  or  $KH_2PO_4$ , for different lengths of time up to 72 hours. The equilibrium time was read from the graph resulted from the plotting of the anion concentration in the solution (as determined by 2.3.1) against time. The equilibrium time was found to vary with type of soils and anions present as shown in Table 2.5. After the equilibrium time was known, the mixing processes in the subsequent experiments were done by shaking soil suspensions for 1 hour in the morning and in the evening, then left overnight and shaken again for another 1 hour in the next morning.

Table 2.5 Equilibrium Time of Different Anions on Three Soils.

Soil Series	The e	quilibrium time	(days)
Soll Series	Chloride	Sulphate	Phosphate
The Chok Chai	1	2	2
The Pak Chong	1	2	2
The Tha Mai	1	1	2

# 2.3.3 Anion Adsorption Isotherms

Five gram samples of soil (0.D.) were shaken periodically until equilibrium was reached in a 25 cm<sup>3</sup> of prepared solutions containing known amounts of each of the following anions i.e., chloride (either in the form of HCl or KCl or HCl-KCl mixture), or sulphate (either in the form of  $H_2SO_4$ or  $K_2SO_4$  or  $H_2SO_4-K_2SO_4$  mixture) or phosphate (either in the form of  $H_3PO_4$ or  $KH_2PO_4$  or  $H_3PO_4-KH_2PO_4$  mixture). After shaking, the slurries were filtered and the pH of filtrates were measured using a pH meter and the amounts of anions were determined by the method mentioned in 2.3.1. The amount of anion adsorbed was the difference of amount added and that found in the equilibrium filtrate. The anion adsorption isotherms were shown in Figs. 3.1-3.9.